



Organochlorine pesticide residues in water and Silver Catfish (*Bagrus bajad* Fabricius, 1775) from a tropical man-made lake, Northwestern Nigeria

Ibrahim Abubakar¹, Rasheed O. Moruf², Ibrahim Ghali-Mohammed³, Hauwa O. Sadiq⁴, Lateef A. Folorunso⁵, Akeem B. Dauda^{1*}

¹Department of Fisheries and Aquaculture, Federal University Dutsin-ma, Katsina State, Nigeria.

²Department of Fisheries and Aquaculture, Bayero University, P.M.B. 3011, Kano, Nigeria.

³Department of Veterinary Public Health and Preventive Medicine, University of Ilorin, Ilorin, Nigeria.

⁴Department of Fisheries, Aquaculture and Wildlife, University of Abuja, Nigeria.

⁵Samaru College of Agriculture, Ahmadu Bello University, Zaria, Nigeria.

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ABSTRACT

Organochloride pesticides remain widely used in Africa, despite their potential public health issues. This study assessed the organochloride pesticides (OCPs) residues in water and silver catfish from Ajiwa Reservoir. The concentrations of OCPs in water and fish samples were determined using GC analysis, while One-way analysis of variance (ANOVA) was used to compare the means for each parameter among the selected stations (Kadaji, Gamji and Kundu waje). Significantly higher concentrations of Aldrin ($2.66 \pm 0.44 \text{ ngL}^{-1}$), Dieldrin ($1.71 \pm 0.38 \text{ ngL}^{-1}$), Endrin aldehyde ($2.31 \pm 0.22 \text{ ngL}^{-1}$), Endrin ketone ($2.56 \pm 0.22 \text{ ngL}^{-1}$), beta-BHC ($1.70 \pm 0.66 \text{ ngL}^{-1}$), gamma-BHC ($0.85 \pm 0.30 \text{ ngL}^{-1}$) and delta-BHC ($1.19 \pm 0.47 \text{ ngL}^{-1}$) were found in the gills of fish from Kadaji. Also, significantly higher Endosulfan ($2.24 \pm 0.70 \text{ gL}^{-1}$), Methoxychlor ($2.63 \pm 0.95 \text{ gL}^{-1}$) and p,p'-DDE ($1.77 \pm 0.34 \text{ ngL}^{-1}$) were found in the muscles of fish from Gamji. In contrast, Dieldrin ($2.111 \pm 0.59 \text{ ngL}^{-1}$) and Endrin aldehyde ($3.84 \pm 0.86 \text{ ngL}^{-1}$) were significantly higher in the fish muscles from Kadaji. The OCPs in the water were not significantly different among the stations except Alpha-BHC ($1.50 \pm 0.63 \text{ ngL}^{-1}$). The study revealed the presence of various organochlorine pesticides in the fish tissues and the water, indicating a possible bioaccumulation in the fish. Although all the organochlorines were not above the safety level, however, it is not impossible that this might lead to possible health issues in the future.

Introduction

The global concern for environmental pollution and its impact on aquatic ecosystems has prompted extensive research into the presence of various contaminants in water bodies and their subsequent bioaccumulation in aquatic organisms. Among the myriad pollutants, organochlorine pesticides (OCPs) have attracted significant attention due to their persistence, bioaccumulation potential, and adverse effects on aquatic life (Cunha *et al.*, 2022). Organochlorine refers to a widerange of chemicals that contain carbon, chlorine and sometimes several other elements. These include herbicides, insecticides, fungicides, among others.

Organochlorine pesticides (OCPs) are a class of non-polar toxic chemical compounds classified as dichlorodiphenyl ethane, cyclodienes and chlorinated benzenes (Tzanetou and Karasali, 2022). OCPs are ubiquitous environmental contaminants spread throughout the world and have been detected invarious biota, including food, meat, drinking water, sediment, and even fish (Oliash *et al.*, 2020). Organochlorine pesticides are both acutely and chronically toxic to fish and are generally more toxic than organophosphate compounds (Akinsanya *et al.*, 2020). However, some of the latter are toxic, as are some of thehighly toxic organochlorine compounds. Organochlorine pesticides such as

* Corresponding author.

Email address: ldabak@gmail.com

dichlorodiphenyltrichloroethane (DDT) remain widely used throughout Africa as an efficient and cost-effective means of vector control for both medical and agricultural purposes (Balaska et al., 2021). Despite bans and restrictions on several organochlorine pesticides due to their environmental persistence and associated health risks, remnants of these chemicals continue to linger in aquatic ecosystems. Farmers in low-income and around less monitored water bodies like Ajiwa reservoir are still using these pesticides.

Silver catfish, a species of economic importance and ecological relevance, serves as an essential indicator organism in environmental monitoring studies (Abubakar et al., 2024). Being a sedentary species with a relatively long lifespan, Silver Catfish has the potential to accumulate contaminants over time, reflecting the environmental quality of its habitat (Ouro-Sama et al., 2021; Sogbanmu and Dauda, 2024).

The Ajiwa Reservoir, a pivotal water source in Katsina State, Nigeria (Abubakar et al., 2024), requires thorough scrutiny to assess the potential risks posed by organochlorine pesticide residues. The understanding of pesticide levels in both water and biota is crucial not only for aquatic ecosystem health but also for safeguarding human health, considering the reservoir's role in supporting various activities (Bogardi et al., 2020; Sadauki et al., 2022).

This study aims to assess the organochlorine pesticide residues in the water and Silver catfish of Ajiwa Reservoir. It employs analytical techniques and assessing the bioaccumulation patterns in Silver catfish. The findings of this research will contribute valuable insights into the environmental fate of organochlorine pesticides in this region. It could have implications for aquatic resource management, water quality regulations, and public health considerations.

Materials and Methods

Study area

The study area, Ajiwa reservoir (Figure 1) is on latitude 12°98' N and longitude 7°75' E, in Batagarawa Local Government Area (LGA), Katsina State. The main purpose of the reservoir is irrigation farming and water supply to the populace of Katsina, Batagarawa, Mashi and Mani LGAs of Katsina State (Abubakar et al., 2024; Sadauki et al., 2022). The reservoir was impounded in 1973 and commissioned in 1975 with water capacity of 22,730,000 m³ (Parkman and Haskoning, 1996). It serves as a source of revenue for the nearest communities through fishing. Neighbouring villages apart from Ajiwa include Masabu, Kunturu, Watsa, Danku, Kadaji, Kundu waje among others, all in Batagarawa LGA of Katsina state, Nigeria (Abubakar et al., 2024).

Sampling design and research duration

Water and fish samples were collected in three selected landing sites, Kadaji, Gamji and Kundu waje as shown in Figure 1. Fish of an average weight of 250-500g were collected monthly from each site. The weight was to ensure the fish are grown enough and could easily indicate the over-time bioaccumulation effect of organochlorine pesticides. This research was carried out for eight (8) months between February and September 2022 and samples were collected once a month covering two seasons i.e., four-month dry and four-month rainy season.

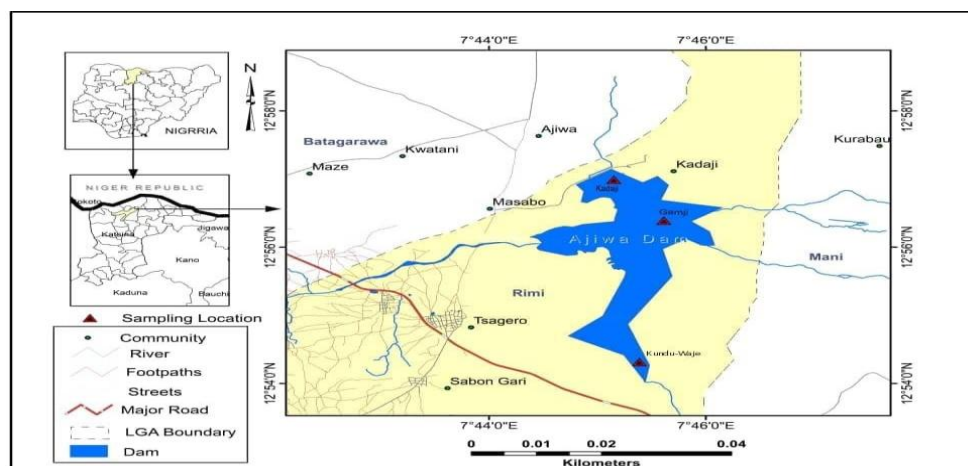


Figure 1. Map of Ajiwa reservoir showing the three sampling stations (Abubakar et al., 2024)

Analytical procedure

The gill and muscle tissues of fish samples were analysed for OCPs. Initially, the tissues were weighed and homogenised using a blender. Subsequently, 10g of the homogenised samples were combined with 100 ml of acetone and homogenised again for 20 minutes. Extraction was conducted utilising a Soxhlet extractor for approximately 25 minutes, employing a solvent mix (hexane/dichloromethane mixture (1:1, v/v)). The sample underwent sonication for an hour, and the resulting extracts were concentrated to approximately 10 mL using a rotatory evaporator (Steinwandter, 1992).

The concentrated extracts were subjected to cleaning utilising the Florisil solid-phase extraction (SPE) method. These cleaned extracts were then transferred to a Florisil column and eluted with an 80 mL eluting mixture (15% hexane). Subsequently, elutes were concentrated and adjusted to 1 mL with hexane before being transferred into vial bottles for gas chromatographic analysis (USEPA, 2007). The extraction of OCPs from the water samples employed a liquid-liquid extraction method following standard procedures (Akinsanya et al., 2020; USEPA, 2007).

Gas chromatographic analysis was performed to ascertain the concentrations of OCP residues in the fish and water samples utilizing a Hewlett-Packard (hp) 5890 Series II, equipped with a Ni electron capture detector (ECD). The carrier and makeup gas comprised nitrogen maintained at a constant flow rate of 1.0 and 29 ml/min, respectively.

To ensure quality control, stringent procedures were implemented. Instrumental calibration using standard OCP pesticide mixture preceded sample analysis. Qualitative and quantitative analysis was facilitated by comparing the retention time and peak area of the sample, respectively, with those of calibrated OCP reference standards. Recovery assays for pesticides were conducted by spiking samples with the working standard mixture of OCPs, followed by extraction, cleanup, and analytical procedures as previously described. The recoveries of OCPs were assessed by comparing the retention time and peak area of the sample with the OCP reference standard residues obtained from the spiked extract. Percentage recovery was then evaluated (Steinwandter, 1992), ranging from 85 to 90%. Method detection limits (MDL) and limit of quantification (LOQ) were determined from the curve obtained from the recovery studies (Akinnusotu

et al., 2022). The MDLs of OCPs were defined as a signal-to-noise ratio (S/N) of 3, with an MDL equivalent to the LOQ, each set at 0.01 µg/kg for the pesticides assessed.

Statistical analysis

Data were presented with mean and standard error, after homogeneous of variance and normality distribution test. The data were subjected to one-way analysis of variance (ANOVA) to compare the mean for each parameter among the selected stations. Where there is significant difference at $P < 0.05$, Duncan multiple range test was used as follow up test.

Results

Organochlorine pesticide residues in fish gills

The study identified a total of 20 organochlorine pesticides (OCPs) classified into three groups: DDT (dichlorodiphenyltrichloroethane) and its analogues, cyclodienes and hexachlorocyclohexanes (HCH). Among these groups, cyclodienes were the most dominant with 13 members, followed by four from the HCH group, and three from the DDT group. The OCPs in the gills are shown in Table 1. p,p'-DDT was exclusively found in fish samples from the Kadaji station. Endosulfan II was not detected in any of the stations, while Beta-bhc and Delta-bhc were not detected in the gills of fish from the Gamji station. Fish gill samples from the Kadaji station exhibited significantly ($P < 0.05$) higher levels of several OCPs compared to other stations. These include Aldrin ($2.66 \pm 0.44 \text{ ngL}^{-1}$), Dieldrin ($1.71 \pm 0.38 \text{ ngL}^{-1}$), Endrin aldehyde ($2.31 \pm 0.22 \text{ ngL}^{-1}$), Endrin ketone ($2.56 \pm 0.22 \text{ ngL}^{-1}$), Beta-bhc ($1.70 \pm 0.66 \text{ ngL}^{-1}$), Gamma-bhc ($0.85 \pm 0.30 \text{ ngL}^{-1}$) and Delta-bhc ($1.19 \pm 0.47 \text{ ngL}^{-1}$).

Organochlorine pesticide residues in fish muscles

The study also analyzed the presence of organochlorine pesticides (OCPs) in the muscles of fish samples (Table 2). p,p'-DDT, Endosulfan II, and Beta-bhc were not detected in any of the stations,

Table 1. Organochlorine pesticides (OCPs) present in the gills

S/N and Classification	OCPs(ngg ⁻¹)	Kadaji	Gamji	Kundu waje
DDT and Its Analogues				
1	p,p'-DDE	1.83±0.22 ^a	1.50±0.56 ^a	1.15±0.34 ^a
2	p,p'-DDD	0.62±0.13 ^a	1.54±0.40 ^a	0.53±0.29 ^a
3	p,p'-DDT	0.21±0.14 ^a	ND	ND
Cyclodienes				
4	Heptachlor	1.25±0.40 ^a	.02±0.32 ^a	1.44±0.63 ^a
5	Aldrin	2.66±0.44 ^a	1.22±0.19 ^b	2.19±0.32 ^a
6	Heptachlor-epoxide	2.34±0.23 ^a	2.64±0.55 ^a	0.72±0.19 ^b
7	Gamma-chlordane	2.33±0.46 ^a	1.94±0.33 ^a	1.53±0.38 ^a
8	Alpha-chlordane	0.34±0.47 ^a	0.88±0.46 ^a	0.82±0.26 ^a
9	Endosulfan I	0.48±0.12 ^a	1.54±0.44 ^b	0.64±0.21 ^a
10	Dieldrin	1.71±0.38 ^b	0.36±0.22 ^a	0.17±0.11 ^a
11	Endrin	0.89±0.21 ^a	1.40±0.49 ^a	0.72±0.28 ^a
12	Endosulfan II	ND	ND	ND
13	Endrin aldehyde	2.31±0.22 ^b	0.30±0.14 ^a	0.54±0.15 ^a
14	Endosulfan	1.49±0.53 ^a	1.15±0.57 ^a	1.45±0.33 ^a
15	Methoxychlor	0.23±0.12 ^a	0.23±0.12 ^a	0.73±0.21 ^a
16	Endrin ketone	2.56±0.22 ^a	0.95±0.47 ^b	2.02±0.35 ^a
HexachloroCyclohexanes				
17	Alpha-BHC	3.48±1.12 ^a	1.00±0.29 ^a	3.61±1.04 ^a
18	Beta-BHC	1.70±0.66 ^b	ND	0.36±0.36 ^a
19	Gamma-BHC	0.85±0.30 ^b	0.26±0.03 ^a	0.33±0.25 ^a
20	Delta-BHC	1.19±0.47 ^b	ND	0.41±0.21 ^a

Different letters as superscripts across rows showed significant differences (P<0.05)

while Methoxychlor was not detected in Kadaji. Fish samples from the Gamji station exhibited significantly higher levels of Endosulfan (2.24 ± 0.70 gL⁻¹), Methoxychlor (2.63 ± 0.95 gL⁻¹) and p,p'-DDE (1.77 ± 0.34 ngL⁻¹) compared to other stations. Dieldrin (2.111 ± 0.59 ngL⁻¹) and Endrin aldehyde (3.84 ± 0.86 ngL⁻¹) were significantly higher in the muscles of fish from the Kadaji station. The distribution of specific OCPs in fish muscles varied across different stations, like the observations in the gills.

Organochlorine pesticides in water

The presence of OCPs in water is shown in Table 3. Specifically, p,p'-DDT was exclusively detected in Kadaji, while Endosulfan II remained undetected across all stations. Statistical analysis reveals no significant differences (P<0.05) in OCPs levels among the stations, except for Alpha-BHC (1.50 ± 0.63 ngL⁻¹) and is notably lower in Kadaji compared to other locations.

Table 2. Organochlorine pesticides present in muscles

S/N and Classification	OCPs(ngg ⁻¹)	Kadaji	Gamji	Kundu waje
DDT and Its Analogues				
1	p,p'-DDE	0.51±0.34 ^a	1.77±0.49 ^b	0.79±0.45 ^a
2	p,p'-DDD	0.15±0.11 ^a	0.27±0.18 ^a	0.33±0.18 ^a
3	p,p'-DDT	ND	ND	ND
Cyclodienes				
4	Heptachlor	0.15±0.10 ^a	1.39±0.62 ^a	0.33±0.15 ^a
5	Aldrin	2.51±0.43 ^a	2.76±0.39 ^a	2.30±0.35 ^a
6	Heptachlor-	2.57±0.47 ^a	2.34±0.23 ^a	1.45±0.25 ^a
7	epoxide	3.73±0.94 ^a	2.82±0.28 ^a	1.76±0.45 ^a
	Gamma-			
8	chlordane	0.43±0.21 ^a	0.86±0.37 ^a	0.26±0.13 ^a
	Alpha-chlordane			
9	Endosulfan I	0.28±0.15 ^a	0.33±0.13 ^a	0.33±0.20 ^a
10	Dieldrin	2.11±0.59 ^b	0.55±0.32 ^a	0.52±0.20 ^a
11	Endrin	1.69±0.18 ^a	2.85±0.75 ^a	1.09±0.47 ^a
12	Endosulfan II	ND	ND	ND
13	Endrin aldehyde	3.84±0.86 ^b	2.27±0.72 ^a	1.14±0.61 ^a
14	Endosulfan	0.05±0.35 ^a	2.24±0.70 ^b	0.19±0.16 ^a
15	Methoxychlor	ND	2.63±0.95 ^b	0.04±0.40 ^a
16	Endrin ketone	2.44±0.74 ^a	4.56±1.00 ^a	3.63±0.42 ^a
HexachloroCyclohexanes				
17	Alpha-BHC	2.85±0.51 ^a	2.56±0.48 ^a	3.56±0.69 ^a
18	Beta-BHC	ND	ND	ND
19	Gamma-BHC	1.25±0.10 ^a	0.60±0.32 ^a	0.75±0.75 ^a
20	Delta-BHC	0.25±0.25 ^a	0.62±0.41 ^a	0.50±0.19 ^a

Different letters as superscripts across rows showed significant differences (P<0.05)

Table 3. Organochlorine pesticides present in water

S/N and Classification	OCPs(ngL ⁻¹)	Kadaji	Gamji	Kundu waje
DDT and Its Analogues				
1	p,p'-DDE	1.47±0.49 ^a	2.56±0.50 ^a	2.05±0.51 ^a
2	p,p'-DDD	1.61±0.51 ^a	2.77±0.10 ^a	2.08±0.48 ^a
3	p,p'-DDT	0.78±0.78 ^a	ND	ND
Cyclodienes				
4	Heptachlor	1.18±0.39 ^a	2.30±0.86 ^a	2.73±0.35 ^a
5	Aldrin	3.55±0.50 ^a	2.66±0.90 ^a	2.95±0.37 ^a
6	Heptachlor-	3.43±0.21 ^a	3.74±0.77 ^a	2.13±0.22 ^a
7	epoxide Gamma- chlordane	3.67±0.26 ^a	2.01±0.51 ^a	2.05±0.41 ^a
8	Alpha-chlordane	3.25±0.85 ^a	1.33±0.44 ^a	2.03±0.41 ^a
9	Endosulfan I	1.98±0.50 ^a	1.63±0.43 ^a	1.41±0.25 ^a
10	Dieldrin	3.25±1.11 ^a	0.98±0.49 ^a	1.63±0.43 ^a
11	Endrin	1.70±0.32 ^a	0.67±0.25 ^a	1.50±0.53 ^a
12	Endosulfan II	ND	ND	ND
13	Endrin aldehyde	3.30±0.56 ^a	1.46±0.58 ^a	2.86±0.53 ^a
14	Endosulfan	1.93±0.55 ^a	3.49±0.86 ^a	2.37±0.80 ^a
15	Methoxychlor	2.00±1.05 ^a	3.38±0.73 ^a	2.88±0.75 ^a
16	Endrin ketone	2.17±0.47 ^a	3.83±0.87 ^a	4.16±0.21 ^a
HexachloroCyclohexanes				
17	Alpha-BHC	1.50±0.63 ^b	4.32±0.96 ^a	4.00±0.44 ^a
18	Beta-BHC	0.91±0.61 ^a	2.11±1.21 ^a	0.77±0.51 ^a
19	Gamma-BHC	0.54±0.28 ^a	2.08±1.13 ^a	1.50±0.47 ^a
20	Delta-BHC	1.96±0.56 ^a	3.64±1.58 ^a	3.31±0.43 ^a

Different letters as superscripts across rows showed significant differences (P<0.05)

Discussion

Organochlorine pesticide residues tend to accumulate in living organisms especially aquatic organisms. The presence of the observed OCPs in the fish species might be attributed to bioaccumulation of pesticides either through feeding (via water and food), direct absorption through the skin, direct uptake through the gills during respiration or orally through drinking pesticides contaminated-water or feeding on pesticide contaminated-prey (Tongo *et al.*, 2019). The present study underscores the widespread contamination of water bodies with organochlorine pesticides. The study detected 20 different organochlorine pesticides (OCPs) in both water and fish samples, indicating significant pesticide contamination in Ajiwa reservoir. The findings align with a previous study by Akinsanya *et al.* (2020), which reported 18 OCPs in the Lekki Lagoon in Lagos. This suggests a widespread use of pesticides near water bodies despite many being banned in Nigeria (Akinsanya *et al.* 2020).

The concentration of OCPs was found to be significantly higher in the Kadaji Station, possibly due to its location in a lower part of the reservoir acting as an outlet. However, the levels remained below the maximum residue limits, which are considered safe for aquatic life (Ibigbami *et al.*, 2015). The concentrations of OCPs in this study were higher compared to those found in River Owan, Edo State, Nigeria (Akinnusotu *et al.*, 2022). This indicates varying degrees of pesticide contamination across different aquatic ecosystems. Furthermore, studies in other regions, such as Ero River (Ibigbami *et al.*, 2015) and Warri River (Tongo *et al.*, 2019), have also reported significant concentrations of OCP residues in water bodies, indicating a widespread issue.

Previous studies have shown comparable values of total pesticide residues in fish; 5.53-9.98 µg/g for *Clarias gariepinus* and 3.49- 4.98 µg/g for *Tilapia zilli* (Ezemonye *et al.*, 2015) and 0.04- 2.34 µg/kg for *C. gariepinus* and 0.02-1.73 µg/kg for *T. zilli* (Ogbeide *et al.*, 2015) in Southern Nigerian waters. The presence of higher concentrations of OCPs

in the fish suggests continuous usage of pesticides notwithstanding the fact that some of the OCPs detected (Aldrin, Endosulfan, Dieldrin and Endrin) have been banned by the environmental protection agency (Akinsanya *et al.* 2020). The continuous use of banned pesticides still thrives in most developing countries as a result of low cost and poor enforcement on usage (Tongo *et al.*, 2019). More worrisome is the fact that these pesticides are probable human carcinogens and have already been implicated in a broad range of adverse human and environmental effects, including reproductive failures and birth defects, immune system dysfunction, endocrine disruptions, and cancers (ATSDR 2018, Tongo *et al.*, 2019).

Conclusion

The results from the experiment showed that organochlorine pesticides were present and mostly similar in the water samples across the various stations. The findings also revealed presence of some organochlorine pesticide in the gills and muscle of *B. bajad* at various levels. The distribution of specific OCPs in fish muscles exhibited variations across the stations, mirroring observations in gill samples, with Kadaji having higher amount compared to the other stations. The higher amount of various organochlorine pesticides in the fish tissues compared to the water, indicate a possible bioaccumulation in the fish. Although all the organochlorine were not above the safety level, however, it is not impossible that this might lead to possible health issue in the future. It is therefore recommended that the use of organochlorine pesticides for farming near the water body should be discouraged. Also, continuous research should be carried out on the status of the pesticides in the water body to serve as an early warning signal and prevent bioaccumulation beyond the recommended limit

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