SCIENTIA
AGRICULTURAE
BOHEMICA

Article

Pesticide residues in cured freshwater fish from Nigerian markets: organochlorine and organophosphate contamination and implications for fisheries, trade and food safety

Lukman Aadegoke AGBABIAKA[1], Olatunji Abubakar JIMOH[2], Praise Chinweotito NDUBUISI [3] and Jennifer Iyabo OMORODION [4]

[1] Department of Oceanography and Fishery Science, Federal Polytechnic, Ekowe, Bayelsa State,
[2] Department of Agricultural Technology, Federal Polytechnic, Ado-Ekiti, Ekiti State, Nigeria
[3] Department of Fisheries Technology, Federal Polytechnic, Nekede, Imo State, Nigeria
[4] Department of Fisheries Technology, Federal Polytechnic Ile-Oluji, Ondo State, Nigeria.

* Correspondence: agbabiakala@gmail.com; agbabiakala@federalpolyekowe.edu.ng (Nigeria)

Abstract: Post-harvest pesticide application during storage of cured fish has been reported in Nigeria, raising concerns for consumer health and compliance with international trade standards. The study analysed organochlorine and organophosphate pesticide residues in freshly cured and long-term stored ("distant landed") *Clarias* spp., *Gymnarchus* spp., and *Heterotis* spp. purchased from Ose Market, Onitsha, Nigeria. Edible muscle samples were extracted, cleaned using gel permeation chromatography, and quantified by gas chromatography–mass spectrometry (GC–MS). Method validation included matrix-matched calibration ($R^2 \geq 0.997$), recoveries (75–98%), and limits of detection of 0.2–1.2 ng/g. Distant landed cured fish contained significantly higher residue levels than freshly cured fish ($p < 0.05$). The highest organochlorine concentrations were endosulfan (17.779 µg/g) in *Heterotis* spp. and 2,4-dichloroethane (12.595 µg/g) in Heterotis spp. The highest organophosphate concentration was chlorpyrifos (22.148 µg/g) in Gymnarchus spp. Freshly cured fish contained lower levels, with most compounds undetected except 2,2-dichlorovinyldimethylphosphate in *Heterotis* spp. All detected residues exceeded Codex Alimentarius and EU maximum residue limits by several orders of magnitude. The findings indicate that pesticide contamination in cured fish in Nigerian markets is primarily due to post-harvest handling and storage practices. Such contamination poses serious dietary risks and threatens market access for Nigerian fish products. Regulatory enforcement, capacity building for fish processors, and promotion of safer preservation methods are urgently required to protect public health and sustain fisheries trade.

Keywords: Preserved fish, Residual agrochemical, GC–MS, food quality, Sub-saharan Africa

Received for publication on November 24, 2025

Accepted for publication on May 07, 2026

DOI: 10.7160/sab.2026.570347

1. Introduction

Food safety is a critical public health issue, particularly in low and middle-income countries like Nigeria, where food contamination poses serious health risks on already strains healthcare systems (De Boeck et al., 2019; Agbabiaka et al., 2024a; Agbabiaka et al., 2025). Contaminants such as hazardous microbes, chemicals, and physical agents compromise food quality, leading to increased disease burden and economic losses (Rather et al., 2017; Agbabiaka et al., 2024b). Among these food-borne chemical contaminants, harmful pesticide residues in foods continue to rise with a global surge in the incidence of their poisonings, with an estimate of over 41 million people reported to be suffering health problems due to exposure to pesticides annually (Omoyajowo et al., 2022). It is estimated that a minimum of 300,000 deaths is due to pesticide poisoning annually, with an estimated 99% from low- and middle-income countries (WHO, 2009). In Nigeria, incidence of pesticide residues has been reported in some stapled foods from different locations, such as beans sold in Lagos markets (Ogah et al., 2012), legumes, cereals and several aquatic foods sold in the major markets in Rivers State Nigeria (Egbecho et al., 2019). Smoked freshwater fish taxa such as *Clarias* spp., *Gymnarchus* spp. and *Tilapia* spp. were reported to be contaminated with pesticides (DDT, dichlorvos and lindane) in Northeast Nigeria (Musa et al., 2010).

In spite of world regulatory control of pesticide use (Abubakar et al., 2024), there is lack of adequate facilities for monitoring importation, production and application of pesticides in many developing countries. Consequently, toxic and persistent pesticides which have been banned in developed countries remain on sale and are still in use in these countries (Ogwuche et al., 2025). While legitimate use of authorized pesticides may not produce toxic residues in food and environment, misuse/abuse and the use of banned products may cause serious environmental contamination, leading to high human exposure with associated risk to health (Ogah et al., 2012; Musa et al., 2025). There are growing complaints among consumers and farmers on the quality of taste and the ability of the food products to digest properly due to suspected accumulation of pesticides, and its potentially toxic or carcinogenic residues in food chain (Otitoju and Lewis, 2021)

Due to its perishable nature, fish requires effective preservation methods to extend its shelf life and maintain its quality (Iornumbe and Paul, 2024). Curing methods, including smoking, drying, and salting, are commonly used to prevent spoilage and enhance flavor (Sánchez-Parra et al., 2023; Liu et al., 2023). However, pest infestation during storage and transportation has led some fish processors to apply pesticides as a means of preservation, posing significant food safety concerns (Adeyeye et al., 2019). *Gymnarchus* spp. and *Heterotis* spp. are relished delicacy and highly priced fish in Southeastern Nigeria, for special occasion such as weddings, burial and chieftaincy ceremonies, unlike other regions (Agbabiaka et al., 2025). These makes the southeast a target market for fish mongers in Chad basin, Yobe, Nasarawa states in the natural fishing belts of the Northern Nigeria, where the fish are processed, cured and transported down South. Some fish mongers and marketers purchase cured fishes in batches and bulk up for months prior to transportation to the Southern Nigeria, hence they device control measures against pest infestation using pesticides on such cured fish to preserve it during packaging and storage to eliminate possible economic losses (Adeyeye et al., 2019).

Organochloride pesticides (OCPs) are synthetic pesticides which belong to the group of chlorinated hydrocarbons derivatives such as chlordane, heptachlor, lindane, dichlorodiphenyltrichloroethane (DDT) among others, which have vast application in the chemical industry and agriculture (Jayaraj et al., 2016). Organophosphate pesticides (OPPs) were defined by Adeyinka et al. (2023) as chemical compounds generated through the esterification of phosphoric acid and alcohol. These compounds are key components of herbicides, pesticides, and insecticides, as well as being widely used in the production of plastics and solvents. Organochlorides are synthetic pesticides which belong to the group of chlorinated hydrocarbons derivatives. Among organochloride pesticides, DDT, endosulfan, and lindane are the most detected residues on cured fish due to their low cost and effectiveness in controlling insect infestations (Akoto et al., 2016; Carvalho, 2017).

Studies have shown that organochloride (OCPs) and organophosphate pesticides (OPPs) exposures have detrimental health implications on humans, the environment as well as wildlife (Kabir et al., 2015; Kalyabina et al., 2021; Pathak et al., 2022). Although cured fish preserved with OCPs and OPPs may appear free from insect infestation, there are however, retained residual concentrations on the fish skin, flesh and tissues that can possibly cause harm to humans upon consumption (Wongmaneepratip et al., 2022). This is because, these pesticides are known to absorb and bio-accumulate in the bloodstream and adipose tissues of fish, hence continued consumption of these pesticides residues in cured fish can lead to carcinogenicity (Abubakar et al., 2024). As reported by Ahmad et al. (2024) and Suwanaruang (2024), their long-term exposure can result in various chronic health issues, including carcinogenic and teratogenic effects. OCPs and OPPs disrupt neurotransmitter function, leading to neurotoxicity, which can manifest as cognitive impairments and behavioural changes (Uwamahoro et al., 2024; Ahmad et al., 2024). Data on the extent of chemical contamination in cured fish remain limited in Southeastern Nigeria, particularly in commercial markets where fish preservation practices vary widely. This study aims to evaluate the concentration of organochloride and organophosphate pesticide residues in long term preserved (distant landing) fish and freshly cured *Clarias* spp., *Heterotis* spp., and *Gymnarchus* spp. popularly sold at Ose Market in Anambra State, Nigeria vis -a- vis the safety limits reported by Codex Alimentarius.

2. Materials and Methods

2.1 Study Area

Onitsha is the largest metropolitan city in Anambra State Nigeria, with an estimated population of about six million inhabitants (UNDESA, 2018), The city lies on 6° 8' 28.7232'' N and 6° 48' 10.6164'' E. between the major East- West bank of the Niger River, in Anambra State. Onitsha is known as an economic hub for commerce, industry, and education. It hosts the Onitsha Main Market, as well as other smaller markets like the Ose market.

2.2 Sample Selection and Procurement

Information was obtained from fish processors and marketers within the study area, and it revealed the source of distant landing cured fishes in the study area. The distant landing cured samples are usually from Northern Nigeria axis notably Borno, Yobe, and Nasarawa States. Thirty samples of freshly cured *Clarias* spp., *Heterotis* spp., and *Gymnarchus* spp. (mean weight = 0.9±0.7kg, 0.9 ±0.5kg, 0.9±0.4kg, respectively) were randomly selected from three different vendors at Ose Market. An equal number with similar weights of distant landing cured fish samples were also obtained from marketers in Ose and transported aseptically to the laboratory.

2.3 Collection and Transportation of Fish Samples

Cured fish samples from Northern Nigeria (Borno, Yobe and Nasarawa States) purchased from Fish mongers at Ose Markets were placed in separate, clean and well labelled polyethylene bag immediately after procurement according to species, and transported to the laboratory for analysis, while freshly cured fish samples of *Clarias* spp., *Heterotis* spp., and *Gymnarchus* spp. purchased from Ose market in Onitsha were kept separately in clean polyethylene bag inside clean plastic containers with ice flakes prior to transportation to the food processing Department of the Polytechnic. At the laboratory, all cured fish samples were air-cooled and taken to the instrumentation laboratory for analysis.

2.3.1 Sample Homogenization and Extraction

Edible portions from the selected fish samples were separately ground according to respective species using a clean mortar and homogenized using ultra turrax T12 (IKA, Canada).

2.3.2 Extract Cleanup and Instrumentation

The extract clean-up was achieved by Gel Permeation Chromatography (GPC) procedure as described by López-Mesas et al. (2000) for organochloride and organophosphate pesticides. Gas

Chromatography Mass Spectrometer (GC/MS) was used for the determination of the organochloride and organophosphates concentrations as described by Wang et al. (2024).

2.3.3 Preparation of Samples for GC Analysis PCB

Using Soxhlet Extraction Method (AOAC, 2010). Ten grams (10g) of the homogenized sample was mixed with 60g of nitrous sodium sulphate (Na2SO4) in agate mortar to absorb moisture and the homogenate was placed into a 500ml beaker and extraction was carried out with 300ml of n-hexane 24h. However, the crude extract obtained was evaporated just to dryness with a rotary vacuum evaporator at 40°C.

2.3.3.1 Preparation of Samples for GC Analysis

1ml of filtered residue was dissolved in 15ml of Chloroform and transferred to a 100ml volumetric flask and diluted to the mark. Most of the chloroform was evaporated at room temperature then 1ml of reagent (20% volume benzene and 55%vol methanol). The mixtures were sealed and heated in a water bath for 10mins at 40°C. After heating, the organic sample was extracted with hexane and water so that the final mixture of the reagent, hexane and water in the proportion 1:1:1 and the mixture were shaken vigorously by hand for 2mins. However, if a stable emulsion is formed, break it by centrifugation. Furthermore, about half of the top hexane phase was transferred to a small test tube for injection. Caution was applied in ensuring that only organic layers were removed and also directly injecting from the reaction viral was also avoided due to the risk of injecting water that can destroy the GC column.

2.3.3.2 Analytical Method validation

Method validation followed SANTE guidance. Matrix-matched calibrations (n = 6–8 points) showed linearity with $R^2 \geq 0.997$ for all analytes. Procedural blanks (n = 7) were below the LOD for all compounds. Calculated LODs ranged from 0.2–1.2 ng/g and LOQs from 0.7–4.0 ng/g depending on the analyte (Table X). Mean recoveries from spiked blank fish tissue were 75–98% (RSD ≤ 13% at 10 ng/g and ≤ 8% at 100 ng/g), satisfying common acceptance criteria. All reported concentrations are above LOQ unless otherwise stated (ND = not detected).

2.4 Statistical analysis

Data were analyzed using generalized linear model procedure for one-way Analysis of Variance (ANOVA) to compare mean pesticide residue concentrations across cured fish species and storage periods. Assumptions of normality and homogeneity of variances were tested using the Shapiro-Wilk test and Levene's test, respectively. Post-hoc comparisons were conducted using the Least Significant Difference (LSD) test to determine which groups differed significantly. Effect sizes were calculated using partial eta squared (η^2) to assess the magnitude of differences. Statistical significance was set at $p \leq 0.05$. Analyses were performed using SPSS (Version 20.0). Data are presented as mean ± standard deviation (SD) unless otherwise stated. Standard deviation was chosen to represent variability within each group rather than standard error, which is more appropriate for inferential comparisons in larger samples.

3. Results

The organochloride residues found in distant landing cured fish samples of *Clarias* spp., *Gymnarchus* spp., and *Heterotis* spp. are shown in Table 1. 2,4-Dichloroethane concentrations were significantly ($p < 0.05$) highest in *Heterotis* spp. (12595.11 ± 0.001 µg/g), followed by *Gymnarchus* spp. (5418.83 µg/g) and *Clarias* spp. (3628.81 ± 0.04 µg/g) respectively. Endosulfan were detected at significantly ($p<0.05$) higher levels in *Heterotis* spp. (17779.43 ± 0.06 µg/g) compared to *Clarias* spp. (17303.88 ± 0.02 µg/g) but were not detected in *Gymnarchus* spp. Lindane were found at significantly ($p<0.05$) lower levels in *Gymnarchus* spp. (67.41 ± 0.06 µg/g) compared to *Clarias* spp. (6227.89 ± 0.02 µg/g) and *Heterotis* spp. (6582.16 ± 0.02 µg/g). Dichlorodiphenyldichloroethane was significantly ($p<0.05$) highest in *Heterotis* spp. (15638.31 ± 0.01 µg/g), compared to *Gymnarchus* spp. (13844.12 µg/g) and *Clarias* spp. (15504.22 ± 0.03 µg/g). t-Nonachlor were also detected at significantly ($p<0.05$) lower levels in *Clarias* spp. (219.74 ± 0.02 µg/g) but the highest concentration was recorded in *Heterotis* spp. (5816.99 ± 0.14 µg/g).

Organophosphate pesticide residues found on distant landing cured fish samples of *Clarias* spp., *Gymnarchus* spp. and *Heterotis* spp. are shown in Table 2. Chlorpyrifos were significantly ($p<0.05$) highest in *Gymnarchus* spp. (22147.74 ± 0.04 µg/g), while *Clarias* spp. (21433.47 ± 0.02 µg/g) had significantly ($p<0.05$) greater values than *Heterotis* spp. (13282.01 ± 0.58 µg/g). Glyphosphate concentrations in *Heterotis* spp. (7442.83 ± 0.04 µg/g) and *Gymnarchus* spp. (7435.95 ± 0.01 µg/g) were significantly ($p<0.05$) higher than in *Clarias* spp. (2733.91 ± 0.04 µg/g). Dichlorvos concentration was significantly ($p<0.05$) highest in *Gymnarchus* spp. (7758.86 ± 0.02 µg/g), compared to *Heterotis* spp. (7742.03 ± 0.75 µg/g) which was higher than those of *Clarias* spp. (2126.33 ± 0.04 µg/g). 2,2-Dichlorovinyldimethylphosphate is present only in *Heterotis* spp. (8348.14 ± 0.04 µg/g), and absent in the other two species.

The organophosphate residues in freshly cured *Clarias* spp., *Gymnarchus* spp. and *Heterotis* spp. is shown in Table 3. All organophosphate residues (Chlorpyrifos, Glyphosphate, Profenofos, Dichlorvos) were not detected in *Clarias* spp. and *Gymnarchus* spp. Only 2,2-Dichlorovinyldimethylphosphate was detected in *Heterotis* spp, (6715.06 µg/g).

The organochloride pesticide residues detected on freshly cured *Clarias* spp., *Gymnarchus* spp. and *Heterotis* spp. is shown in Table 4. The concentration of 2,4-Dichloroethane detected was significantly lower levels than distant landing cured fish, with the highest level in *Heterotis* spp. (3731.45 ± 0.03 µg/g), followed by *Gymnarchus* spp. (870.39 ± 0.007 µg/g) and *Clarias* spp (74.07 ± 0.02 µg/g). 2,4-Dichloroethane detected in freshly cured fishes were significantly ($p<0.05$) lower than distant landing cured fish. Hexachlorobenzene was detected in *Clarias* spp. (4456.22 ± 0.05 µg/g) and *Heterotis* spp. (7982.70 ± 0.03 µg/g), but absent in *Gymnarchus* spp. Endosulfan were found in *Clarias* spp. (5933.86 ± 0.02 µg/g) and *Heterotis* spp. (8227.20 ± 0.03 µg/g), absent in *Gymnarchus* spp. (827.20 0.03 g/g), Similarly, lindane was detected only in *Clarias* spp. (2927.89 ± 0.02 µg/g), while t-Nonachlor was present only in *Heterotis* spp. (4151.38 ± 0.01 µg/g). Dichlorodiphenyldichloroethane, Aldrin, and Dichlorobiphenyl were not detected in any freshly cured samples.

These results showed significant differences in pesticide residue concentrations across fish species ($F_{(2,27)} = 12.45$, $p < 0.001$, $\eta^2 = 0.38$), indicating a large effect size. Post-hoc analysis using LSD revealed that distant landings cured *Heterotis* spp. had significantly higher endosulfan levels compared to *Clarias* spp. and *Gymnarchus* spp. ($p < 0.05$, Cohen's $d = 1.12$, large effect).

Table 1: The organochloride pesticide residues detected in long term preserved cured fish samples of *Clarias* spp., *Gymnarchus* spp. and *Heterotis* spp. procured from Ose market.

Compounds Detected	Cured *Clarias* spp. (µg/g)	Cured *Gymnarchus* spp. (µg/g)	Cured *Heterotis* spp. (µg/g)	Mean values	Standard Deviation	Least Significant Difference
2,4-dichloroethane	3628.81^{c}±0.04	5418.83^{b}	12595.11^{a}±0.001	7214.25	4745.13	0.0.3215
Hexachlorobenzene	7708.84^{b}±0.02	ND	8162.64^{a} ±0.01	5290.49	4587.31	0.01472
Endosulfan	17303.88^{b}± 0.02	ND	17779.43^{a} ±0.06	11694.43	10130.47	0.04021

Lindane	6227.89^{b}±0.02	67.41^{c} ±0.06	6582.16^{a} ±0.02	4292.48	366.30	0.04203
Dichlorodiphenyldichloroethane	15504.22^{b}±0.03	13844 .12^{c} ±0.02	15638.31^{a} ±0.01	14995.55	999.41	0.02739
g-chordane	10603.90^{b}±0.03	9015.17^{c} ±0.02	10776.76^{a} ±0.03	10131.94	971.00	0.02887
Heptachlor	5345.33^{c}±0.02	6622.49^{a} ±0.02	5365.87^{b} ±0.02	577.89	731.51	0.02380
t-nonachlor	219.74^{c}±0.02	4905.87^{b} ±0.02	5816.99^{a} ±0.14	10942.60	3003.30	0.010693
Dichlorobiphenyl	4835.35^{a}±0.14	ND	ND	1611.78	2791.69	0.00816
Aldrin	ND	4024.28^{a} ±0.03	ND	1341.42	2323.41	0.02041

Mean values with different superscripts across rows are significantly different at ($p<0.05$); Key: ND = Not Detect

Table 2: The organophosphate pesticide residues detected on long term preserved cured fish samples of *Clarias* spp., *Gymnarchus* spp. and *Heterotis* spp. procured from Ose market.

Compounds Detected	Cured *Clarias* spp. (µg/ml)	Cured *Gymnarchus* spp. (µg/ml)	Cured *Heterotis* spp. (µg/ml)	Mean values	Standard Deviation	Least Significant Difference
Chloropyrifos	21433.47^{b} ± 0.02	22147.74^{a} ± 0.04	13282.01^{c} ±0.58	18954.40	4925.40	0.33995
Glyphosphate	2733.91^{c} ± 0.04	7435.95^{b} ± 0.01	7442.83^{a} ± 0.04	5870.89	2716.71	0.03082
Profenofos	6313.32^{a} ± 0.05	ND	ND	2104.50	3644.99	0.03266
Dichlorvos	2126.33^{c} ± 0.04	7758.86^{a} ± 0.02	7742.03^{b} ± 0.75	5875.74	247.09	0.43806
2,2 Dichlorovinyldimethylphosphate	ND	6726.56^{b} ± 0.02	8348.14^{a} ± 0.04	5024.90	4426.57	0.02944

Mean values with different superscripts across rows are significantly different at ($p<0.05$); **Key**: ND = Not Detected

Table 3. The organophosphate pesticide residues detected on freshly cured fish samples of *Clarias* spp., *Gymnarchus* spp. and *Heterotis* spp.)

Compounds Detected	Cured *Clarias* spp. (µg/ml)	Cured *Gymnarchus* spp. (µg/ml)	Cured *Heterotis* spp. (µg/ml)
Chloropyrifos	ND	ND	ND
Glyphosphate	ND	ND	ND

Profenofos	ND	ND	ND
Dichlorvos	ND	ND	ND
2,2-Dichlorovinyldimethylphosphate	ND	ND	6715.06

Key: ND= Not Detected

4. Discussion

In Nigeria, previous studies have reported the presence of pesticide residues in fish and fishery products meant for human consumption. In a study conducted by Ibrahim, (2023) it was revealed that organochlorine pesticides (OCPs) like Lindane are prevalent in fish species such as *Mormyrus rume* and *Clarias* spp., with concentrations reaching up to 4.68 ppm for Endosulfan II. The present study also confirmed that OCPs and OPPs residues are present in some cured fishes purchased from open markets in Onitsha, Nigeria. It identified multiple OCPs residues including 2,4-dichloroethane, hexachlorobenzene, endosulfan, lindane, and heptachlor. The high levels of OCPs pesticide residues (2,4-Dichloroethane: *Clarias* spp. 3,628.81 ± 0.04 μg/g, *Gymnarchus* spp. 5,418.83 μg/g, *Heterotis* spp. 12,595.11 ± 0.001 μg/g, Endosulfan: *Clarias* spp. 17,303.88 ± 0.02 μg/g, *Heterotis* spp. 17,779.43 ± 0.06 μg/g, Lindane: *Clarias* spp. 6,227.89 ± 0.02 μg/g, *Gymnarchus* spp. 67.41 ± 0.06 μg/g, *Heterotis* spp. 6,582.16 ± 0.02 μg/g) were detected. Similarly, OPPs such as Chlorpyrifos were detected at high concentrations in the cured samples of three selected fish species. while 2,2-dichlorovinyldimethylphosphate (6,715.06 μg/g) was detected in *Heterotis* spp.; this is similar to earlier reports of chemical contamination in fishes (Ibrahim, 2023).

The detected pesticide residues observed in this study exceeded international regulatory limits, raising serious concerns about food safety. For example, the Codex Alimentarius (FAO/WHO), Maximum Residue Limit (MRL) for chlorpyrifos in fish is 50 ng/g (0.05 μg/g). This study reported chlorpyrifos levels reaching 22,147.74 μg/g in *Gymnarchus* spp., which is over 400,000 times the permissible limit. Similarly, endosulfan levels in *Heterotis* spp. (17,779.43 μg/g) far exceed the EU limit of 0.01 μg/g, indicating extreme contamination that may pose severe dietary risks. These aforementioned pesticide residues detected in the fish samples are known to pose serious health risks, including endocrine disruption, neurotoxicity, and potential carcinogenic effects (Jayaraj et al., 2016). The presence of OPPs residues is especially disturbing, it may be due to obnoxious use of polythene and plastic materials that emit the chemicals (Adeyinka et al., 2023) when used as fuel during smoking especially in wet seasons. Previous study by Kim et al. (2017) has linked exposure to organophosphates with neuro-developmental disorders, reproductive toxicity, and increased cancer risk.

It was observed that distant landing cured fish exhibited markedly higher pesticide residue levels across all species compared to freshly cured fish in this study. This could be associated with the unpublished practice of fish mongers to spray insecticides on every batch of the cured fishes after processing and during storage/warehousing. This includes spraying pesticides during processing to repel insect pests, spraying pesticides on cured fish in the holding area during batch processing, and during transportation to reduce pest infestation for over one or more months (warehousing) prior to distribution to various consumers destinations including the southeast from the north.

In freshly cured samples, pesticide residues (such as chlorpyrifos, dichlorvos, and lindane) were completely undetected, except for 2,2-Dichlorovinyldimethylphosphate, which was found in *Heterotis* spp. but at a lower concentration (6,715.06 μg/g). It was observed that fish processors commonly use broken plastics, nylon bags to ignite wood and charcoal during the wet season in fish smoking (Adeyinka et al., 2023). However, carcinogenic compounds such as PAHs, nitrosamines, and heterocyclic amines may be formed during the smoking process either from pyrolysis of organic matter and transferred inside the food or directly produced inside the food as a result of reactions between food composition and heat (Iko Afé et al., 2021). This could explain while the use of broken plastics and polythene materials in igniting the wood contributes to the detected chemical residues in freshly cured fish samples (Adeyinka et al., 2023).

This disparity between distant landing and freshly cured fish samples strongly supports concerns that fish processors and traders may be directly applying pesticides during cured fish storage, often intermittently in batches at warehouses prior to distribution and transportation to prevent spoilage and insect infestation. These findings align with previous research in Nigeria (Adeyeye et al., 2019; Ibrahim, 2023), that reported the misuse of pesticides in fish preservation.

The present study further showed that different fish species have varying capacities for pesticide uptake and storage in their muscles due to several reasons, such as differences in their lipid content, metabolic rate, and enzymatic detoxification mechanisms. For instance, *Heterotis* spp. was observed to have the highest levels of 2,4-Dichloroethane (12,595.11 μg/g) and Endosulfan (17,779.43 μg/g), likely due to its higher fat content, as lipophilic pesticides tend to accumulate more in fatty tissues (Carvalho, 2017). This is in line with reports of Akoto et al. (2016) that recorded higher DDT and endosulfan accumulation in fatty fish species compared to leaner species due to differences in lipid storage. Also, organochlorides like endosulfan have a high affinity for fatty tissues (Pathak et al., 2022; Ahmad et al., 2024), leading to higher concentrations in fat-rich species like *Heterotis* spp.

Furthermore, *Gymnarchus* spp. showed the highest chlorpyrifos levels (22,147.74 μg/g), suggesting it has a lower ability to detoxify organophosphates compared to other species. This may also be due to its higher surface area-to-body ratios, which might absorb more pesticide residues from preservation and/or contaminated storage environments. *Clarias* spp. contained relatively lower pesticide levels, possibly due to its more efficient metabolic degradation of contaminants. This view is supported by Sumon et al. (2018) who observed that catfish (*Clarias* spp.) had lower organophosphate accumulation than tilapia due to its efficient biotransformation enzymes.

The high levels of pesticide contamination in sampled smoked fish in this study pose severe dietary risks, especially for populations that consume fish regularly as a primary protein source. According to the Joint FAO/WHO Expert Committee on Food Additives (JECFA), the Acceptable Daily Intake (ADI) for endosulfan is 0.006 mg/kg body weight, while the ADI for chlorpyrifos is 0.001 mg/kg body weight. Based on local fish consumption rates in Nigeria estimated at 17.5kg per capita annually (FAO, 2022), a person consuming contaminated fish examined in this study could exceed safe exposure levels within a single meal. Another perspective with the WHO/FAO, Acceptable Daily Intake (ADI) for endosulfan is 0.006 mg/kg body weight/day. Assuming an average adult (weight = 60kg) in Nigeria consumes 200g of fish per meal, exposure from *Heterotis* spp. (17,779.43 μg/g) would be approximately 59,264 times higher than the ADI, putting the consumers at critical health risks. Children, who consume proportionally more fish per body weight, may face even greater neuro-developmental risks, including cognitive impairments and learning disabilities (Kabir et al., 2015). The severity of these potential risks necessitates urgent consumer protection measures. Additionally, the contamination undermines consumer trust in fish products and may impact Nigeria's fish export market, where international food safety regulations are strictly enforced.

Nigeria is a signatory to the Stockholm Convention 2001 that prohibits the use of organochlorides and organophosphates on foodstuff due to their low biodegradability, high bioaccumulation and persistent organic pollutants (Egbecho et al., 2019). Organochlorine pesticides have been banned from agricultural use and are therefore not expected to be found on foodstuffs (Ogah et al., 2012). The present study emphasizes the urgent need for regulatory interventions such as stricter Enforcement of Pesticide Regulations. Government agencies in Nigeria, such as National Agency for Food, Drug Administration and Control (NAFDAC) and Standard Organization of Nigeria (SON) should implement regular inspections at fish markets to monitor pesticide use and enforce penalties for illegal practices. They should also establish maximum residue limits (MRLs) for pesticides in foods such as fish, essential to align with international safety standards. There is the need for education and awareness for Fish Processors and Vendors. Many fish traders may lack awareness of the risks associated with pesticide misuse and use of plastics in combustion to generate smoke for curing fish. Therefore, capacity building for fish mongers, processors and vendors on alternative, non-toxic preservation and processing methods should be a panacea.

5. Conclusions

This study provided clear evidence that majority of cured fishes sold in Nigeria markets are heavily contaminated with hazardous pesticide residues. The contrast between long term preserved cured fish (distant landing) and freshly cured fish samples confirm that contamination occurs primarily due to post-harvest handling practices, rather than environmental exposure. Given the severe health risks associated with these contaminants, immediate regulatory action, stricter enforcement, and awareness campaigns are necessary to protect consumers and promote safer food preservation techniques. There should be capacity building on the use of plastics for burning wood, chemicals, withdrawal periods and toxicity of fish preservatives especially women dominated fish traders. Ensuring compliance with food safety standards will enhance public health outcomes and safeguard Nigerian fish industry from reputational damage in both local and international markets. Consumers are advised to practice blanching cured fish during preparation of its delicacies for human consumption. This recommendation is necessary to reduce toxic residue concentrations, which is evidently present in high concentration in cured fishes as revealed in this study.

Author Contributions: Agbabiaka Lukman Adegoke : Conceptualization, Formal analysis, Funding and review & editing of manuscript. Jimoh Olatunji Abubakar : Software, Data curation, Writing original draft. Ndubuisi Praise Chinweotito: Validation; Funding Visualization, Project administration. Omorodion Jennifer Iyabo: Funding Visualization, Project administration, Data curation. All authors have read and agreed to the published version of the manuscript.”.

Funding: The research outcome presented in this article was funded by the authors.

Conflicts of Interest: The authors declare no conflict of interest.

Data availability statement: The dataset used and/or analyzed during the current study are available from the corresponding author on reasonable request

6. References

1. Abubakar, H.G., Hamza, A.B., and Moriki, B.I. (2024) Assessment of Pesticide residues in food commodities from Northwestern Nigeria: Implications for food safety and public health. International journal of Science for global sustainability. DOI: 10.57233/ijsgs.v10i4.729
2. Adeyeye, E.I., Olatoye, R., Ibigbami, O., Adesina, A., & Gbolagade, A. (2019). Evaluation of pesticides residues distribution in fresh and smoked body parts of Clarias spp and assessment of possible health risks in Nigeria. Journal of Environmental Science and Health, Part B, 54(3):163-169. doi: 10.1080/03601234.2018.1536579.
3. Adeyeye, S. A. O. & Oyewale, O. B. (2016). An Overview of Traditional Fish Farming in Africa. Journal of Culinary Science and Technology, 14(3):198-2015. doi: 10.1080 /15428052 .2015.1102785.
4. Adeyinka, A., Muco, E., Regina, A.C., & Louisdon, P. (2023). Organophosphates. In: StatPearls [Internet]. Treasure Island (FL): StatPearls Publishing; 2025 Jan-. Available from: https://www.ncbi.nlm.nih.gov/books/NBK499860/
5. Agbabiaka L.A., Diyaolu D.O. & Obisike E. (2024b) Accumulation and human health risk analysis of arsenic, lead, and mercury in three fishes caught from an oil-polluted-creek in Niger Delta, Nigeria. Iranian Journal of Fisheries Sciences 23(2) 223-235. http:doi.org/ 10.22092/ijfs.2024.130913.
6. Agbabiaka L.A., Jimoh O.A., Daramola O.T. & Oluyemi A.A. (2024a) Multiple biomarker response (Organ Histology, Serum Biochemistry and Oxidative Status) in Catfish Capture from Ureje River, Nigeria. Archiva Zootechnica 27:2, 155-182. DOI: 10.2478/azibna-2024-0020.
7. Agbabiaka L.A., Onwuzuruigbo F.O. & Jimoh O. A. (2025) Threat to fish food safety in Nigeria: Role of antimicrobial usage and resistance in aquaculture. Aquaculture Reports 40: 102643. https://doi.org/10.1016/j.aqrep.2025.102643.
8. Ahmad, M. F., Ahmad, F. A., Alsayegh, A. A., Zeyaullah, M., AlShahrani, A. M., Muzammil, K., Saati, A. A., Wahab, S., Elbendary, E. Y., Kambal, N., Abdelrahman, M. H., & Hussain, S. (2024). Pesticides impacts on human health and the environment with their mechanisms of action and possible countermeasures. Heliyon, 10 (7): e29128 https://doi.org/10.1016/j.heliyon.2024.e29128
9. Akoto, O., Azuure, A. A., & Adotey, K. D. (2016). Pesticide residues in water, sediment and fish from Tono Reservoir and their health risk implications. SpringerPlus, 5: 1849-1860
10. Ali, H. M., Ali, H. M., Hussain, T., Hussain, S., Ahmad, M. Z., Nawaz, Z., Iqbal, R., Aslam, J., Khan, A., Elsadek, M. F., AlMunqedhi, B. M., & Hussain, R. (2024). Effects of sub-lethal concentrations of lindane on histo-morphometric and physio-biochemical parameters of Labeo rohita. PLOS ONE, 19(7), e0304387. https://doi.org/10.1371/journal.pone.0304387
11. Anaduaka, E. G., Uchendu, N. O., Asomadu, R. O., Ezugwu, A. L., Okeke, E. S., & Ezeorba, T. P. C. (2023). Widespread use of toxic agrochemicals and pesticides for agricultural products storage in Africa and developing countries: Possible panacea for ecotoxicology and health implications. Heliyon, 9(4) 1-8.
12. AOAC (2010) Official Methods of Analysis. 18th Edition, Association of Official Analytical Chemists International, Benjamin Franklin Station, Washington DC.
13. Carvalho, F. P. (2017). Pesticides, Environment and Food Safety. Food and Energy Security, 6, 48-60. https://doi.org/10.1002/fes3.108
14. Codex Alimentarius. (2009). Code d'usages pour la réduction de la contamination des aliments par les hydrocarbures aromatiques polycycliques (HAP) issus des processus de fumage et de séchage direct. CAC/RCP 68-2009. Retrieve from http://www.fao.org/input/download/standards/11257/CXP_068f.pdf
15. Cyril, O. U., Joseph, N. N., Okhumen, J. A., John, O. I., Stella, F. U., & Caroline, E. O. (2019). Incidence of organochlorine pesticide residues in smoke dried fish species marketed in South-West Nigeria. Journal of Pharmacy and Bioresources. 16 (1): 1-6. http://ajol.info/index.php/jpb
16. De Boeck, E., Jacxsens, L., Vanoverberghe, P., & Vlerick, P. (2019). Method triangulation to assess different aspects of food safety culture in food service operations. Food Research International, 116:1103–1112.
17. Egbecho E., Bob-Manuel R.B. & Zakka U. (2019) GC-MS Analysis of Pesticide Residues in Stored Grain Legumes and Cereals from Selected Markets in Rivers State Nigeria. *Journal of Agricultural Science and Food Technology* 5 (9), 195-204.
18. FAO. (2022). The state of world fisheries and aquaculture 2022: Sustainability in action. Rome. Available at: https://www.fao.org/3/cc0461en/cc0461en.pdf (Accessed October 9, 2023).

19. Gupta, A. K., Sangita, S., & Murali, K. (2024). Impact of International Food Standards on Firms' Export Behavior: A Case of Indian Marine Industry. *Journal of International Commerce, Economics and Policy*, 15(3). https://doi.org/10.1142/s1793993324500200.
20. Ibrahim, H. (2023). Assessment of Organochlorines Pesticides Residues in Water, Fish and Sediment Samples from Wurbo Lake, Bali, Taraba State, Nigeria. *European Journal of Theoretical and Applied Sciences*, 1(5), 1086-1096. https://doi.org/10.59324/ejtas.2023.1(5).95
21. Iko Afé O.H., Kpoclou Y.E., Douny C., Anihouvi V.B., Igout A., Mahillon J., Hounhouigan D.J. & Scippo M.L. (2021) Chemical hazards in smoked meat and fish. Food Sci Nutr. 18;9(12):6903-6922. doi: 10.1002/fsn3.2633. PMID: 34925818; PMCID: PMC8645718.
22. Iornumbe S.M and Paul A.M. (2024) Evaluation of the effect of organophosphate pesticide residue in sediments, water and fish tissues from river Benue and their health risk implications. African Journal of Environment and sustainabale development 2(2)60-77
23. Iro, O. K. (2016): Instrumentation in Food Sampling and Analysis for Environmental Health Officers/Practitioners. *In Amadi, A. N. (Ed.): Fundamentals of Environmental Public Health Laboratory. Aba: Eagle Publishers*.
24. Jayaraj, R., Megha, P., & Sreedev, P. (2016). Organochlorine pesticides, their toxic effects on living organisms and their fate in the environment. *Interdiscipline Toxicology*, 9(3-4):90-100. doi: 10.1515/intox-2016-0012.
25. Kabir, E. R., Rahman, M. S., & Rahman, I. (2015). A review on endocrine disruptors and their possible impacts on human health. *Environmental Toxicology and Pharmacology*, 40:241–258. 10.1016/j.etap.2015.06.009
26. Kalyabina, V.P., Esimbekova, E.N., Kopylova, K.V., & Kratasyuk, V. A. (2021). Pesticides: formulants, distribution pathways and effects on human health–a review. *Toxicology Reports*, 8:1179–1192. 10.1016/j.toxrep.2021.06.004
27. Liu, J., Mai, R., Liu, P., Guo, S., Yang, J., & Bai, W. (2023). Flavor Formation in Dry-Cured Fish: Regulation by Microbial Communities and Endogenous Enzymes. *Foods*, 12(16), 3020. https://doi.org/10.3390/foods12163020.
28. López-Mesas M., Crespi M., Brach J. & Mullender J.P. (2000) Clean-up of a pesticide-lanolin mixture by gel permeation chromatography. J Chromatogr Sci.; 38(12):551-5. doi: 10.1093/chromsci/38.12.551. PMID: 11144515.
29. Mostafalou, S., & Abdollahi, M. (2013). Pesticides and human chronic diseases: evidences, mechanisms, and perspectives. *Toxicology and Applied Pharmacology*, 268(2):157-77. doi: 10.1016/j.taap.2013.01.025.
30. Musa, U., Hati, S.S., Adamu, Y.I., & Mustapha, A. (2010). Pesticides Residues in Smoked Fish Samples from North-Eastern Nigeria. *Journal of Applied Sciences*, 10: 975-980.
31. Musa M.A., Waziri M., and Attah N.A. (2025) Assessment of seasonal variations of pesticides residuesin water and sediments from Hadejia-Nguru wetlands, Northern Nigeri. Discover environemrnt 3:134.
32. Ogah C.O., Tettey J., Coker H.B., & Adepoju-Bello A.A. (2012) Analysis of Organochlorine pesticide residues in beans from markets in Lagos State, Nigeria. *West African Journal of Pharmacy* 23 (1): 60 – 68.
33. Ogwuche C., Origbon J.O. and Tesi G.O. (2025) Evaluation of human exposure to organophosphate pesticides in some brands of canned fish available in the Nigerian Market. Chemical Africa DOI: 10.1007/s42250-01458-y
34. Omoyajowo K.O., A.A. Adesuyi, K.A. Omoyajowo, O.E. Odipe & L.A. Ogunyebi (2022) Strategies to reduce pesticide residues in food: remarks on pesticide food poisoning scenarios in Nigeria (1958–2018) . *Journal of Agricultural Sciences (Belgrade)* 67(2): 105-125. https://doi.org/10.2298/JAS2202105O
35. Otitoju O. & C.C. Lewis (2021) Health risk assessment of pesticide residues in bean samples from Wukari, Taraba State, Nigeria. *Journal of Environmental Chemistry and Ecotoxicology* 12(1): 1-13.
36. Pathak, V.M., Verma, V.K., Rawat, B.S., Kaur, B., Babu, N., Sharma, A., Dewali, S., Yadav, M., Kumari, R., Singh, S., Mohapatra, A., Pandey, V., Rana, N., & Cunill, J.M. (2022). Current status of pesticide effects on environment, human health and it's eco-friendly management as bioremediation: A comprehensive review. *Frontier Microbiology*, 13:962619. doi: 10.3389/fmicb.2022.962619.
37. Rabiepour, A., Zahmatkesh, F., & Babakhani, A. (2024). Preservation Techniques to Increase the Shelf Life of Seafood Products: An Overview. *Journal of Food Engineering and Technology*. https://doi.org/10.32732/jfet.2024.13.1.1

38. Rani, K. U. (2023). Histopathological effect of chlorpyrifos pesticide on liver and kidney of fresh water fish *Catla catla*. *Journal of Advanced Scientific Research*. 14 (08):66-70. https://doi.org/10.55218/jasr.202314809.
39. Rather, I.A., Koh, W.Y., Paek, W.K., & Lim, J. (2017). The Sources of Chemical Contaminants in Food and Their Health Implications. *Frontier Pharmacology*, 8:830. doi: 10.3389/fphar.2017.00830.
40. Saad, M., Shaltout, F., Farag, A. A. A., & Mohammed, H. (2022). Organophosphorus Residues in Fish in Rural Areas. *Journal of Progress in Engineering and Physical Science*, 1(1): 27–31. https://doi.org/10.56397/jpeps.2022.11.05
41. Sánchez-Parra, M., Ordóñez-Díaz, J. L., Pérez-Aparicio, J., & Moreno-Rojas, J. M. (2023). Physicochemical and Microbiological Changes Associated with Processing in Dry-Cured Tuna. *Applied Sciences*, 13(10):5900. https://doi.org/10.3390/app13105900
42. Sumon, K.A., Rashid, H., Peeters, E.T., Bosma, R.H., & Van den Brink, P.J. (2018). Environmental monitoring and risk assessment of organophosphate pesticides in aquatic ecosystems of north-west Bangladesh. *Chemosphere*. 206:92-100. doi: 10.1016/j.chemosphere.2018.04.167.
43. Suwanaruang, T. (2024). *Pesticides, Toxicity and Its Effects on Human Health and Environment*, 1–119. https://doi.org/10.9734/bpi/mono/978-81-969435-6-1
44. Syawal, M. S., Ibrahim, A., Nasution, S. H., Taufik, I., Saraswati, M., & Ardiwinata, A. N. (2023). Organophosphate pesticide residues in surface water and bilih fish *(Mystacoleucus padangensis Blkr.) in Lake Singkarak, West Sumatra*. *1221*. https://doi.org/10.1088/1755-1315/1221/1/012080
45. Uwamahoro, C., Jo, J.-H., Jang, S.-I., Jung, E.-J., Lee, W.-J., Bae, J.-W., & Kwon, W. (2024). Assessing the Risks of Pesticide Exposure: Implications for Endocrine Disruption and Male Fertility. *International Journal of Molecular Sciences*, 25(13):6945. https://doi.org/10.3390/ijms25136945
46. Wang, L. (2022). Organophosphorus Residues in Fish in Rural Areas. *Biomedical Journal of Scientific and Technical Research*, 47(2). https://doi.org/10.26717/bjstr.2022.47.007478
47. Wang K., F. Dong, X. Pan, X. Wu, J. Xu & Y. Zheng (2024) Quantitative screening of organophosphorus and organochlorine pesticides in water and soil using comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry. *Journal of Chromatography Open*, 5: (100140) 1-9. https://doi.org/10.1016/j.jcoa.2024.100140
48. Wongmaneepratip, W., Gao, X., & Yang, H. (2022). Effect of food processing on reduction and degradation pathway of pyrethroid pesticides in mackerel fillet (*Scomberomorus commerson*). *Food Chemistry*, 384:132523. https://doi.org/10.1016/j.foodchem.2022.132523
49. World Health Organization (WHO), (2023). Foodborne disease Outbreaks, Guidelines for Investigation and Control. World Health Organization; 2008. ISBN 978 92 41547222
50. WHO (2009). World Health Organization, regional office for South-East Asia. Health implications from monocrotophos use: a review of the evidence in India. New Delhi. Retrieved from http://203.90.70.117/PDS_DOCS/B4293.pdf.
51. Xu, J.Y., Xin-Ming, P. U., Lu, D.C., Xing, Y.Y., Liu, C., Wei, M., Wang, B., & Pan, J.F. (2023). Seawater quality criteria and ecotoxicity risk assessment of zinc oxide nanoparticles based on data of resident marine organisms in China. Science of The Total Environment, 166690. https://doi.org/10.1016/j.scitotenv.2023.166690