

Assessment of Organochlorine Pesticide Residues on *Oreochromis niloticus* and Sediments in River Galma, Kaduna State, Nigeria

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Abstract

Pesticides usage in agricultural fields to control pests could be extremely toxic to non-target organisms like fish and affects fish health through impairment of metabolism, sometimes leading to mortality. The present study was carried out to assess the presence of organochlorine pesticide residues in sediment samples and *Oreochromis niloticus* obtained from River Galma. Soxhlet extraction process was employed for fish while sediment extraction was done using a mixture of n-hexane and acetone. The extracts were cleaned-up and analyzed using a gas chromatograph mass spectrometer (GC-MS). DDT, Mirex, Pentachlorophenol, Malathion, Chlordane and TNT were detected in the study area. Endrin, aldrin, dieldrin, endosulfan I, endosulfan II, alpha BHC and heptachlor were not detected in all the samples analyzed. Chlordane was the only detected pesticide in fish sample during wet season. The findings provide evidence of pollution of some of the organochlorine pesticide residues in River Galma. This shows that the River Galma is contaminated with organochlorine pesticides. Hence, serious need for continuous monitoring of these pesticide residues in water, sediments, fish and the environment is required in order to prevent various environmental and public health hazards that may arise from these pollutants.

Keywords: Organochlorines • Pesticides • Sediments • *Oreochromis niloticus* • River Galma

Introduction

Pesticides are compounds or mixes of substances used to prevent, eradicate, repel, or mitigate pests, which can include insects, mice, undesired plants (weeds), fungus, and microorganisms like bacteria and viruses. Pesticides include fumigants, fungicides, herbicides, rodenticides, and a variety of other compounds used to manage pests, while the phrase is frequently misconstrued to relate primarily to insecticides. Pesticides can be classified chemically based on functional groups in their molecular structures or their biological activity on target species [1]. Pesticides are frequently referred to by the type of pest that they can control. Another way to describe pesticides is to consider those that are chemical pesticides or are derived from a common source or production method. Other categories include biopesticides, antimicrobials, and pest control devices. Numerous studies on both humans and experimental animals show that pesticide residues have a high toxicity potential [2,3]. As a result, determining the risk of pesticides in environmental samples, foods, and dietary items is a key step and a critical tool in determining the risk of foodborne illness [4-6]. The hazardous consequences of pesticides on humans and the environment are a serious concern at local, national, regional, and global levels, and they are the basis for pesticide regulation, monitoring, and restriction in food [7]. A number of pesticides residues monitoring researches have been conducted on various samples, such as eggplant [8], drinking water from household wells and in human breast milk [9,10]. Organochlorines (OC) are a group of chlorinated compounds widely used as pesticides. These chemicals belong to the class of Persistent Organic Pollutants (POPs) with high persistence in the environment.

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Received 03 March, 2022, Manuscript No. jeat-22-57281; **Editor Assigned:** 05 March, 2022, PreQC No. P-57281; QC No. Q-57281; **Reviewed:** 17 March, 2022; **Revised:** 22 March, 2022, Manuscript No. R-57281; **Published:** 29 March, 2022, DOI: 10.37421/2161-0525.2022.12.647

OC insecticides were earlier successfully used in control of malaria and typhus, yet they are banned in most of the advanced countries [11]. The review statistics on the use of different pesticides shows that 40% of all pesticides used belong to the organochlorine class of chemicals [12,13]. Due to their low cost and the need against various pests, organochlorine insecticides such as DDT, Hexachlorocyclohexane (HCH), aldrin and dieldrin are among the most widely used pesticides in developing countries of Asia [12-14].

These pesticides are commonly used in agriculture to boost yield and avoid arthropod-borne illnesses. However, they are misused due to a lack of understanding of their applications and their side effects. The over consumption is damaging to eco-system as they damage soil, surface and under-ground water resources [15]. Nile tilapia *Oreochromis niloticus* [16] is native to Africa, ranging from the upper Nile River south to the equator and west to the Atlantic coast [17]. The species is favored among aqua culturists due to its ability to tolerate a wide range of environmental conditions, fast growth, reproductive strategies, and ability to feed at different trophic levels. These same characteristics enable them to be a highly effective invasive species in both subtropical and temperate climates [18].

Methodology

Study area

Zaria, a city in Kaduna State, Northern Nigeria is located at latitude 11°3'N and longitude 7°40'E. The city is about 128 km South-East of Kano and 64 km North-East of Kaduna, the State capital. The Galma river basin is a booming agricultural area. Crops are planted on both sides of the riverbank throughout the year. Fertilizers, herbicides, and insecticides are used on these crops, and surface runoff eventually washes them into the river [19,20]. The river is a major source of water supply to a number of communities located along its course (Figure 1).

Samples collection

Samples from the river, sediments and biota (fish) were collected at four different locations from the study area for a period of dry and wet season respectively. Each of the samples was prepared into four composite samples

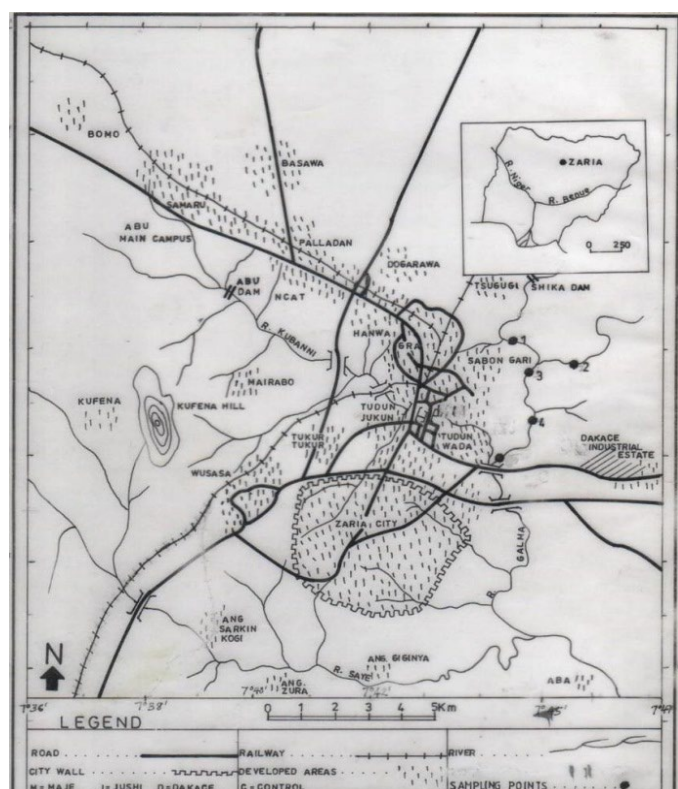


Figure 1. Map of River Galma showing the Sampling points.

each during the dry and wet seasons. The fish samples were collected using traps and nets and packed individually in plastic bags with eugenol, stored in a Styrofoam box with ice, and transported to the laboratory (Multiusers laboratory, chemistry department, Ahmadu Bello University Zaria) where they were stored at temperature of -20°C until ready for use. The fish samples collected were identified by a specialist from Department of biology Ahmadu Bello University, Zaria. The sediment samples were collected using the hand scooping PVC tubes. Sediment samples were collected at 2 and 8 cm depths each from different locations of the lake where there is high accumulation of sediments, mixed thoroughly to form a composite sample and transported to the laboratory (Multiusers Laboratory, Chemistry Department, Ahmadu Bello University, Zaria) as soon as possible under temperature-controlled conditions to prevent pesticides degradation. In the laboratory, sediment cores were weighed dried at 40°C until constant dry weight samples were obtained. Samples were then homogenized using agatha mortar and pestle and sieved with 0.5mm mesh sieves and the samples were stored at 4°C until further analysis.

Extraction of fish samples

The frozen composite whole-body of fish tissue samples were homogenized using an agate mortar and pestle. Approximately 20 g of the properly chopped fish sample was further mixed with 10 g of anhydrous sodium sulphate. This was Soxhlet extracted for eight hours using dichloromethane/n-hexane mixture. The extract was dried over anhydrous sodium sulphate and concentrated to about 2 mL as described earlier in readiness for the clean-up procedure.

Extraction of sediment samples

Dry Sediment sample was extracted according to Darko G, et al. [21], with some slight modifications. About 10 g of sediment samples were weighed and transferred into extraction thimble that had been previously washed with n-hexane and acetone and oven dried. The sample was extracted using 100mL of n-hexane acetone mixture 4:1 v/v for eight hours using Soxhlet extractor. The extract was evaporated to dryness using a rotary evaporator at 45°C . Each extract was dissolved in 10 mL n-hexane and subjected to clean-up procedure.

Clean-up procedure

A column of about 15 cm (length) $\times$ 1 cm (internal diameter) was packed

with about 10 g activated silica gel prepared in a slurry form in nhexane. About 10 g of anhydrous sodium sulphate was placed at the top of the column to absorb any water in the sample or the solvent. The column was pre-eluted with 20 mL of nhexane without the exposure of the sodium sulphate layer to air. The reduced extract was placed in the column and allowed to sink below the sodium sulphate layer. Elution was done with 2×10 mL portions of the extracting solvent (DCM). The eluate was then collected, dried with anhydrous sodium sulphate and then evaporated to dryness under a stream of analytical grade nitrogen (99.999%). The dried eluate above was then reconstituted with 1mL spectra grade nhexane and 0.5 mL of 20mg/kg mixture of organochlorines pesticides we added as an internal standard. $1.0 \mu\text{L}$ of the mixture was injected into the GC-MS column for analysis. Organochlorine Pesticide Mix AB #2 reference standard was obtained from Restek, USA. The modern Shimadzu GC-MS QP-2010 was employed in analyzing the standards and the calibration curve for each organochlorine compound was prepared automatically. The sample extracts were then analyzed under the same conditions as for the standards, and in the Selective Ion Mode (SIM) with m/z values ranging from 65 to 410. Splitless injection mode was used with the injection temperature as 250°C while the column oven temperature was ramped between 80 and 280°C . The GC-MS was operated at a pressure of 79.5 kPa and the flow rate was 1.18 mL/min. Method validation for this study maintained a RSD $\pm 13\%$ and percentage recoveries were in the range of 85 -115%.

GC-MS conditions

The GC-MS model QP-2010 (Shimadzu) with splitless/split injector and equipped connected to Restek Stx-CL pesticide column (Length 30 m, 0.25 mm I.D, 0.25 μm thickness) was used to identify the target OCPs. An energy of 70 eV was used to generate the mass spectra by monitoring ions (m/z = 5 - 450) in full-scan and selected recording mode (SIM). Injection volume and temperature $1 \mu\text{L}$ and 250°C respectively. Ion and interface source temperature were 200°C and 250°C respectively, injection and detection temperatures were adjusted to 250°C . The column temperature was set at 60°C and increased immediately at the rate of $10^{\circ}\text{C}/\text{min}$. to 180°C where it was held for 2 min., this was increased by $15^{\circ}\text{C}/\text{min}$., to 280°C , and held finally for 4 min.

Results

The molecular mass, percentage area and retention time of various chlorinated pesticides in sediments and fish samples obtained from river Galma during dry and wet seasons are summarized in Tables 1 and 2 respectively. Molecular Mass, percentage area and retention time of the detected OCPs are as follows. Molecular Mass (kg/mol): DDT; 354, Malathion; 330, Pentachlorophenol; 266, Chlordane; 410, TNT; 227, Mirex; 546, Percentage area (%): DDT; 1.15, Malathion; 8.98, Pentachlorophenol; 21.19, Chlordane; 1.50, TNT; 0.61, Mirex; 0.95. Retention time(s): DDT; 19.369, Malathion; 18.317, Pentachlorophenol; 19.786, Chlordane; 29.153, TNT; 12.877, Mirex; 29.176. Out of all the OCPs detected, only Chlordane was detected on fish sample during the wet season and none was detected on fish samples during the dry season (Tables 1 and 2).

Figures 2-7 are the chromatograms of the various detected organochlorine pesticides during dry and wet season respectively, Figures 2-4 are the chromatograms of OCPs detected during dry season, while Figures 5-7 are the chromatograms of the OCPs detected during wet season. The peaks of the chromatograms show the molecular mass of the detected OCPs which are as follows: DDT; 354, Malathion; 330, Pentachlorophenol; 266, Chlordane; 410, TNT; 227, Mirex; 546.

The Chromatogram of the detected OCPs during dry season and Figures 5-7 showing the detected OCPs during wet season (Figures 2-7).

Discussion

Some organochlorine pesticides were detected in sediment and fish samples during the study. DDT, malathion, Pentachlorophenol, were detected in sediment samples during the dry season, TNT, and mirex were detected in

Table 1. The molecular mass, percentages area and retention time of organochlorines in sediments and fish found in river Galma in dry season.

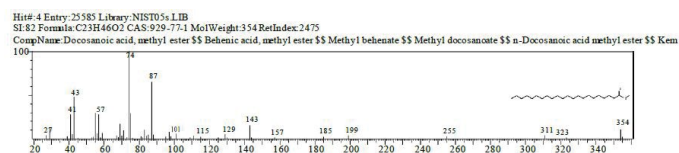
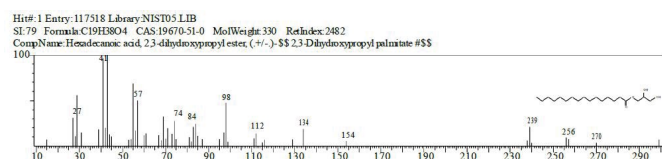
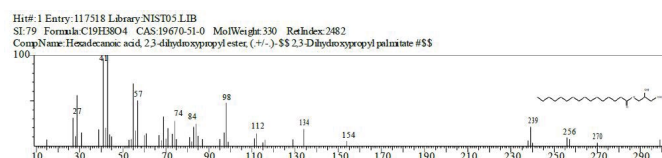
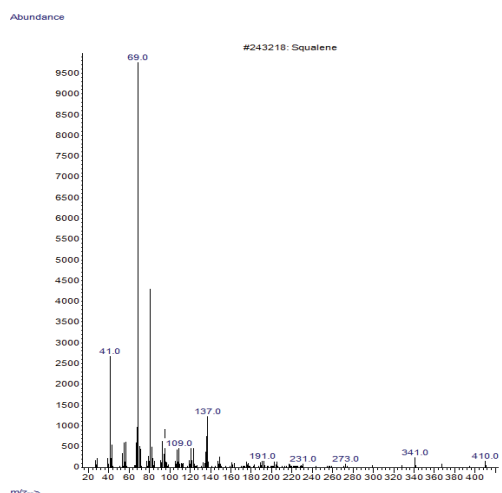
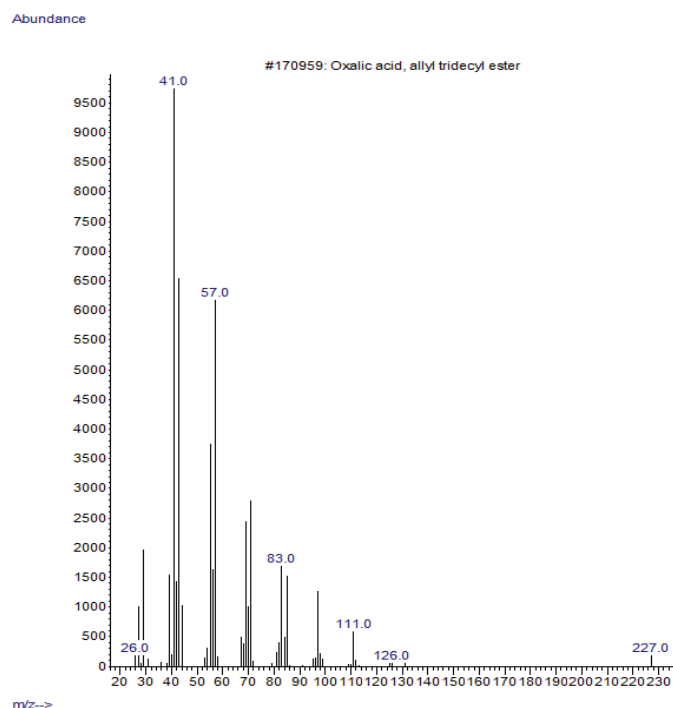
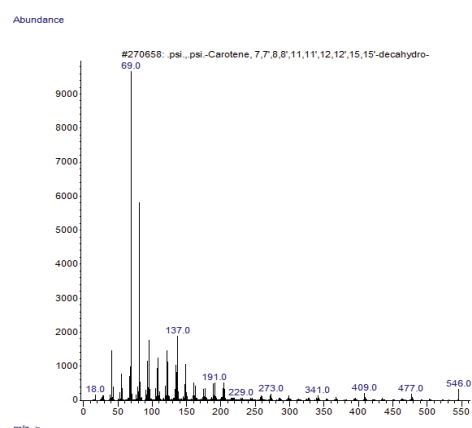
Pesticides	Molecular Mass	Area (%)	Retention Time	Fish Samples	Soil Samples
DDT	354	1.15	19.369	ND	D
Malathion	330	8.98	18.317	ND	D
Chlordane	ND	ND	ND	ND	ND
TNT	ND	ND	ND	ND	ND
Mirex	ND	ND	ND	ND	ND
Pentachlorophenol	266	21.19	19.786	ND	D

ND: Not Detected; D: Detected.

Table 2. The molecular mass, percentage area and retention time of organochlorines in sediments and fish found in river Galma in wet season.

Pesticides	Molecular Mass	Area (%)	Retention Time	Fish Samples	Soil Samples
DDT	ND	ND	ND	ND	ND
Malathion	ND	ND	ND	ND	ND
Chlordane	410	1.5	29.153	D	D
TNT	227	0.61	12.877	ND	D
Mirex	546	0.95	29.176	ND	D
Pentachlorophenol	ND	ND	ND	ND	ND

ND: Not Detected; D: Detected.

**Figure 2.** Chromatogram of the OCPs (DDT) detected on sediment sample during the dry season.**Figure 3.** Chromatogram of OCPs (Malathion) detected on sediment sample during the dry season.**Figure 4.** The Chromatogram of OCPs (pentachlorophenol) detected on sediment sample during the dry season.**Figure 5.** Chromatogram of OCPs (Chlordane), detected on Nile tilapia (Oreochromis niloticus) during the wet season.**Figure 6.** Chromatogram of OCPs (TNT) detected on sediment sample during the wet season.**Figure 7.** Chromatogram of OCPs (Mirex) detected on sediment sample during the wet season.

sediments during the wet season, while chlordane was detected on fish sample during the wet season. The presence of these organochlorine pesticides agrees with studies carried out by Lanfranchi AL, et al. [22] which reported that fish are suitable indicators for environmental pollution monitoring because they concentrate pollutants in their tissues directly from water and also through their diets. This research has clearly proved that some of these organochlorine pesticides are still been used in the study area. The presence of DDT in River Galma may be due to the high used of this chemical in controlling mosquitoes in the surrounding environments. DDT is a persistent.

Organic pollutant that is readily adsorbed to soils and sediments which can act both as sinks and as long-term sources of exposure contributing to terrestrial organisms. Depending on conditions, its soil half-life can range from 22 days to 30 years [23]. Due to hydrophobic properties, in aquatic ecosystems DDT and its metabolites are absorbed by aquatic organisms like fish and can be adsorbed on suspended particles, leaving little DDT dissolved in the water itself. Its breakdown products and metabolites, DDE and DDD, are also highly persistent and have similar chemical and physical properties [24]. Because of its lipophilic properties, DDT has a high potential to bio-accumulate especially in predatory birds. DDT, DDE, and DDD magnify through the food chain, with apex predators such as raptor birds concentrating more chemicals than other animals in the same environment [25].

Conclusion

The result of this study has shown that; Sediments samples obtained from River Galma are slightly contaminated with some organochlorine pesticides residues. *Oreochromis niloticus* obtained from River Galma are also slightly contaminated with some organochlorine pesticide residues.

Recommendations

River Galma is contaminated with organochlorine pesticides, there is therefore serious need for the continuous monitoring of these pesticide residues in water, sediments, fish and the environment, as this will go a long way towards mitigating various environmental and public health hazards. Further researches should be carried on other pesticides such as Organophosphates and other fish species.

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How to cite this article: Ndagi, Abubakar Mohammed, Umar Bayero, Adesakin Taiwo Adekanmi, and Yusuf Abdullateef, et al. "Assessment of Organochlorine Pesticide Residues on *Oreochromis niloticus* and Sediments in River Galma, Kaduna State, Nigeria". *J Environ Anal Toxicol* 12 (2022): 647.