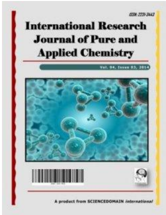

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Determinatiion of Organochlorine Pesticides Residue in Fish, Water and Sediment in Lake Geriyo Adamawa State Nigeria

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Authors' contributions

This work was carried out in collaboration between all authors. Authors DYS, ONM and JTB designed the study and wrote the protocol, preformed the statistical analysis, managed the literature search and wrote the first draft of the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Pesticides usage in agricultural fields to control pests is extremely toxic to non-target organisms like fish and affects fish health through impairment of metabolism, sometimes leading to mortality. Present study was carried out to determine the level of organochlorine pesticides residue in, water, sediments and two species of fish in Lake Geriyo. Soxhlet extraction process was employed for fish and sediment extraction using a mixture of hexane and acetone while water sample was extracted using dichloromethane liquid- liquid extraction method. The extracts were cleaned-up and analyzed using a gas chromatograph mass spectrometer (GC-MS). Endrin, aldrin, dieldrin, endosulfan I, endosulfan II, alpha BHC heptachlor and DDT were detected in the study area. While chlordane, methoxychlor, other isomers of BHC, DDT and their degradation products were not detected in all the samples analyzed. Dieldrin had the highest value of 0.566 mg/Kg in catfish while the lowest dieldrin value of 0.001 mg/Kg was found in Tilapia. The findings provide evidence of pollution of

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some the organochlorine pesticides residue in Lake Geriyo, this shows that the lake is slightly contaminated with organochlorine pesticides. Hence serious need for continuous monitoring of these pesticide residues in water, sediments, fish and the environment is required to in order to prevent various environmental and public health hazards that may arise from these pollutants.

Keywords: Organochlorines; pesticides; sediments; water; fish; lake geriyo; pollution.

1. INTRODUCTION

Organochlorine pesticides are persistent organic pollutants which have caused worldwide concern as one of the most toxic environmental pollutants [1]. They are lipophilic, hydrophobic and are ubiquitous contaminants which have been detected far from their sources of origin because of their persistency in nature, long-range transport stemming from atmospheric exchange, water currents and debris, animal migration and other anthropogenic pathways [2]. Organochlorine pesticides namely aldrin, dieldrin, endrin, chlordane, dichlorodiphenyl-trichloroethane (DDT), heptachlor, mirex, toxaphene and hexachlorobenzene (HCB) are among the twelve chemical substances called "dirty dozen" and are defined under the Stockholm Convention [3]. The manufacture and use of some pesticides have been banned or restricted in developed countries but some developing countries are still using organochlorine pesticides for agricultural and public health purposes. They are being used in developing countries such Nigeria due to lack of appropriate regulatory control and management on the production, trade and use uncontrolled of these chemicals.

However, this chemical and several other chemical contaminants from the agricultural fields, comprising of pesticides and other agrochemicals have been reported in the drainage systems, dam reservoirs and lakes which are likely to jeopardize the quality of the water bodies that support the fishery industry, irrigation, industrial, domestic and human consumption [4].

Africa has therefore been suggested as a highly relevant area for increased research activities to establish possible links between the increasing number of unexplained cancer cases and exposure to anthropogenic chemical pollutants in food, air, water, sediments and soil [5]. Some developing countries are still using organochlorine pesticides for agricultural and public health purposes to prevent the spread of mosquitoes [6]. They are being used in developing countries such as Nigeria due to lack of appropriate regulatory control and management on the production, trade and use of these chemicals [7]. Organochlorine pesticides such as DDT are still frequently in use all over Africa both as a means for effective and cheap vector control health and for agricultural purposes [8]. One of the most controversial pesticides of all time, dichlorodiphenyl-trichloroethane (DDT) is today even being introduced on a broader scale to defeat malaria, despite being banned in most countries of the world [9].

From an African perspective this might be understandable, since malaria still is a tremendous problem killing one child every 30 minutes in sub-Saharan Africa [10]. Many ignorant farmers, fishermen and some other have abused their use for agricultural and fishing purposes [11]. Chlorinated pesticide residues can enter aquatic environment through effluent release, discharges of domestic sewage and industrial wastewater, atmospheric deposition, runoff from agricultural fields, leaching, equipment washing, and disposal of empty containers and direct dumping of wastes into the water systems [12]. Pesticide residues could distribute among the components of an ecosystem, such as water sediment and fish. Due to their persistence, pesticides in water and sediments can be transferred into the food chain and accumulate in aquatic organisms like zooplanktons and fish [13]. Sediment is one of the principal reservoirs of environmental pesticides, representing a source from which residues can be released to the atmosphere, groundwater and living organisms [5]. Persistence of these organic pollutants in sediment is possible due to their low solubility and tendency to associate with suspended particulate matter. Due to their low water solubility, organochlorine pesticides have a strong affinity for particulate matter. They are hydrophobic compounds that tend to adsorb to suspended particulate matter and benthic sediments in aquatic ecosystems. Sediments serve as ultimate sinks for them. Indirect exposure to contaminated sediments takes place when fish feeds on benthic invertebrates that are ingesting particulate matter. Direct exposure through the sediment takes place by release of contaminated particulate matter into the water column by both natural and anthropogenic processes [4].

The health effects from uncontrolled pesticides usage may result from short or long-term exposure, low or high-level exposure through dermal absorption, inhalation and oral ingestion. Some of the symptoms of pesticides poisoning include irritation, dizziness, tremor, tonic and chronic convulsion [14]. Persistent organic pollutant (POP) contamination is usually monitored by measuring levels either in inorganic ecosystem compartments such as water, air and sediment or in biota. The quality of fish, sediment and water from Lake Geriyo must be constantly monitored. Fishes are suitable indicators for environmental pollution monitoring because they concentrate pollutants in their tissues directly from water and also through their diet [15]. Acute (immediate) toxic effects can influence the survival or reproduction of aquatic species leading to the disruption of predator-prey relationships and a loss of biodiversity. If aquatic organisms are not harmed immediately, they may accumulate chemicals from their environment into their tissues. This bio-concentration can lead to bio-magnification, a process in which the concentrations of pesticides and other chemicals are increasingly magnified in tissues and other organs as they are transferred up in the food chain [4]. There is a need to monitor the concentrations of chlorinated pesticides in pelagic columns in order to assess the level of compliance with the POPs Stockholm convention in developing countries such as Nigeria. Given the potential for contamination by pesticides on the environment, therefore, this study was undertaken to determine the level of organochlorine pesticide residues in water, sediments and two species of fish in Lake Geriyo, Jimeta-yola, Adamawa State.

2. MATERIALS AND METHODS

2.1 Study Area

Lake Geriyo is situated in Jimeta-Yola Adamawa state capital and located on latitude 09°18’11” N and longitude 12°25’36” E. Lake Geriyo is the lake which is flooded by the River Benue. Lake Geriyo occupies natural depression near the upper Benue River in north eastern Nigeria. The lake is flooded by the river during the raining season spanning the months of May to September. The water from the lake is primarily used in intensive irrigation, fishing and source of water large cattle farmer in the areas shown in Fig. 1. According to the information from the head of the local fishermen, the lake was formed naturally from River Benue that was cut off as a result of heavy siltation about 60 years ago, thereby forming a small gully. The gully was later filled with water from the rains and flooding from River Benue. It is a shallow water body with a mean depth of about 2 metres. Aquatic vegetation on the lake consist of mass of flooding weeds such as water hyacinth, typha grass, water lily and wild guinea corn which move around the lake surface due to the prevailing winds [16].

2.2 Sample Collection

Samples of the environmental matrices-sediment, water and biota (fish) were collected at four locations from the study area for a period of January – May, July- October, 2013 and 2014 dry and wet season respectively. Each of the samples were prepared into five composite samples each during the dry and wet seasons for 2 years. Five fish sample were selected at least and packed individually in plastic bags with eugenol, stored in a Styrofoam box with ice, and transported to the laboratorywhere they were stored at temperature of -20°C until ready for use. The fish species collected were *Tilapia zilli* (Tilapia) and *chrysichthys nigrodigitatus* (Catfish).

They were identified by a specialist from Department of fisheries and Aquaculture Adamawa state university, Mubi. The sediment samples were collected using the triple sediment corer made by Uwitec fitted with plexiglas tubes of 50 cm height. Five sediment samples were collected at 2 and 8 cm depths each from different location of the lake, mixed thoroughly to form a composite sample and transported to the laboratory 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 0.5 mm mesh sieves and the samples were stored at – 4°C until further analysis.

Five water samples from Lake Geriyo were collected using 2 L amber glass bottles from different locations, mixed thoroughly to form a composite sample for replicate analysis. The composite water samples were pre-filtered through 0.45 Im fiber glass filters (whatman) to remove suspended materialand then preserved by the adding of concentrated H_2SO_4 to prevent biological activity [16].

$$H_2SO_4\,(aq) + H_2O\,(l) \longrightarrow H_3O^+(aq) + HSO_4^-\,(aq).$$

The samples were preserved by refrigeration at -10°C until analysis was undertaken.

2.2.1 Extraction of water samples

Liquid-liquid extraction method was used to extract organochlorine pesticides from the 2 L water samples earlier collected as described by Shinggu et al. [4] with some slight modifications. About 500 mL of water sample was put into a 1 L separatory funnel at a time. This was extracted with 30 mL (3 portions) of dichloromethane DCM by vigorous shaking for 45 minutes for each of the triplicate extraction. The combined extracts were dried with anhydrous Na_2SO_4 and concentrated to about 2 mL under a pure stream of nitrogen of 99.999% purity.

2.2.2 Extraction of sediment samples

Dry Sediment sample was extracted according to Darko et al. [17], 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 100 mL 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.

2.2.3 Extraction of fish samples

The frozen composite whole-body of fish tissue samples were homogenized using an agate mortar and pestle [18,19]. 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.

2.2.4 Clean-up procedure

A column of about 15 cm (length) × 1 cm (internal diameter) was packed with about 10 g activated silica gel prepared in a slurry form in n-hexane. 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 n-hexane 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 1 mL spectra grade n-hexane and 0.5 mL of 20 mg/kg mixture of organochlorines pesticides we added as an internal standard. 1.0 µL 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/m. Method validation for this study maintained a RSD ±13% and percentage recoveries were in the range of 85 -115%..Data obtained were subjected to analysis of variance (ANOVA) using SPSS version16 to determine the differences in the concentration of each of the organochlorine pesticides residue in each sample analyzed.

3. RESULTS AND DISCUSSION

The level and distribution of various chlorinated pesticides in water, sediment and two species of fish found in Lake Geriyo during dry and wet seasons are summarized in Tables 1 & 2 respectively. Some organochlorine pesticides were detected in water, sediment and fish samples, only two pesticides residue alpha BHC and Aldrin were detected in water during the dry and wet seasons with concentration of the pesticides detected low than that of dry season. Dieldrin pesticide residue had the highest value of 0.566 mg/Kg and 0.456 mg/Kg in *chrysichthys nigrodigitatus* (cat fish) samples analyzed during dry and wet seasons respectively. Alpha BHC, Endosulfan I&II, lambda-cyhalothrin, Dieldrin and pp' DDT were all detected in Catfish but in Tilapia only.

Dieldrin and Endosulfan I in Tilapia were detected during both seasons with concentration lower in wet season. The lower concentration of pesticides residue observed in wet season may be due to dilution since the volume of water in the lake increases during the wet season. This, by implication, means that the fish samples were more contaminated with OCPs during the dry season than in the rainy season. The higher dry season levels of OCPs in fish generally might be due accumulation of the pesticides in the lake and their feeding habit during dry season some fish feed more on insect larvae, coarse vegetable matters and sediment-associated particles that had accumulated the OCPs overtime [11].

Aldrin was detected in water and sediment only while dieldrin was detected in Tilapia and Catfish only and endrin was detected only in sediment Tables 1 and 2, but heptachlor was detected only in Catish during dry season Table 1. The value of endrin in this study is low than that reported by Shinggu et al. [4], and higher than those reported by Williams [20].

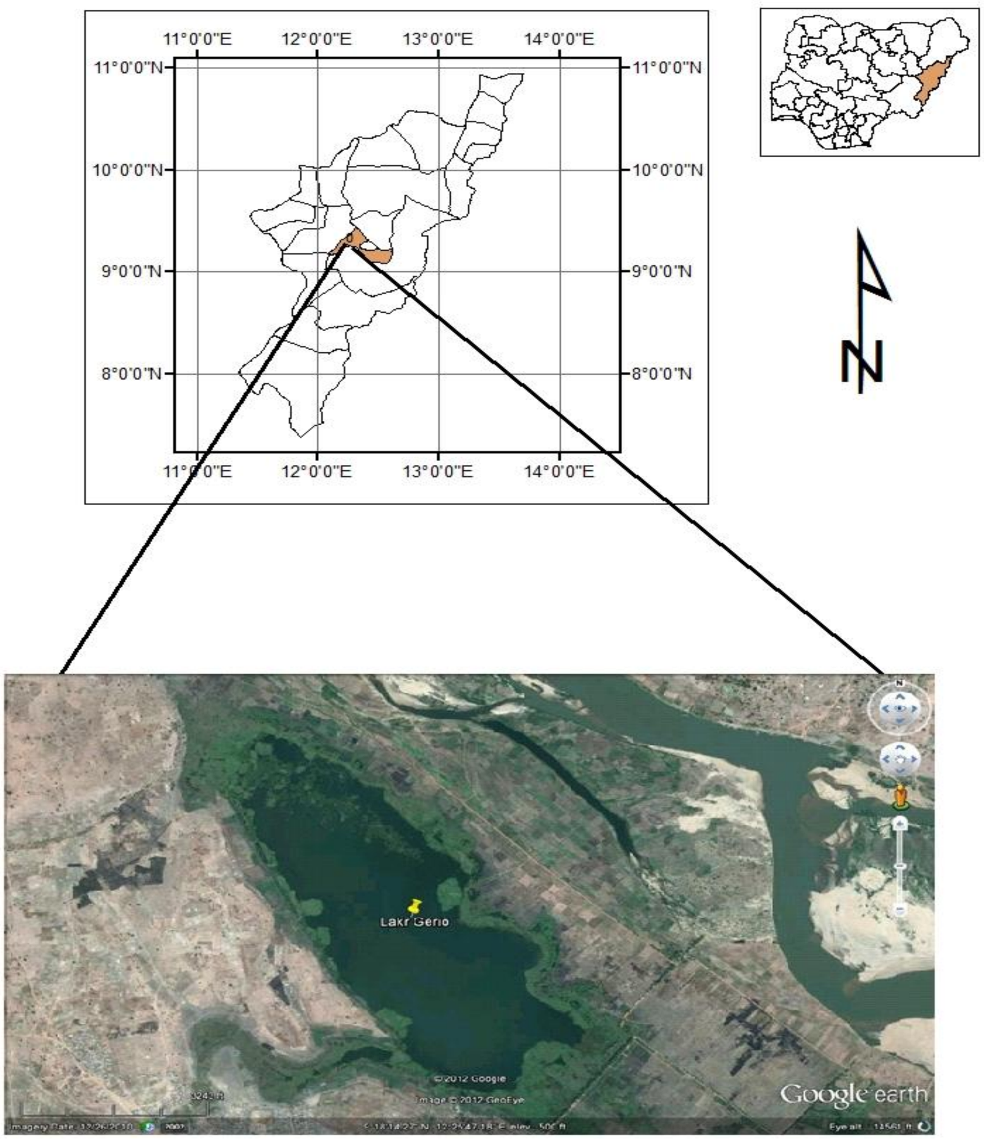

Fig. 1. Map of Adamawa state showing the location of Lake Geriyo

Endosulfan II was detected in sediment and Catfish while endosulfan I was detected in both fish sample. Endosulfan has been shown to be highly toxic to fish and marine invertebrates and is readily absorbed in sediments. It therefore represents a potential hazard in the aquatic environment [21]. The value of endosulfan in this study 0.086 mg/L in fish is higher than study carried out by Idowu et al. [22] and Shinggu et al. [4].

The presence of these organochlorine pesticides agrees with studies carried out by Lanfranchi et al. [15], 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 uncontrolled in the study area since aldrin, dieldrin and endrin are used as a soil insecticide to control root worms, beetles, and termites which are common pest in in the study area over the years. Dieldrin has been used in agriculture for the treatment of soil and seed and in public health to control disease vectors such as mosquitoes and tsetse flies [23]. The level of aldrin and dieldrin in this study is higher than those of fish reported in Allau dam reservoir by Akan, et al. [23].

DDT was detected on in Catfish with concentration of 0.043 mg/Kg, the result of this study suggest a higher value as compared to studies carried out by Clark et al. [24].

This high concentration of DDT in lake Geriyo may due high used of this chemical in controlling mosquitoes since lake Geriyo is situated in the state capital with high population density. Municipal waste from the town may flow into the lake and accumulate in water and sediment. 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 [25]. Depending on conditions, its soil half –life can range from 22 days to 30 years [25]. Routes of loss and degradation include runoff, volatilization, photolysis and aerobic and anaerobic biodegradation. 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 [26]. 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 [27].

Table 1. Shows the Mean±SD level of Organochlorines (mg/kg) in water, sediment, and Fish found in Lake Geriyo in dry season

Pesticides	Samples			
	Water (mg/L)	**Sediment (mg/Kg)**	**Tilapia (mg/Kg)**	**Catfish (mg/Kg)**
Gamma BHC	ND	ND	ND	ND
beta-BHC	ND	ND	ND	ND
alpha BHC	0.290±0.001a	ND	ND	0.033±0.002a
Endosulfan I	ND	ND	0.024±0.001a	0.086±0.009a
Endosulfan II	ND	0.043±0.001a	ND	0.086±0.003a
Heptachlor	ND	ND	ND	0.001±0.000
Aldrin	0.333±0.002a	0.102±0.002a	ND	ND
Dieldrin	ND	ND	0.001±0.000a	0.566±0.062b
Endrin	ND	0.007±0.000a	ND	ND
Lambda-Cyhalothrin	ND	ND	ND	0.372±0.043a
Gamma-chlordane	ND	ND	ND	ND
p,p'-DDT	ND	ND	ND	0.043±0.001a
pp'-DDE	ND	ND	ND	ND
DDD	ND	ND	ND	ND
Methoxychlor	ND	ND	ND	ND

All values represent Mean±SD (Standard Deviation). Comparison was done across the period and values with different Superscripts are statistically different ($p < 0.05$). ND: Not Detected

Table 2. Shows the Mean±SD level of Organochlorines (mg/kg) in water, sediment, and Fish found in Lake Geriyo in wet season

Pesticides	Samples			
	Water (mg/L)	Sediment (mg/Kg)	Tilapia (mg/Kg)	Catfish (mg/Kg)
Gamma BHC	ND	ND	ND	ND
beta-BHC	ND	ND	ND	ND
alpha BHC	0.190±0.002a	ND	ND	0.033±0.002a
Endosulfan I	ND	ND	0.022±0.001a	0.083±0.009a
Endosulfan II	ND	0.041±0.001a	ND	0.087±0.003a
Heptachlor	ND	ND	ND	ND
Aldrin	0.313±0.001a	0.102±0.002a	ND	ND
Dieldrin	ND	ND	0.001±0.000a	0.465±0.072b
Endrin	ND	0.007±0.000a	ND	ND
Lambda-Cyhalothrin	ND	ND	ND	0.342±0.033a
Gamma-chlordane	ND	ND	ND	ND
p,p'-DDT	ND	ND	0.012±0.000a	0.043±0.001a
pp'-DDE	ND	ND	ND	ND
DDD	ND	ND	ND	ND
Methoxychlor	ND	ND	ND	ND

All values represent Mean±SD (Standard Deviation). Comparison was done across the period and values with different Superscripts are statistically different (p < 0.05). ND: Not Detected

Commercial insecticide hexachlorocyclohexane HCH sometimes referred to as benzene hexachloride BHC is a mixture of different isomers mainly α, β and γ-HCH (Lindane). Other isomers in the group are ∂ and ε. Lindane (γ -HCH) has been used as an insecticide and is the most toxic. β - HCH is the most symmetric and stable isomer; it is also the most persistent in nature [28].

β-HCH is eliminated five times more slowly from the body than other isomers and has a much higher ability to accumulate in the fat tissue than lindane [28,29]. This may likely be the reason why some of this isomers were not detected in all the samples analyzed except α- BHC in Catfish and water. The remaining two isomers β and γ -BHC were not detected in the in all the samples analyzed in the lake. The non-detection of some of these pesticides shows that they are not in use in the surroundings of the lake which implies that the banned of these pesticides usage is being enforced by NAFDAC in Nigeria. Lindane is also rapidly degraded in the environment [28], this explains the reason why γ -BHC in all the samples analysis were not detected. Lindane degradation may be as a result of high temperature in the study area since temperature increase the rate of formation or disappearance of a substance and Jimeta-Yola the Adamawa state capital use to experience the highest temperature in the Nigeria. Non detection may also be due to continue monitoring of these banned pesticides by regulatory agencies in such as NAFDAC Nigeria. The result of this study aggress with the study carried out by Idowu et al. [22]; Shinggu et al. [4] and Williams [20], which reported the organochlorine pesticide residue levels in river water and sediment from cocoa-producing areas of Ondo State, water, sediment and fish in Biu dam Borno state, and lagoons in Lagos state respectively. Statistical analysis by ANOVA showed no significant differences (P < 0.05) between the pesticide residues as indicated in Tables 1 & 2. This suggests that, most of the indicated pesticides in Lake Geriyo originate from agricultural runoff and probably mosquitoes control program in the state may be the sources of other pesticides detected in the study area.

4. CONCLUSION

The result of this study has shown that water, sediment and fish samples from Lake Geriyo are slightly contaminated with some organochlorine pesticides residue in varied degrees in both dry and wet seasons with concentration high in dry season. Higher levels of OCPs were recorded in fish, however, higher levels of OCPs were found in the fish samples in the dry season than during the rainy season. This may be possible due feeding habit and bioaccumulation tendency of OCPs in the fish species during the dry season. Generally, the seasonal variation of OCPs in the fish samples were higher in most cases, than the recommended WHO Maximum Residue Levels (MRLs) of 0.05 mg/Kg in food items and should give cause for concern. The occurrence of the OPCs in fish sediments and water from Lake Geriyo is consistent with the agricultural activities of the study area due to pesticide usage by the farmers and municipal waste from drainages and runoff that flows in the lake. Lake Geriyo is slightly 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.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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