



ORIGINAL ARTICLE

Assessment of Organochlorine (OC) Pesticides Residues in Sediment, Soil, and Fish Samples from River Owan, Edo State, Nigeria

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KEYWORDS

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ABSTRACT: In this study, sixteen (16) organochlorine (OC) pesticides in sediment, soil and fish (*Clarias anguillaris*) samples of River Owan, Edo State, Southern Nigeria were investigated in accordance with acceptable analytical methods. Our findings confirmed the presence of the 16 OCPs in different concentration values in soil, sediment, and fish (*Clarias anguillaris*). The Σ OCP in the sediments samples were between 0.0033-0.0319 (ng g⁻¹) with total value of 0.1506 (ng g⁻¹) for the sediment sample 1 (SE1); 0.0021-0.0587(ng g⁻¹) with total value of 0.0291(ng g⁻¹) for the sediment sample 2 (SE2). The level of the OCPs in the soil ranged between 0.0069-0.0626 (ng g⁻¹), total concentrations of 0.3986(ng g⁻¹) for soil sample 1 (SO1); 0.0026-0.0630 (ng g⁻¹), total concentration of 0.3678 (ng g⁻¹) for soil sample 2 (SO2). The concentration level of the OC pesticides were between 0.0046-0.1707 (ng g⁻¹) in the fish sample with total concentration of 0.1490 (ng g⁻¹). These pesticides were found in higher concentrations in soil than in sediment samples. The level of OC pesticide residues detected in the fish was greater than the sediment samples. The hazard quotient is moderate (1.49) when compared with the guidelines.

INTRODUCTION

Organochlorine pesticides (OCPs) are synthetic pesticides which are generally used all over the world. OCP is one of the persistent organic pollutants (POPs) with a global environmental problem [1-3]. They are members of the group known as chlorinated hydrocarbon derivatives, with major application in the agricultural sector and chemical industry. OCPs have been successfully utilized to reduce crop damage caused by insects and diseases, as well as to control weeds and increase crop output all over the world.

The public is informed of the hazardous consequences of these substances despite the benefits they provide [1, 4 - 6]. Despite the fact that their manufacturing, use, and management have been restricted or prohibited in developing and developed nations, their usage is still on the increase [7 - 10].

These chemical compounds are recognized for their persistency, severe toxicity, degradation (slow) including bioaccumulation in the ecosystem [8, 11 and 12]. Despite

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the fact that pesticides are designed towards specific organism toxicity, non-target organisms are negatively impacted [8, 5].

Pesticides applied on agricultural soils are transported by various means: soil, air, moving water or rain into rivers. Upon entering into the river they interact with various types of materials including water, sediments, suspended matter, and organisms to undergo series of transitions into different compounds making them becoming a hazard to both human and the wildlife [4, 12 - 14]. OCPs may impact the proper operation of the endocrine system in humans and wildlife, according to studies, with increasing endocrine and neurological damage, with cancer, and birth problems as a result of exposure [5, 7, 8, and 11].

Many researchers have worked on OCPs in Nigeria and around the globe. Laigi, *et al.* [11], researched on the OC pesticides in the soils from India, and Honghu, *et al.* [15] reported OCPs in surface waters (Qingshitan Reservoir) Southwest China, both publications were looking at ecological risk assessment, spatial distribution, and human health. Akoto *et al.* [16] and Akan [18, 19] studied pesticide residues in several matrices, looking at their

levels and distributions. Ogbeide *et al.* [17] assessed the risk associated with agricultural pesticides from Owan River, Nigeria. Bolor *et al.* [20] reported OCPs residues in vegetables from Ghana. Teklit [21] and Adeboyejo *et al.* [22] carried out OC pesticide residues in aquatic specimens including sediment and water.

There has been no data on the amounts of organochlorine (OCP) pesticide residues in soil surrounding Owan, which is one of the major sources of pesticides in the River, necessitating this study. As a result, the goals of this preliminary investigation are to quantify organochlorine (OC) pesticides in soil, fish, and sediment samples from the River Owan, Edo State, Nigeria.

MATERIALS AND METHODS

The Research Area

River Owan community is located between latitudes 5°30' N and 6° 30' N, and longitudes 7° 30' E and 7° 30' E [17] with a prominent river River Owan in Edo State, Nigeria (Figure 1). The climate in this area is tropical, having rainy (April–October) and dry (November–March) seasons.

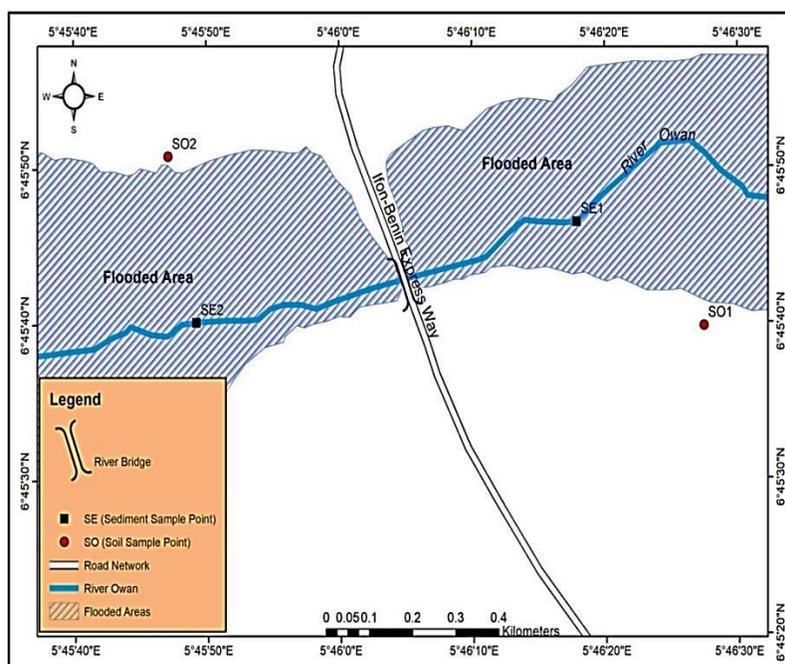


Figure 1. The study region and sampling stations are depicted on the map.

The river is utilized for a variety of purposes by the communities, including fishing, washing, transportation, cooking, bathing, swimming, and drinking. Typically, residents living near the river discharge a variety of household garbage into the river. The river flows through Okpokhumi, Sabongidda, and the River Ose before emptying into the latter. The area is known for extensive agriculture ranging from plantain, cassava and cocoa plantations, with many of these fields located along the river's edge.

Sampling Period

The sampling took place in February of year 2019 (during the dry season).

Sediment sampling

A sediment grab sampler was used to collect two sediment samples. Two locations were sampled: upstream (sediment sample 1) and downstream (sediment sample 2). Extraneous materials such as stones, leaves, shell and so on were manually taken off. Sediment samples were collected in a labelled aluminium foil separately, kept in a cooler containing ice, and transferred to the laboratory where it was then kept in the refrigerator at 4°C.

Fish collection

A sample of catfish (*Clarias anguillaris*) was taken from the river in aluminum foil, tagged, and moved to the laboratory at 4°C in a chest cooler. The fish specimen was identified at Rufus Giwa Polytechnic Owo, Nigeria by an aqua-culturist in the Department of Fishery and Aquaculture.

Soil sampling

Two soil samples were taken from two agricultural sites using a soil auger (site 1 and site 2) shown on the map (Figure 1) as SO1 and SO₂ around the river at a depth of 0–30 cm. Collected soil samples were contained in aluminum foil and refrigerated at <4°C until analysis.

Sample preparation, treatment and extraction of sediment and soil

In the laboratory, experimented samples were dried in the open air at ambient temperature (soil and sediment), pulverized with a porcelain mortar and pestle (previously washed and cleaned with n-hexane), and sieved through a laboratory 2mm sieve. Extraction vials earlier dried free from organic was precisely weighed with ten grams (10g) of sample. Laboratory glass rod was used to homogenize ten grams (10g) of anhydrous sodium sulphate. The sample was mixed in a 3:1 ratio with twenty millilitres (20mL) of n-hexane and dichloromethane (90mL of hexane and 30mL of dichloromethane were made in a standard flask). For 30 minutes, the sample was shaken at 500osc/min in an organic flask shaker (Innova 2000 model). Ultrasonic method was used for the extraction. The silica gel method is used to fractionate one millilitre of the extract. Silica gel columns were used to clean the sample extract.

Fish sample preparation and treatment

The US EPA's standard analytical technique for OCPs [23] was applied. A laboratory warring blender 7010BU (previously washed and cleaned with hexane) was used to homogenize the frozen fish sample, along with 100 mL acetone as solvent. Homogenization of the sample for 20 minutes was done at 100 rpm before being combined with 5g of anhydrous sodium sulphate. In a Soxhlet extraction system, extraction was carried out for around 5 hours with dichloromethane and n-hexane. The resultant solvent was eluted with 50 mL n-hexane solvent and reduced to 1-2 mL by evaporating on a steam bath.

Determination of organochlorine (OC) pesticides

Concentration of OCPs in the different samples was done using Agilent 7890A gas chromatography, column Varian VF-5 Pesticide capillary column, 30m x 0.25um x 0.25mmID equipped with electron capture detector (GC/ECD) using standard analytical methods [23, 24].

Gas chromatography conditions

The carrier gas used was nitrogen. Inlet temperature was 250°C. Column flow rate was 2 mL. Detector temperature was 310°C. Injection mode: split less and injection volume: 1µL. Oven initial temperature was 100°C, ramp 1 was 200°C at 10°C/min hold 2min, ramp 2 was 300°C at 10°C/min hold 5min with a total run time of 28minutes.

Hazard Quotient

The hazard quotient, abbreviated as HQ, is the proportion of possible chemical exposure to the level at which no negative consequence is expected. Thus, HQ is calculated with this model by [25]:

$$HQ = \text{Dose Rate} / \text{Reference Dose}$$

The interpretation guidelines for HQ calculations given by [26] are:

Whenever HQ is less than 0.1 means no hazard exists, if HQ is between 0.1 and 1.0 means hazard is low, when HQ equals 1.1 – 10 means hazard is moderate and when HQ > 10 means that hazard is high.

Analysis of data

Data collected from the experiment were subjected to analysis using the Microsoft Excel for charts.

RESULTS AND DISCUSSION

Results of OC pesticides concentration levels obtained are presented in Table 1.

Table 1. The result of Organochlorine pesticides (OCPs) of samples.

S/N	Components	Soil sample 1 (ng g ⁻¹)	Soil sample 2 (ng g ⁻¹)	Sediment sample 1 (ng g ⁻¹)	Sediment sample 2 (ng g ⁻¹)	Fish sample (ng g ⁻¹)
1	α-BHC	0.00658899	0.00917037	0.00483613	0.00460131	0.00464826
2	β-BHC	0.0626182	0.0587649	0.0318674	0.0360356	0.00853905
3	Lindane	0.0606480	0.0629570	0.0285334	0.0329570	0.00853714
4	Chlorothalonil	0.0283191	0.0355270	0.0157886	0.0177559	0.0139416
5	δ-BHC	0.00819486	0.00731544	0.0109096	0.0127379	-
6	Heptachlor	0.00823266	0.00892066	0.0229141	0.0278204	0.00878064
7	Aldrin	0.0179319	0.0191598	0.0107604	0.0138046	0.00459682
8	Heptachlor epoxide	0.00629601	0.0100603	-	0.00640827	0.0308637
9	o, p – DDE	0.0195911	0.0255088	0.0109707	0.00914215	0.00999613
10	Endosulfan I	0.0101109	0.0225133	0.00332214	0.0124332	0.00623429
11	p, p – DDE	0.00738842	0.00810882	0.00525274	0.00577174	0.00829405
12	Dieldrin	0.0119471	0.00446493	0.00543665	0.00870575	0.0121635
13	Endrin	0.0294989	0.0138845	-	0.0168180	0.0179197
14	Endosulfan II	-	-	-	0.00296867	0.0145270
15	p, p- DDT	-	0.0426577	-	0.0434704	-
16	L- Cyhalothrin	0.0299212	0.0387327	-	0.0586601	-
Total		0.398595	0.367746	0.150592	0.0291265	0.149042

OC pesticides in soil

The results of OC pesticide components in soil samples are shown in Figures 2 and 3. The value of OCPs components in the soil samples: alpha Lindane, β-BHC, Lindane, Chlorothalonil, δ-BHC and Heptachlor were 0.0066 ng g⁻¹,

0.0626 ng g⁻¹, 0.0606 ng g⁻¹, 0.0283 ng g⁻¹, 0.0082 ng g⁻¹ and 0.0082 ng g⁻¹ respectively in SO1 while it was 0.0092 ng g⁻¹, 0.05881 ng g⁻¹, 0.0630 ng g⁻¹, 0.0355 ng g⁻¹, 0.0073 ng g⁻¹ and 0.0089

ng g⁻¹ respectively for the same pesticide components in soil sample 2. The concentration of aldrin, heptachlor epoxide, o,p-DDE, endosulphan I, p,p-DDE, dieldrin and endrin were 0.0179 ng g⁻¹, 0.0063 ng g⁻¹, 0.0196 ng g⁻¹, 0.0101 ng g⁻¹, 0.0074 ng g⁻¹, 0.0119 ng g⁻¹ and 0.0295 ng g⁻¹ for soil sample 1 while 0.0192 ng g⁻¹, 0.0101 ng g⁻¹, 0.0255 ng g⁻¹, 0.0225 ng g⁻¹, 0.0081 ng g⁻¹, 0.0447 ng g⁻¹ and 0.0139 ng g⁻¹ respectively for the same OCPs in soil sample 2.

Endosulfan II was not present in the soil samples. The concentration of L-Cyhalothrin was 0.0299 ng g⁻¹ for the first soil sample and 0.0387 ng g⁻¹ for the second soil sample. The absence of some OCPs might be due to the type of pesticides/herbicides (agrochemicals) used. The overall summation of OCPs in the soil sample from the upstream (soil sample 1) was $\sum 0.3986$ ng g⁻¹ and $\sum 0.3678$ ng g⁻¹ for the downstream soil sample (soil sample 2). From this, the total OCPs concentration in soil sample 2 was slightly higher than soil sample 1. There are many factors that could account for this, they include: frequency and dosage application of the pesticides, the characteristics of the agrochemical used, the season and so on. This result was similar to what was reported by Ayeisanni and Idowu [27], who researched on soil of some selected cocoa farms in Ondo State and found out that there were varying

concentrations in the samples analysed. According to Issa [28] and Mokwunye *et al.* [29] farmers across Ondo and Edo States use pesticides and agrochemicals of various brands to improve crop yield which finally find their way into the rivers in the areas. Ogbonoya *et al.* [3] reported varying concentrations (endosulfan II ranged from between 0.056±0.03mg kg⁻¹ – 0.049±0.02mg kg⁻¹, p,p'-DDT 0.296±0.04mg kg⁻¹ - 0.289±0.0mg kg⁻¹ while some such as lindane, heptachlor, dieldrin were not detected) of OCPs in soil samples from farming communities in Minna, Niger State, Nigeria. A research conducted by Lupi *et al.*, [30] carried out to determine the OCPs in agricultural soil and associated biota at Argentine Pampean region, the soil samples analysed revealed a differential pesticide patterns throughout the profile. The level of α -endosulfans (0.08±0.07 – 0.03±0.03 ng g⁻¹), heptachlor (0.10±0.06 – 0.0004 ng g⁻¹), heptachlor epoxide (0.02±0.01 – 0.08±0.12ng g⁻¹) and so on were recorded. The total concentration level of the OCPs in various soil samples ranged between 0.71– 0.18 ng g⁻¹. Intensive used of agrochemicals at these sites were also reported. The total concentration of OCPs in the soil analysed were lesser than others report by Weaver *et al.* [31] from Australia; Yu *et al.* [32] from China; Syed *et al.* [33] from Pakistan.

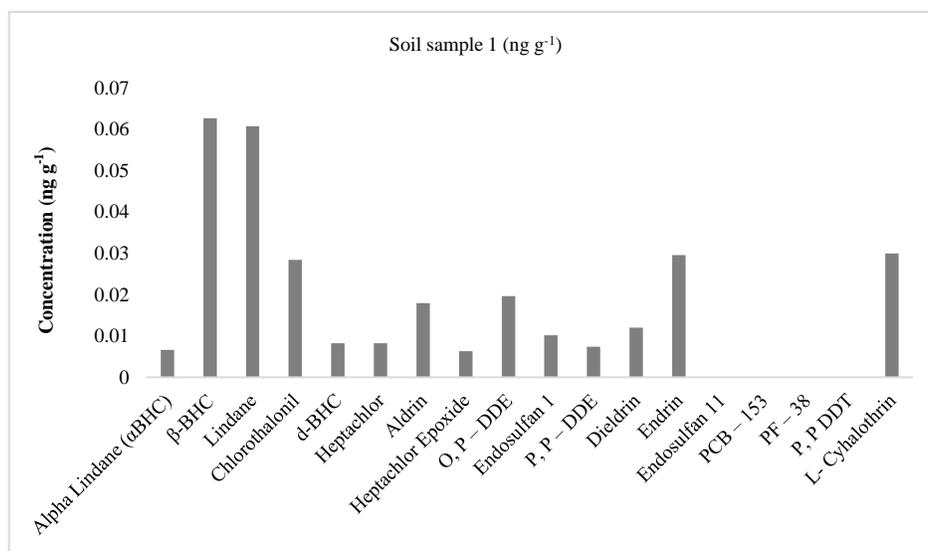


Figure 2. Concentration of OCP in soil sample 1.

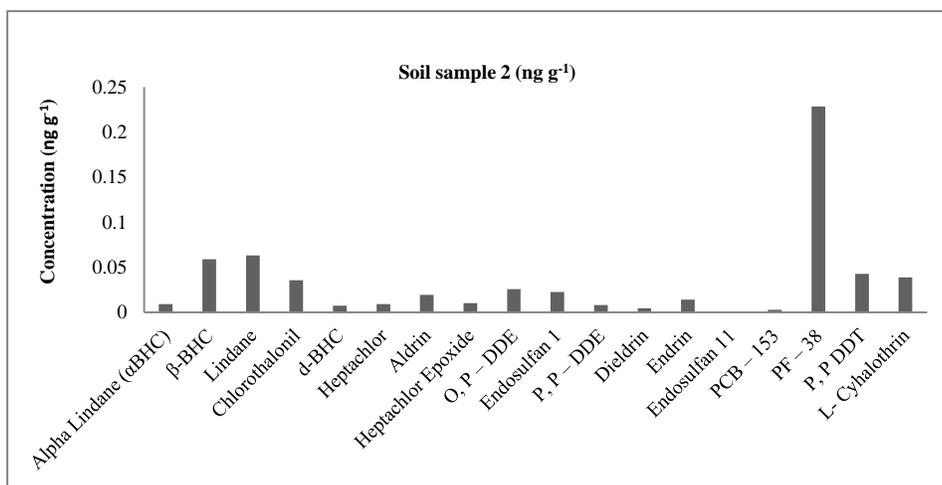


Figure 3. Concentration of OCP in soil sample 2.

OCPs in sediment

The results of OCPs component in sediment samples are shown in Figures 4 and 5. The concentrations of α -lindane, β -lindane, lindane, chlorothalonil, δ -BHC and Heptachlor were 0.0048 ng g^{-1} , 0.0319 ng g^{-1} , 0.0285 ng g^{-1} , 0.0158 ng g^{-1} , 0.0109 ng g^{-1} and 0.0229 ng g^{-1} for the first sediment sample taken at the upstream of the river (sediment sample 1) while the corresponding values for the same components of the second sediment sample (downstream) were 0.0046 ng g^{-1} , 0.0360 ng g^{-1} , 0.0330 ng g^{-1} , 0.0178 ng g^{-1} , 0.0127 ng g^{-1} and 0.0278 ng g^{-1} respectively. The following OCPs components were absent in the first sediment sample: heptachlor epoxide, endrin, endosulfan II, p,p-DDT and L-cyhalothrin but, with the following corresponding concentrations in sediment sample 2 from the first sediment sample: 0.0064 ng g^{-1} , 0.0168 ng g^{-1} , 0.0030 ng g^{-1} , 0.0435 ng g^{-1} and 0.0587 ng g^{-1} respectively.

The concentrations of o,p-DDE, endosulfan, p,p-DDE and dieldrin were 0.0109 ng g^{-1} , 0.0033 ng g^{-1} , 0.0053 ng g^{-1} and 0.0054 ng g^{-1} respectively for sediment sample 1 and 0.0091 ng g^{-1} , 0.0124 ng g^{-1} , 0.0058 ng g^{-1} and 0.0087 ng g^{-1} for same OCP components in sediment sample 2. The total OCPs concentration in sediment sample 1 was 0.1506 ng g^{-1} while that of sediment sample 2 was 0.02913

ng g^{-1} the concentration of the OCP is significantly higher ($p \geq 0.05$) in first sediment sample than the second sediment sample. According to Idowu *et al.* [34], most of the pesticides were detected with endosulfan isomers occurring most frequently in sediment samples analysed. Harieth [35] investigated OC pesticide residues in water and sediment samples from four rivers running through an intense agricultural area in Kilimanjaro, and found out that concentration levels ranged from trace (endrin) to 120 ng l^{-1} (p,p'-DDD) for water, and from trace (aldrin) to 132 ng g^{-1} dry weight (p,p'-DDD) for sediments, and were higher during the dry season. In the rivers of Kilimanjaro, Tanzania, a major water contamination was discovered which exceeds the European Union's allowable limit for human health protection. A research conducted by Akan *et al.* [36] on organophosphorous, pyrethroid, and OC pesticides residues revealed the presences of these OCPs in the water and sediment samples. Akan, [18] analysed for the presence of OCPs in sediment and water from Agboyi Creek, Lagos, samples were analyzed for aldrin, dieldrin, endrin, DDT, heptachlor, HCH, endosulfan, chlordane and methoxychlor, results showed varying levels of organochlorine pesticides.

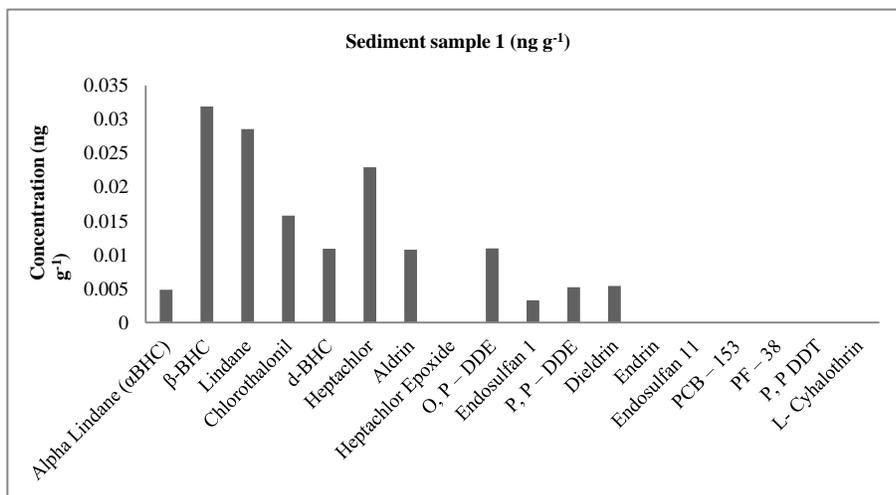


Figure 4. Concentrations of OCPs in sediment sample 1.

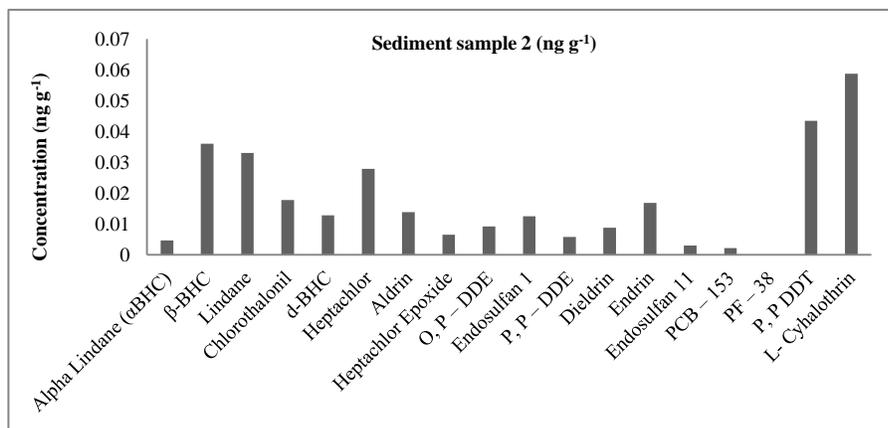


Figure 5. Concentrations of OCPs in sediment sample 2.

OCPs in fish sample

The result is shown in Figure 6. Out of the 16 OCPs analysed for in the fish sample, four components were not detected which were δ BHC, p,p-DDT and L-Cyhalothrin. The concentration of other components varies: α-BHC (0.0046 ng g⁻¹), β-BHC (0.0085 ng g⁻¹), Lindane (0.0085 ng g⁻¹) and Chlorothalonil (0.0139 ng g⁻¹). The concentration of β-BHC and Lindane were the same in the fish sample (0.0085 ng g⁻¹). The concentration of heptachlor, aldrin, heptachlor epoxide, o,p-DDE and Endosulfan I were 0.0088 ng g⁻¹, 0.0046 ng g⁻¹, 0.0309 ng g⁻¹, 0.0100 ng g⁻¹ and 0.0062 ng g⁻¹ respectively. The concentration of the remaining components were 0.0083 ng g⁻¹, 0.0122 ng g⁻¹, 0.0179 ng g⁻¹, and 0.0145 ng g⁻¹ for p,p-DDE, dieldrin, endrin and endosulfan II. In the fish sample, the total value of OCPs was 0.149042 ng g⁻¹.

A research conducted by Kafilzadeh [37] revealed the presence of OCP with varying concentration range. The concentration range of DDT was 3.751-5.273ppb, 3.952-5.681ppb for DDE, 0.027-0.045ppb for chlordane. Other concentrations were 0.037-0.049ppb, 0.143-0.251ppb and 0.613-0.877ppb for heptachlor, lindane and endosulfan respectively. DDE a metabolite of DDT decompose slowly by micro-organisms which is more stable [38- 40]. Farmers employ endosulfan, a broad-spectrum pesticide [37]. Fish and other aquatic organisms, particularly invertebrates, are particularly poisoned by Endosulfan [41].

According to Atoko *et al.* [16] that studied OC in some fish species at Tono reservoir Northern Ghana with concentration range of pesticides: aldrin, p,p'-DDE and p,p,

DDD were 0.03-0.06µg g⁻¹, 0.20-0.42µg g⁻¹ and 0.14-0.15µg g⁻¹ respectively.

Fish is one of the major aquatic invertebrates having different feeding habitat and exhibit varying profile of accumulation of environmental pollutants, hence they are susceptible to contamination [16, 42] , as they tend to accumulate sediments bound environmental pollutants.

The HQ was calculated to ascertain the hazard level of the fish sample from Owan River, Edo State, Nigeria (Table 2). The result showed that the hazard is moderate (1.49) comparing the value with the guidelines for the interpretation of HQ. This result is in consonant with the study of Edjere et al. [43].

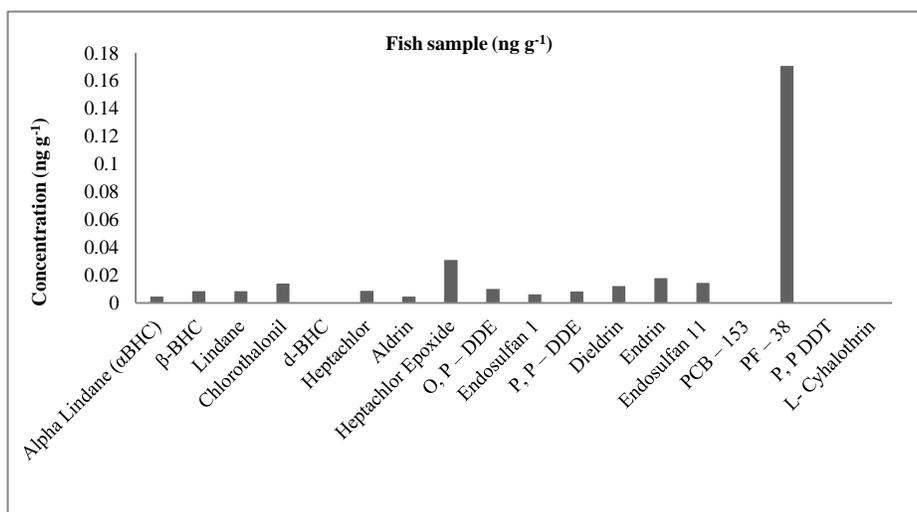


Figure 6. Concentrations of OCPs in fish sample.

Table 2. Hazard quotient (HQ) for the fish sample

	ΣOCPs concentrations (ng g ⁻¹)	HQ
Toxicity	0.149	1.49
Reference level [26]	0.1	

CONCLUSIONS

The 16 OCPs identified in this study were found at various amounts in soil, sediment, and fish (*Clarias anguillaris*) samples. Pesticide concentrations were higher in soil samples from farm locations near the river, and the pesticides bio-accumulated in the fish tissues. The concentration of OC pesticide in the fish was greater when compared to the sediment samples. Hazard quotient assessment revealed that the hazard is moderate.

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Conflict of interest

Authors declare that they have no conflict of interest regarding the publication of this research article.

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