

## PHYSIOCHEMICAL CHARACTERISTICS AND ORGANOCHLORINE PESTICIDES IN SURFACE WATER AND FISH, AND POTENTIAL HEALTH RISK IN LOWER DELTA OF OUÉMÉ RIVER, BÉNIN REPUBLIC, WEST AFRICA

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### ABSTRACT

*This study identified the levels and potential risks of OCP residues in water and Oreochromis niloticus tissues from Adjohoun, Dangbo and Aguégus of the Lower Delta of Ouémé River. The presence of seventeen OCP residues was detected using gas chromatography equipped with an electron capture detector. The concentration of pesticide residues ranged from below the limit of detection (BLD) to 16.48 µg/L for water samples and BLD to 334.78 µg/kg/ww for fish. Endosulfan sulphate was the predominant residue in all the samples analyzed, at the mean concentrations of 32.2 ± 27.04, 281.91 ± 55.22 and 49.33 ± 5.33 µg/kg/ww in gills, liver and muscle samples respectively. From the health risk analyses, ΣEndosulfan and ΣBHCs were above the Maximum Residual Limit recommended by Codex Alimentarius Commission. The estimated daily intake of all detected OCP residues in fish muscles exceeded the Permissible Tolerable Daily Intake and Acceptable Dietary Intake recommended by WHO/FAO, with exception of ΣEndosulfan. The assessment of the Target Hazard Quotient and Hazard Index indicated no health risk from the consumption of the fish flesh. Routine monitoring of pesticide residues is necessary for the prevention, control and reduction of OCP pollution, and minimizes health risks to humans.*

**Keywords:** Organochlorine pesticides, Lower Delta of Ouémé River, Surface water, *Oreochromis niloticus*, Human health hazard

### INTRODUCTION

The agricultural sector is the backbone of the economy of the Benin Republic and represented 25.6% of the GDP in 2017. Out of this 25.6%, cotton accounts for 40% of the GDP and roughly 80% of official export receipts, which directly or indirectly secures the income of a large part of the population (World Bank, 2019). The country is currently ranked first in Africa and 11<sup>th</sup> in the world with 1,450 (1000 480 lb. bales), followed by Mali second in Africa and 12<sup>th</sup> in the World with 1,425 (1000 480 lb. bales) in cotton production (USDA, 2019). To

improve agricultural yield, excessive pesticides are used by farmers to control the growth of weeds and to prevent crop damage by insects, rodents and mites. Pesticides are divided into many classes, of which the most important are organochlorines and organophosphorus compounds. OCP comprise a large group of structurally diverse compounds used to control agricultural pests, plant diseases and vectors of humans in Central Asia, India, China and Africa (Olisah *et al.*, 2020). They include DDT, methoxychlor, aldrin, dieldrin, mirex, lindane, benzohexachloride, taxopane, endosulfan, heptachlor and endrin. Most of the OCPs are limited and restricted

globally based on the Stockholm Convention on the persistence of organic pollutants recommendations due to their high lipophilicity, persistence and biological effects on the environment (Aktar *et al.*, 2009, Akan *et al.*, 2014). Moreover, the stated countries have banned the use of OCP due to its toxic effects on humans and their negative impacts on the ecosystems, in favour of more modern pesticide formulations. They are, nevertheless, still being used unofficially in large quantities in most developing countries including Nigeria, Ghana, Togo, Benin, Pakistan, and Argentina among many others, due to their effectiveness as pesticides and their relatively low cost and possibly due to weak import control and lack of logistics to monitor pesticides (Fosu-Mensah *et al.*, 2016; Jayaraj *et al.*, 2016; Ali *et al.*, 2017). The OCPs are lipophilic and hydrophobic and are ubiquitous contaminants that have been detected far from their sources of origin and they bio-accumulate along the food chain posing threat to human health and the global environment (Lari *et al.*, 2014; Dat and Chang, 2017). Ideally, the pesticide should be lethal to the pest targeted, but not to non-target pest species, including man. Unfortunately, this is not so. The dispute over the use and abuse of pesticides has surfaced in recent times, with arguments for and against their use. OCPs are volatile and can be transported into the aquatic environment as residues through different input pathways, such as through effluent release, discharges of domestic sewage and industrial wastewater, atmospheric deposition, runoff from agricultural fields, leaching, equipment washing, careless disposal of empty containers and direct dumping of wastes into the water systems (Bhadouria *et al.*, 2012; Kuranchie-Mensah *et al.*, 2012; Akan *et al.*, 2014). Thus, surface water contamination may have ecotoxicological effects on aquatic flora and fauna as well as on human health if consumed (Botwe *et al.*, 2012). Because of these facts, it is necessary and imperative to monitor the levels of these pollutants in aquatic environments and food chains. Consumption of food contaminated with OCP leads to exposure of humans to different health risks (Oyeyiola *et al.*, 2017).<sup>[14]</sup> Several monitoring studies have reported the

levels of OCP in different environmental matrices globally, most especially in surface water and sediments (Fosu-Mensah *et al.*, 2016; Lakhili *et al.*, 2018; Zeng *et al.*, 2018; Grondona *et al.*, 2019; Olisah *et al.*, 2020). Also, fishes have been widely used as biomonitors to assess bio-accumulation and bio-magnifications of OCP within the ecosystem and the health risks arising from contaminated fish consumption (Veljanoska-Sarafiloska *et al.*, 2013; Gbeddy *et al.*, 2015; Arisekar *et al.*, 2018). Fishes absorb these compounds directly from water by dermal absorption, respiration or by ingesting contaminated food. The region of accumulation of pesticides within a fish varies with the route of uptake. The gills are directly in contact with water; therefore, the concentration of pesticides in the gills reflects their concentration in the water where the fish live (Teklit, 2016). In agreement with their lipophilic nature, higher levels of OCP were measured in fish samples than in water and sediment samples (Gbeddy *et al.*, 2015). Fish is an essential source of proteins and vitamins for human beings. The most commonly detected OCP in Beninese freshwater systems are DDT, lindane, endosulfan, aldrin and dieldrin and hexachlorocyclohexane (BHC) (Soclo *et al.*, 2004; Pazou *et al.*, 2006; 2013).

The persistent nature of organochlorine pesticide residues in the environment may pose the problem of acute or chronic toxicity to animals and human beings via air, skin, drinking water and food intake (Bernardes *et al.*, 2015; Olisah *et al.*, 2020). Their mixture in the environment or human body poses a greater risk than when single (Zeng *et al.*, 2018). Most OCP are classified as persistent organic pollutants (POP) (UNEP, 2015), and may potentially affect public health, biodiversity, and non-target organisms, such as fish, algae and aquatic invertebrates (Papadakis *et al.*, 2015). Notably, OCP's adverse human health effects include; non-Hodgkin's lymphoma (Cantor *et al.*, 1992), female hormonal dysfunction (Bretveld *et al.*, 2006), breast cancer (Cohn *et al.*, 2007), neurodegenerative diseases (Zhao *et al.*, 2008), birth defects, dysfunctional immunity, neurological damage (Singh *et al.*, 2012), increased cancer burden (Alavanja *et al.*, 2013), nervous and endocrine systems disorder (Jara *et al.*, 2013),

reproductive defects, endocrine and immunological toxicities (Mrema *et al.*, 2013), disrupting DNA (deoxyribonucleic acid) in unborn children, the endocrine system, as well as damaging nerves and brain cells (Williams and Unyimadu, 2013), headache, vomiting, skin rash, respiratory problems and convulsions (Blair *et al.*, 2015), and other diseases in humans (Nicolopoulou-Stamati *et al.*, 2016).

Nowadays, issues of water quality are gaining much attention as aquatic ecosystems are getting heavily polluted. In recent decades, most countries are undergoing rapid industrial development, urbanization, extensive agriculture, mining activities and deforestation. These activities lead to environmental problems such as water pollution (Singh and Singh, 2017).

Ouémé River, the largest freshwater ecosystem in the Benin Republic, is a source of water and fish for many communities from North to the South Benin Republic. It also receives wastes and effluents from many cities along the bank of the river, wastewater discharge from industries such as Chinese Industry (YUEKEN) and the sugar manufacturing company [Sucrerie du Complant du Bénin (SUCOBE)], domestic residencies, mechanical workshops, artisanal fishing and agricultural runoff (intensive production of cotton and food crops).

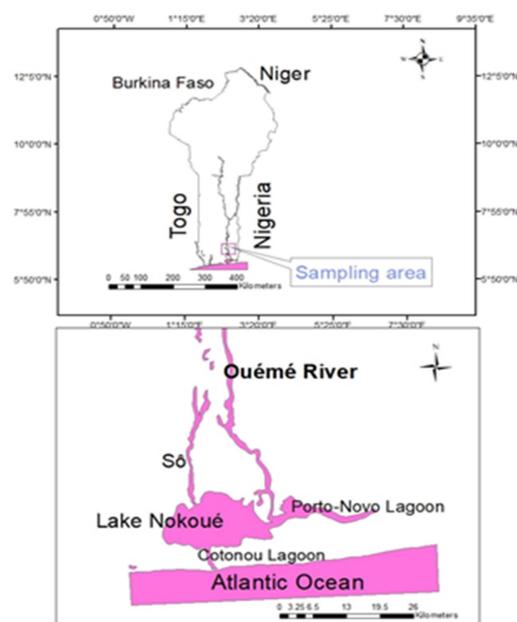
Assessment of OCP residue concentrations in water and fish is important for monitoring the aquatic environment pollution and human safety on the consumption of polluted fish. This is due to the toxicity of these contaminants to living organisms and humans even at low concentrations. Unfortunately, very few researchers have focused on this context, especially in the Lower Delta of the Ouémé River. To the best of our knowledge, this is the third study that has investigated the levels of selected OCP residues in *O. niloticus* from the Lower Delta of the Ouémé River after Pazou *et al.* (2006; 2013) who investigated the influence of fishing technique on organochlorine pesticide accumulation in water and fish and its possible human health risk. Moreover, it is recommended that the practice of early detection of pollutants should be continued to update whether the OCP residues concentrations are above or below the

permissible limit, and if it is above the limit then precautions must be taken to avoid consumption of contaminated aquatic plants and animals, and management measures put in place to bio-remediate the ecosystem (Awofolu and Fatoki, 2003; Zhang *et al.*, 2019; Bashir *et al.*, 2020).

This research, therefore, investigated OCP residues concentration in water and fish from the Lower Delta of the Ouémé River, which constitutes a source of protein in the region and a potential health hazard posed to humans consuming such fishes.

## MATERIALS AND METHODS

**Study Area:** Ouémé River is a very important freshwater body in the Bénin Republic. It provides drinking water, fishes as well as snails for a large population in Bénin. Ouémé River is the largest freshwater in Bénin. It rises in the Atakora Mountains; about 510 km (320 miles) long and has an area of 50,000 km<sup>2</sup>. The river flows from north to south. Ouémé River passes through several agroecological zones and runs downstream into Lake Nokoué through a Delta zone. The lower Delta of Ouémé is located between latitudes 6° 33' and 8° 15' and the meridians at 1° 50' and 2° 00' (Zinsou *et al.*, 2016) (Figure 1).



**Figure 1: Map of Ouémé River showing the study area and its position in the Republic of Bénin**

The Lower Delta of Ouémé begins after Adjohoun municipality and ends at the south facade where the river flows into Lake Nokoué at Porto-Novo (Lalèyè *et al.*, 2004). Many tributaries of Ouémé River carry wastewater from agricultural land especially pesticides from cotton farms, industries such as the Chinese industry YUEKEN and sugar-making company (SUCOBE) located at Atchakpa and domestic wastewater from various towns. These discharges into the river cause pollution in the river and deteriorate its water quality. Water and *O. niloticus* samples were collected from three locations: Adjohoun (6°42'4.82" N and 2°28'34.58" E), Aguégoués (6°27'23.98" N and 2°32'37.97" E) and Dangbo (6°35'15.17" N and 2°30'2.13" E) to study the impact of pesticides pollution, on water physicochemical characteristics, fish and human health. These three sampling sites are located downstream which is considered to be the most polluted.

### Field Sampling and Sample Preparation

**Water samples:** Water samples were collected according to the standard methods for the examination of water and wastewater (Baird *et al.*, 2017). A total of 26 water samples were collected from each site during the period of sampling from December 2018 to March 2019. Water samples from each site were collected using new and cleaned polyethene bottles of one-litre capacity for physicochemical analysis. In contrast, amber glass bottles of one-litre capacity were used for pesticide analysis.

Water samples for pesticide analysis were collected in one-litre amber glass bottles previously washed with hot water and detergent and rinsed three times with sulphuric acid and deionized water respectively. Bottles were rinsed twice with the river water to be sampled, and then carefully filled just to overflow at 20 cm below the water surface without passing air bubbles through the sample or trapping air bubbles in sealed bottles. Samples were kept in an icebox containing ice blocks while they were transported to the laboratory. Samples were acidified with 1 mL of H<sub>2</sub>SO<sub>4</sub> (50% v/v) to prevent breakdown and bio-degradation and stored in the dark at a temperature between 0°

C and 4° C until extraction, which was done within two days.

**Fish samples:** Freshwater fish; *Oreochromis niloticus* (25 samples) at its commercial size were collected from the same areas where water samples were collected with the help of a professional fisherman (Figure 2) using fishing nets within the same period.



**Figure 1: Fish sampling from Ouémé River by professional fishermen. Source: Simon Zonkpoedjre**

Samples were washed with clean water at the point of collection, placed immediately in polyethene bags, put into an icebox containing ice blocks and transferred to the laboratory at the Nigerian Institute of Oceanography and Marine Research (NIOMR) for analyses. The total length and the body weight of each captured fish were measured to the nearest centimetre and gram respectively. The fish lengths were between 17 and 20 cm, and the wet fish weight ranged from 160 to 250 g. The fish were washed carefully with distilled water before dissection.

### Physicochemical Parameters of Ouémé River

**Temperature:** Water temperature was measured *in situ* in degree centigrade (°C) using a "SANITAS Thermometer Multifunction" at the sampled stations.

**pH:** The pH was measured *in situ* with a digital HANNA pH meter. The digital pH meter was

calibrated before use. The value was read directly from the screen.

**Dissolved oxygen:** The dissolved oxygen concentration was determined following the Winkler method (Bruckner, 2011). A 300 mL glass BOD stoppered bottle was brim-full with sample water. Immediately, 2 mL of manganese sulphate was added to the bottle by inserting the calibrated pipette just below the surface of the liquid in order not to introduce oxygen into the sample. 2 mL of alkali-iodide-azide reagent was added in the same manner using a pipette. The bottle was stoppered with care to ensure that no air was introduced. The sample was mixed by inverting the bottle seven times. The bottle was checked for air bubbles and discarded where air bubbles are seen. The presence of oxygen was indicated by the appearance of a brownish-orange cloud of precipitate or floc. Allow the floc to settle on the bottom, and mix the sample again by turning the bottle upside down several times, and let it settle again. 2 mL of concentrated sulphuric acid through a pipette held just above the surface of the sample was added. The bottle was carefully stoppered and inverted several times to dissolve the floc. At this point, the sample was fixed and can be stored for up to eight hours in a cool dark place. As an added precaution, the stoppered bottle was capped with aluminium foil and a rubber band during storage. In a glass flask, 201 mL of the sample was titrated with sodium thiosulphate to a pale straw colour. Titration was done by slowly dropping the titrant from a calibrated pipette into the flask and continually stirring the sample water. 2 mL of starch solution was added to produce a blue colour. Titrate slowly until the sample turns clear. At the experimental endpoint, it will take only one drop of the titrant to eliminate the blue colour. Caution was taken that each drop was fully mixed into the sample before adding the next drop. The dissolved oxygen concentration in the sample is equivalent to the millilitres of titrant used. Each mL of sodium thiosulphate added equals 1 mg/L dissolved oxygen.

**Determination of alkalinity:** 50 mL water sample was collected and 3 drops of

phenolphthalein indicator were added, titrate the 50 mL sample with 0.02N sulphuric acid to pH 8.3 and estimate phenolphthalein alkalinity (phenolphthalein indicator changed colour, from pink to clear, at pH 8.3). Phenolphthalein alkalinity (in mg/L as  $\text{CaCO}_3$ ) =  $(A1 \times N \times 50,000) / V$  (2a) Where: A1 = volume of sulphuric acid used in mL; N = normality of acid used for the titration; V = volume of sample used in millilitres. To the same sample, three drops of bromocresol green indicator were added. 50 mL sample was titrated with 0.02N sulphuric acid to pH 4.5 and total alkalinity was estimated when the bromocresol green indicator changed colour, from blue to yellow, at pH 4.5. Amount of acid used starting from step 1 (i.e., A2) reacts with the hydroxide, carbonate and bicarbonate and contributes to the total alkalinity (Eq. 2b): Total alkalinity (in mg/L as  $\text{CaCO}_3$ ) =  $(A \times N \times 50,000) / V$  (2b), where: A2 = volume of acid used in mL starting from step 1 (i.e.,  $A2 > A1$ ) (Snoeyink and Jenkins, 1980).

### Organochlorine Pesticides Analysis

**Extraction of organochlorine pesticides from water samples:** Water samples were subjected to liquid-liquid extraction. 200 cm<sup>3</sup> aliquot of water sample was placed into a 300 cm<sup>3</sup> capacity separating funnel, to which 30 cm<sup>3</sup> of HPLC grade dichloromethane was added. The mixture was vigorously shaken and the stopper was removed continuously to release the gas built up in the bottle. The extracted solvent was carefully drained into a glass container. The extraction process was repeated with 20 cm<sup>3</sup> dichloromethane and the solvent extract was again drained out and combined with the first portion (USEPA, 2010).

**Extraction of organochlorine pesticides from *Oreochromis niloticus* tissues samples:** The fishes were dissected between the pectoral fin and vent of each fish to open up the viscera. The separated organs of the *O. niloticus* (livers, gills and muscles) were extracted. 5 g of fish sample was homogenized with 5 g of anhydrous granulated  $\text{Na}_2\text{SO}_4$  using a mortar and pestle. Cold solvent extraction was performed. 50 cm<sup>3</sup> of the n-hexane was introduced into a 250 cm<sup>3</sup>

reagent bottle containing the homogenized fish sample. The mixture was shaken in a mechanical shaker for 45 minutes and the stopper was removed intermittently to release the gas built up in the bottle. The mixture was allowed to stand for 30 minutes and then filtered into a glass container using Whatman filter paper No. 1 (USEPA, 2002).

**Pre-concentration of extracts:** The extracted solvents were concentrated to 1 cm<sup>3</sup> using a rotary evaporator and kept for the clean-up procedure.

**Clean-up of extracts:** The clean-up of the extracts was done using column chromatography (USEPA, 1996). The glass separating column (20 cm) was packed with activated silica gel (90% < 45 µm) and washed down with hexane to remove any dirt. The extracts were demineralized over 1 g of anhydrous granulated Na<sub>2</sub>SO<sub>4</sub> and separated into two fractions using mixtures of dichloromethane, hexane and acetonitrile as eluting solvents. 30 cm<sup>3</sup> of a dichloromethane/hexane (20/80) mixture was used for the first fraction, while 30 cm<sup>3</sup> of a dichloromethane/hexane/acetonitrile (50/49.5/0.5) mixture was used for the second fraction to make sure that the polar acetonitrile eluted any remaining residue. The fractions were combined, and concentrated to 1 cm<sup>3</sup> volume using a rotary evaporator and subsequently analysed.

**Analysis of organochlorine pesticides:** Organochlorine pesticides were analysed using an Agilent 7820A series Gas Chromatography equipped with an Electron Capture Detector (Agilent 7820A GC-ECD). The gas chromatography conditions for pesticide analysis are in Table 1. Chromatographic separation was carried out using a DB-17 capillary column (30 m × 250 µm × 0.25 µm) with a flow rate of 2 mL/minute. Helium (He) was used as carrier gas with a flow rate of 2 mL/minute. Samples (1 µL) were injected under the splitless injection mode. The injection and detector temperatures were 250°C and 290°C respectively.

**Table 1: Gas chromatograph conditions for pesticide analysis**

Item	Condition
Acq. Operator	NIOMR Central Lab
Model	Agilent 7820A GC-ECD
Column	DB 17 (30m x 250µm x 0.25µm)
Carrier gas	Helium (He)
Injection mode	Splitless
Injection temperature	250°C
Flow rate	2 mL/min
Oven temperature	Initial temp: 150°C Increase to 280 @ 6°C/min Total run time: 21.667mins
Detector Temperature	290°C

The oven temperature was programmed to increase from 150°C to 280°C at 6°C/minute and held for 21.667 minutes. Quantification of OCP and their residues was performed using the OCP standards. The concentrations of the pesticide residues were calculated directly by the gas chromatograph after inputting the volume and weight of the samples.

**Human Health Risk Assessment:** Human health assessment has been studied to determine the potential health hazard due to exposure to toxic substances in different environmental media and foodstuffs (Liu *et al.*, 2010). Risk assessment for OCP residue was estimated using parameters such as Estimated Daily Intake (EDI), Target Hazard Quotient (THQ) and Hazard Index (HI) (USEPA, 1987).

**Estimated daily intake (EDI) of organochlorine pesticides:** EDI was assessed according to Łuczyńska *et al.* (2018) thus:  $EDI = C \times IR/BW$ , where: EDI – is the estimated daily intake (µgkg body weight<sup>-1</sup> day<sup>-1</sup>), C – is the mean concentration of OCP in foodstuff (mgkg<sup>-1</sup> wet weight), IR – is the daily ingestion rate for fish consumption in the Benin Republic (13 Kg/year equivalent to 37 g/day) (FAO, 2013) and BW – is the mean body weight (60 kg).

**Target hazard quotient (THQ):** THQ was determined using the methods explained by USEPA (1987) and Kawser Ahmed *et al.* (2016). THQ estimated the non-carcinogenic health risk of consumers due to intake of OCP-contaminated fish using an oral reference dose. When  $THQ < 1$  then it means the health benefit of fish consumption and that the consumers are safe, whereas  $THQ > 1$  suggested a high adverse health risk.  $THQ = (Efr \times ED \times FIR \times C / RfD \times BW \times TA) \times 10^{-3}$ , where: Efr – is the exposure frequency (365 days year<sup>-1</sup>), ED – is the exposure duration (70 years), FIR – the fish ingestion rate (g person<sup>-1</sup> day<sup>-1</sup>), C – the mean concentration of OCP in foodstuff ( $\mu\text{g g}^{-1}$  wet weight), RfD – the oral reference dose ( $\text{mg kg}^{-1}$  day<sup>-1</sup>) (USEPA, 1987), BW – the mean body weight (60 kg), TA – the mean exposure time (365 days year<sup>-1</sup> × ED).

**Hazardous index (HI):** This Index was calculated according to Kawser Ahmed *et al.* (2016) thus:  $HI = \sum THQ = THQ (\text{Aldrin}) + THQ (\text{Dieldrin}) + THQ (\text{Endrin}) + THQ (\text{Endrin aldehyde}) + THQ (\text{Methoxychlor}) + THQ (\text{Endosulfan I}) + THQ (\text{Endosulfan II}) + THQ (\text{Endosulfan sulphate}) + THQ (\text{Heptachlor}) + THQ (\text{Heptachlor epoxide}) + THQ (\text{Alpha-BHC}) + THQ (\text{Beta-BHC}) + THQ (\text{Delta-BHC}) + THQ (\text{Gamma-BHC}) + THQ (p,p'-DDT) + THQ (p,p'-DDD) + THQ (p,p'-DDE)$ .

**Data Analysis:** Experimental data collected were subjected to a one-way analysis of variance (ANOVA) to compare the means. A post hoc test – the Duncan test of multiple comparisons was used to determine the significantly different means. Regression and correlation statistics were used to establish the relationship between OCP concentrations in water and fish. All statistical analyses were performed using SPSS (Statistical Package of Social Sciences) software version 22. Results with  $p < 0.05$  were considered to be statistically significant.

## RESULTS

**Physicochemical Parameters of Ouémé River:** The physicochemical parameters

including pH, water temperature, dissolved oxygen (DO) and alkalinity of the study area are presented in Table 2. The mean concentration of pH was between  $7.24 \pm 0.38$  (Avagbodji) and  $7.88 \pm 0.44$  (Hêtin). The water temperature varied from  $26.77 \pm 0.65$  (Hozin) to  $29.74 \pm 1.09^\circ \text{C}$  (Avagbodji), whereas the mean concentration of alkalinity varied from  $39.00 \pm 2.00$  (Gbada) to  $45.00 \pm 5.77 \text{ mg L}^{-1}$  (Avagbodji). There was no significant difference ( $p > 0.05$ ) among all the sampled sites except Gbada and Avagbodji. In the case of DO, Adjohoun ( $6.15 \pm 1.81 \text{ mg/L}$ ) differed significantly ( $p < 0.05$ ) from other sites.

### Organochlorine Pesticides Residues in Surface Water:

The mean concentration of various OCP detected in water from the three locations, namely, Adjohoun, Dangbo and Aguégué of the Lower Delta of Ouémé River are summarized in Table 3. The concentration of OCP residues ranged from BLD to  $16.48 \mu\text{g/L}$  in water from the Lower Delta of Ouémé River. In this study, endosulfan sulphate had the highest concentration ranging from  $12.88 \mu\text{g/L}$  to  $16.48 \mu\text{g/L}$  in the water samples. The mean concentrations of aldrin and endosulfan sulphate were relatively similar at all sites with Adjohoun and Dangbo having the highest concentration of  $1.84 \mu\text{g/L}$  and  $16.48 \mu\text{g/L}$  respectively. Heptachlor, p,p-DDE and Methoxychlor were only detected at Dangbo and Aguégué respectively. The total OCP concentrations at Adjohoun, Dangbo and Aguégué ranged from BLD to  $15.32 \mu\text{g/L}$ , BLD to  $16.48 \mu\text{g/L}$  and BLD to  $14.12 \mu\text{g/L}$  respectively. Among all the sites the highest mean concentrations were found at Aguégué ( $3.41 \pm 0.51 \mu\text{g/L}$ ), which is downstream of Ouémé River, where its discharge into Porto-Novo Lagoon and Cotonou Lagoon. The difference in the mean concentrations for all locations was however not significant ( $p > 0.05$ ). Meanwhile, significant differences ( $p < 0.05$ ) existed for the mean concentration of dieldrin ( $1.96 \pm 0.33 \mu\text{g/L}$ ) in Aguégué when compared to other sites. Other OCP not listed in Table 3 were BLD in all the sites. Nonetheless, variations in the frequency of OCP groups were detected in each site.

**Table 1: Physicochemical parameters of water collected from different sites of Ouémé River, Bénin Republic, West Africa**

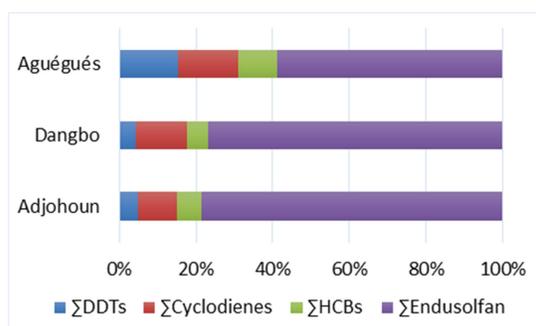
Parameters	Adjohoun		Dangbo		Aguégués	
	Adjohoun	Gbada	Hêtin	Hozin	Avagbodji	Houedome
<b>Coordinates</b>	6°42'4.82"N 2°28'34.58"E	6°41'5374"N 2°28'3628"E	6°35'15.17"N 2°30'2.13" E	6°32'5"N 2°32'12"E	6°27'23.98"N 2°32'37.97"E	6°29'50"N 2°32'38"E
<b>pH</b>	7.51 ± 0.52	7.32 ± 0.51	7.88 ± 0.44	7.74 ± 0.62	7.24 ± 0.38	7.45 ± 0.44
<b>Temperature (°C)</b>	27.26 ± 0.96 <sup>ab</sup>	28.85 ± 1.60 <sup>bc</sup>	28.08 ± 1.26 <sup>abc</sup>	26.77 ± 0.65 <sup>a</sup>	29.74 ± 1.09 <sup>c</sup>	28.14 ± 1.30 <sup>abc</sup>
<b>Alkalinity (mg/L)</b>	42.50 ± 5.97	39.00 ± 2.00	45.5 ± 4.43	40.50 ± 2.51	45.00 ± 5.77	42.00 ± 2.00
<b>DO (mg/L)</b>	6.15 ± 1.81 <sup>a</sup>	8.35 ± 1.11 <sup>b</sup>	8.82 ± 1.04 <sup>b</sup>	8.52 ± 0.20 <sup>b</sup>	9.15 ± 0.52 <sup>b</sup>	7.62 ± 2.33 <sup>ab</sup>

Means on the same row with the same letter superscript are not significantly different ( $p > 0.05$ )

**Table 3: Concentrations of organochlorine pesticides in water from studied sites of Ouémé River, Bénin Republic, West Africa**

Pesticides	Adjohoun		Dangbo		Aguégués	
	Range	Mean ± SD	Range	Mean ± SD	Range	Mean ± SD
<b>Heptachlor</b>	BLD	BLD	BLD-2.16	1.08±1.52	BLD	BLD
<b>Aldrin</b>	1.72-1.84	1.78±0.08	1.68-1.76	1.72±0.05	1.72-1.76	1.74±0.02
<b>D-BHC</b>	BLD-2.36	1.18±1.66	BLD-2.32	1.16±1.64	2.32-2.36	2.34±0.02
<b>p,p-DDE</b>	BLD-1.80	0.90±1.27	BLD-1.80	0.90±1.27	1.96-2.12	2.04±0.11
<b>Dieldrin</b>	BLD	BLD	BLD	BLD	1.72-2.20	1.96±0.33
<b>Endosulfan Sulphate</b>	12.88-15.32	14.10±1.72	15.76-16.48	16.12±0.50	13.48-14.12	13.80±0.45
<b>Methoxychlor</b>	BLD	BLD	BLD	BLD	BLD-3.08	1.54±2.17

At Adjohoun and Dangbo,  $\Sigma$ Endosulfan comprised 78.5% and 76.83% of total OCP respectively, while at Dangbo the frequency (58.92%) was relatively low compared to other sites (Figure 3).

**Figure 2: Composition of organochlorine pesticides group in water collected from different sampled sites of Ouémé River, Bénin Republic, West Africa**

The compositional trends of OCP groups in all collected water samples followed the pattern:  $\Sigma$ Endosulfan (71.41%) >  $\Sigma$ Cyclodienes (13.01%) >  $\Sigma$ DDTs (8.19%) >  $\Sigma$ BCHs (7.36%). The compositional trends of OCP groups in Aguégués had the same trend as other locations, whereas, there were differences in

Adjohoun and Dangbo that had  $\Sigma$ Endosulfan >  $\Sigma$ Cyclodienes >  $\Sigma$ BCHs >  $\Sigma$ DDTs.

### Organochlorine Pesticides Distribution in *Oreochromis niloticus* Tissues:

Out of the 17 OCP analyzed, 15 OCP (alpha-BHC, p,p-DDD, p,p-DDE, heptachlor, heptachlor epoxide, aldrin, dieldrin, endosulfan I, endosulfan II, endosulfan sulphate and methoxychlor) were detected in fish tissue whereas p,p-DDT and endrin were below the limit of detection. The mean concentrations and concentration ranges of detected OCP as well as BLD in fish tissues, namely, gills, liver and muscle are listed in Table 4, ranging from BLD to 334.78  $\mu\text{gkg}^{-1}$ . The highest OCP accumulations in fish from Adjohoun, Dangbo and Aguégués were detected in the liver (224.59  $\mu\text{gkg}^{-1}$ , 286.36  $\mu\text{gkg}^{-1}$  and 334.7  $\mu\text{gkg}^{-1}$  respectively), while the lowest was in the muscle (9.4  $\mu\text{gkg}^{-1}$ , 11.2  $\mu\text{gkg}^{-1}$  and 9.4  $\mu\text{gkg}^{-1}$  respectively). However, the occurrence of OCP in fish liver tissues ranged from BLD to 334.7  $\mu\text{gkg}^{-1}$ , which was comparatively higher than that in gills (BLD to 50.40  $\mu\text{gkg}^{-1}$ ) and the muscle (BLD to 55.00  $\mu\text{gkg}^{-1}$ ).

**Table 4: Organochlorine pesticide concentrations in tissues of *Oreochromis niloticus* sampled from Ouémé River, Bénin Republic, West Africa**

Pesticides	Organochlorine pesticide concentrations ( $\mu\text{g}/\text{kg}$ fresh weight)					
	Gills		Liver		Muscle	
	Range	Mean $\pm$ SD	Range	Mean $\pm$ SD	Range	Mean $\pm$ SD
Alpha-BHC	17.00-29.2	23.33-6.1 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
B-BHC	BLD-50.40	17.46. $\pm$ 28.52 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
Heptachlor	19.80-39.00	29.86 $\pm$ 9.6 <sup>b</sup>	74.59-136.23	112.09 $\pm$ 32.92 <sup>c</sup>	BLD-16.8	6.26 $\pm$ 9.12 <sup>a</sup>
Aldrin	14.60-30.20	20.60 $\pm$ 8.4 <sup>b</sup>	51.81-81.15	63.44 $\pm$ 15.58 <sup>c</sup>	9.40-11.20	10 $\pm$ 1.03 <sup>a</sup>
G-BHC	18.60-26.80	23.00 $\pm$ 4.1 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
Delta-BHC	15.20-33.60	22.00 $\pm$ 10 <sup>b</sup>	56.36-130.43	86.30 $\pm$ 39.01 <sup>c</sup>	BLD-13.8	8.93 $\pm$ 6.92 <sup>a</sup>
H-epoxide	BLD-17.20	6.40 $\pm$ 9.35 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
Endosulfan	BLD-11.60	4.53 $\pm$ 6.11 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
p,p-DDE	BLD-13.00	5.00 $\pm$ 6.92 <sup>a</sup>	BLD-68.11	11.79 $\pm$ 00 <sup>c</sup>	BLD-10.4	7.00 $\pm$ 5.2 <sup>b</sup>
Dieldrin	BLD-12.00	4.66 $\pm$ 6.35 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
Endrin	BLD-14.8	5.60 $\pm$ 7.96 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
p,p-DDD	BLD-11.80	4.60 $\pm$ 6.23 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
Endosulfan II	BLD-18.60	6.86 $\pm$ 10.16 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
p,p-DDT	BLD	BLD	BLD	BLD	BLD	BLD
Endrin aldehyde	BLD	BLD	BLD	BLD	BLD	BLD
Endosulfan Sulphate	BLD-49.00	32.2 $\pm$ 27.04 <sup>a</sup>	224.59-334.78	281.91 $\pm$ 55.22 <sup>c</sup>	44.40-55.00	49.33 $\pm$ 5.33 <sup>b</sup>
Methoxychlor	BLD-19.20	7.06 $\pm$ 10.50 <sup>b</sup>	BLD	BLD <sup>a</sup>	BLD	BLD <sup>a</sup>
<b><math>\Sigma</math>OCP</b>		213.16 $\pm$ 157.43 <sup>b</sup>		555.53 $\pm$ 142.73 <sup>c</sup>		81.52 $\pm$ 22.4 <sup>a</sup>

Mean concentration with the same letter superscript in the same row for each *O. niloticus* tissue are not significantly different ( $p > 0.05$ )

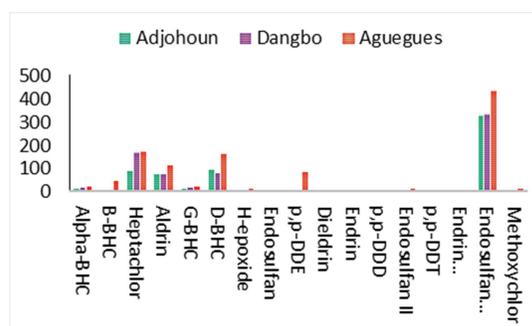
The ranges of the most abundant OCP were endosulfan sulphate (BLD - 334.78  $\mu\text{g}/\text{kg}$ ), heptachlor (BLD - 136.23  $\mu\text{g}/\text{kg}$ ), Aldrin (9.40-81.15  $\mu\text{g}/\text{kg}$ ), D-BHC (BLD - 130.43  $\mu\text{g}/\text{kg}$ ) and p,p-DDE (BLD - 68.11  $\mu\text{g}/\text{kg}$ ).

The mean concentration of endosulfan sulphate, heptachlor, aldrin and D-BHC in the fish liver was significantly different ( $p < 0.05$ ) from the mean concentration of the same OCP in gills and muscle. Nevertheless, there were no significant differences in the mean concentrations of p,p-DDE in the fish organs ( $p > 0.05$ ). However, the level of endosulfan sulphate was high in fish collected from the three locations followed by heptachlor, delta-BHC and aldrin.

The result showed the presence of all detectable OCP in the fish from Aguégués whereas only seven and six OCP were found in the *O. niloticus* from Adjohoun and Dangbo respectively.

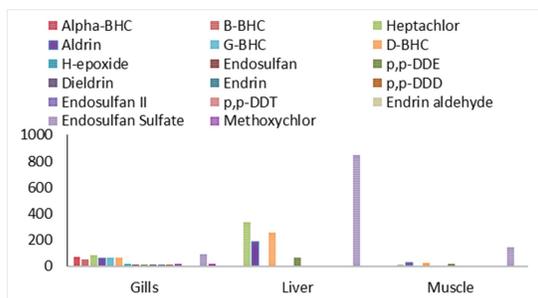
The majority of the OCP were detected in *O. niloticus* gills, while only five were found in the liver as well as in the muscle.

Endosulfan sulphate and heptachlor had the highest level of OCP measured in this study with a concentration of 334.78  $\mu\text{g}/\text{kg}$  and 136.23  $\mu\text{g}/\text{kg}$  in the liver respectively (Figure 4).

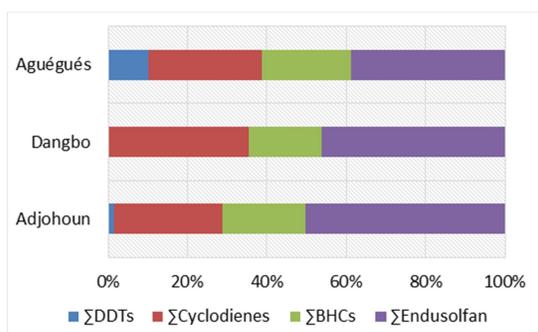


**Figure 4: Comparative levels of organochlorine pesticides in different sampled sites of Ouémé River, Bénin Republic, West Africa**

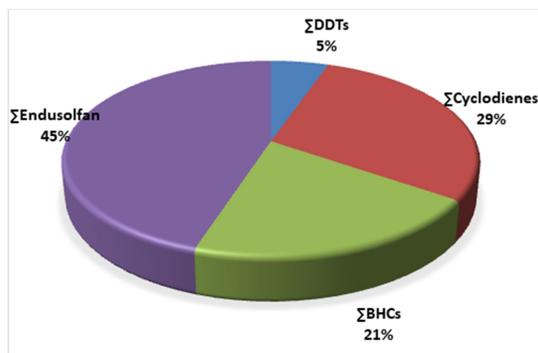
Moreover, the levels of all OCP in the liver tissues were generally higher than those of its corresponding gills and muscle tissues (Figures 5 – 7). The endosulfan sulphate level in the liver of *O. niloticus* was 9 and 6 times higher than that found in gills and muscle respectively.



**Figure 5: Organochlorine pesticide levels across various tissues of *Oreochromis niloticus* sampled from Ouémé River, Bénin Republic, West Africa**



**Figure 6: Composition of organochlorine pesticide groups in *Oreochromis niloticus* from different sampled sites of Ouémé River, Bénin Republic, West Africa**



**Figure 7: Percentage concentrations of organochlorine pesticide groups in *Oreochromis niloticus* and water from different sampled sites of Ouémé River, Bénin Republic, West Africa**

**Comparison of Levels of the Organochlorine Pesticides Residues in Water and Fish with the WHO and CAC Standards:** The OCP residues in the surface water were above the MRL established by World Health Organization (WHO) in all locations, except endosulfan (20  $\mu\text{g/L}$ ).

Ultimately, the concentrations of heptachlor, heptachlor epoxide, aldrin, dieldrin, and methoxychlor, were above MRL (0.03) set by WHO in all sites (Table 5). Nevertheless, in fish tissues,  $\Sigma\text{Endosulfan}$  were above the MRL of 200  $\mu\text{gkg}^{-1}$  recommended by CAC in all locations. Similarly to  $\Sigma\text{Endosulfan}$ ,  $\Sigma\text{BHC}$  was above the MRL of 10  $\mu\text{gkg}^{-1}$  recommended by CAC in all locations. On the other hand, dieldrin and aldrin were below the MRL of 200  $\mu\text{gkg}^{-1}$ , in the same way,  $\Sigma\text{DDT}$  was below the MRL of 5000  $\mu\text{gkg}^{-1}$  recommended by CAC in all locations. Despite, its presence in all tissues and all locations, heptachlor residues never exceeded the MRL established by FAO. Methoxychlor residues were also far below the MRL established by Agency for Toxic Substances and Disease Registry (ATSDR).

**Human Health Risk of Organochlorine Pesticides:**

Based on the concentration of OCP in fish collected from Lower Ouémé Delta, the per capita fish consumption in the Benin Republic is 13 Kg/year or equivalent to 37 g/day for an adult (60 kg) as well as the Oral reference dose (RfD), some parameters such as Estimated daily intake (EDI), Target Hazard Quotient (THQ) and Hazardous Index (HI) were calculated to estimate the potential risk to human health through these fish consumption.

The results of EDI are shown in Table 6. The EDI of OCP residues from the fish muscle of  $\Sigma\text{DDT}$ ,  $\Sigma\text{Endosulfan}$ , Heptachlor, delta-BHC and Aldrin were 6.41, 33.91, 10.36, 8.51 and 6.9  $\mu\text{g/person/day}$  respectively.

The THQ of OCP residues through edible muscle consumption are listed in Table 6. THQ of OCP residues were: Heptachlor ( $2 \times 10^{-2}$ ), Delta-BHC ( $1 \times 10^{-2}$ ),  $\Sigma\text{DDT}$  ( $12 \times 10^{-3}$ ) and  $\Sigma\text{Endosulfan}$  ( $5 \times 10^{-3}$ ). Moreover, the THQ of each OCP residue was below 1 which means there was no non-carcinogenic health risk for the people consuming fish muscle from Lower Ouémé Delta. Also, the HI of OCP residues were below 1, which means there was no adverse health effect. On the other hand, the HI of gills OCP residues was above 1.

**Table 5: Comparison of levels of organochlorine pesticide residues in water and fish with guideline values of World Health Organisation and Codex Alimentarius Commission**

OCP	Adjohoun	Dangbo	Aguegues	MRL ( $\mu\text{gkg}^{-1}$ ) <sup>a</sup>
	Pesticides in <i>Oreochromis niloticus</i> ( $\mu\text{gkg}^{-1}$ )			
Heptachlor	94.39	173.05	175.23	-
Aldrin	81.37	80.01	120.75	200
Delta-BHC	101.33	85.36	164.03	10
p,p'-DDE	10.4	-	90.71	5000
Dieldrin	-	-	12.0	200
Endosulfan Sulphate	326.19	330.76	432.38	200
Methoxychlor	-	-	19.2	-
Pesticides in water ( $\mu\text{g/L}$ )			MTL ( $\mu\text{g/L}$ ) <sup>b</sup>	
Heptachlor	BLD	1.08±1.52	BLD	0.03
Aldrin	1.78±0.08	1.72±0.05	1.74±0.02	0.03
Delta-BHC	1.18±1.66	1.16±1.64	2.34±0.02	-
P,P'-DDE	0.90±1.27	0.90±1.27	2.04±0.11	2
Dieldrin	BLD	BLD	1.96±0.33	0.03
Endosulfan Sulphate	14.10±1.72	16.12±0.50	13.80±0.45	20
Methoxychlor	BLD	BLD	1.54±2.17	0.03

<sup>a</sup>CAC (2019); <sup>b</sup>Rickert *et al.* (2016)

**Table 6: Comparison of estimated dietary intake (EDI), permissible tolerable daily intake (PTDI) with acceptable daily intake (ADI) stipulated by WHO/FAO**

OCP Residues	Concentration in fish muscle (mg/kg)	RfD (mg/kg/d)	THQ	EDI	PTDI (mg/kg)	ADI <sup>a</sup> (mg/kg)
Heptachlor	16.8	$1.3 \times 10^{-5}$	0.020	10.36	0.20	0.0005
Delta-BHC	13.8	$5 \times 10^{-4}$	0.010	8.51	0.006	0.0125
ΣDDT	10.4	$5 \times 10^{-4}$	0.010	6.41	0.01	0.010
ΣEndosulfan	55.0	$6 \times 10^{-3}$	0.005	33.91	1000	0.002
HI	0.045					

<sup>a</sup>WHO/FAO (1981)

**DISCUSSION**

**Physicochemical parameters**

In the present study, the mean pH of water samples from all sites was within the normal range (6.5 – 8.5) of WHO (2011). Unlike the result of this study, Chikou *et al.* (2011), Zinsou *et al.* (2016) and Nireti *et al.* (2017) recorded low pH in the same river. The mean temperatures recorded in this study were within the normal range for tropical water (25 to 30° C). According to Hoque *et al.* (2018), temperature plays a prominent role in dissolved oxygen levels. High water temperatures above 30 °C reduce the dissolved oxygen level. The mean DO levels reported in this study were within the recommended values by the Canadian Council of Ministers of the Environment for Warm

Freshwater (CCME, 1994). However, low levels of dissolved oxygen can put undue stress on the fish, and levels reaching less than 2 mg/L may result in death and can also change the oxidation state of substances from the oxidized to the reduced form (Hoque *et al.*, 2018). Alkalinity is defined as the measure of the capacity of water to neutralize or buffer acids using carbonate, bicarbonate ions, and in rare cases, hydroxide, thus protecting the organisms from major fluctuations in pH (PHILMINAQ, 2009). According to Warne *et al.* (2014), the acceptable level of alkalinity in a freshwater environment is > 20 mg/L which falls within the mean values obtained in this study.

**Organochlorine Pesticides Residues in Surface Water:** Organochlorine pesticides are fat-soluble, non-polar, hydrophobic, toxic, and

bio-accumulating chemical compounds made up of carbon, hydrogen and chlorine. OCP has been widely used as an insecticide to control insect pests that cause great damage to crops and against the diseases of domestic animals and humans that have mites and/or insect vectors. As a result, they were considered an advantage for agriculture and medical entomology and hence were used indiscriminately (Lal and Saxena, 1982). However, due to their great risk to the global environment, endangered human health, transportation over long distances, persistence and bioaccumulation in the food chain, they have been banned. Despite the ban, some OCPs are still used illegally in countries like Nigeria, Ghana, Togo, Benin, Pakistan, Argentina etc. (Fosu-Mensah *et al.*, 2016; Jayaraj *et al.*, 2016; Ali *et al.*, 2017). In Benin, OCP (DDT, lindane, dieldrin, heptachlor, etc.) were recommended in the 1960s and from 1999 till 2007 (endosulfan) for the control of insects devastation of the cotton plant (Fangninou *et al.*, 2019).

This study analyzed the content of 13 banned OCP, namely, Alpha-BHC, p,p-DDD, p,p-DDE, heptachlor, heptachlor epoxide, aldrin, dieldrin, endosulfan I, endosulfan II, endosulfan sulphate and methoxychlor, p,p-DDT and endrin in surface water and fish from Lower Delta of Ouémé River. The study showed the presence of these banned compounds in surface water and fish from the Lower Delta of Ouémé River in high concentrations. This may be due to the extensive use of OCP in agriculture activity and tsetse flies and malaria vectors control along the bank of the river (Pazou *et al.*, 2013). The result showed that endosulfan sulphate was the most dominant OCP in surface water samples followed by Aldrin. The highest detected OCP concentration in surface water was endosulfan sulphate (16.48  $\mu\text{g/L}$ ), which was the only metabolite present in the group of endosulfan in surface water, this means a rapid rate of degradation of its parents and the occurrence frequency was 100%. Endosulfan is a cyclodiene pesticide extensively used throughout the world to control a wide variety of insects and mites on crops (Jia *et al.*, 2009; Gbeddy *et al.*, 2015). It consists of endosulfan  $\alpha$ - and  $\beta$ - isomers; they are both fairly resistant

to photodegradation, but the metabolite endosulfan sulphate is susceptible to photolysis (Schuphan *et al.*, 1972). Endosulfan sulphate, which is a major degradation product of endosulfan, is known to be more persistent and toxic than its parents. Currently, there are few data comparable available on the concentration of endosulfan sulphate in surface water and fish in Benin. When considered as a group of endosulfan the level obtained in this study was comparatively lower than the concentrations 46, 45 and 35  $\mu\text{g/L}$  reported by Soclo *et al.* (2004) in Zone Cynegetique of Pendjari, Atacora and Djona respectively, in the cotton basin of Benin. Fosu-Mensah *et al.* (2016) reported endosulfan sulphate levels of 0.01 – 0.04  $\mu\text{g/L}$  in soils and drinking water sources from cocoa farms in Ghana, this value was much lower than what was observed in all locations in this study. Many previous studies; Zhou *et al.* (2008), Kuranchie-Mensah *et al.* (2012), Aydin *et al.* (2013) and Arisekar *et al.* (2018) have reported the presence of aldrin in surface water which was the second more frequent OCP in this study. Only p,p-DDE and delta-BHC were detected among all the DDT and BHC metabolites respectively. This may be due to their continuous use or the rapid degradation of their parents compounds (Sosan *et al.*, 2008; Muralidharan *et al.*, 2009; Liu *et al.*, 2010). Contrarily, to this study, many previous studies reported that DDT and BHC and their metabolites, dieldrin and heptachlor were the most frequent OCP in surface water (Darko *et al.*, 2008; Kuranchie-Mensah *et al.*, 2012; Aydin *et al.*, 2013). In this study all the OCP residues in the surface water were above the MRL in all locations; except endosulfan (20  $\mu\text{g/L}$ ) set by WHO (Rickert *et al.*, 2016). Ultimately, the concentrations of heptachlor, heptachlor epoxide, aldrin, dieldrin, and methoxychlor, were above MRL (0.03) set by the WHO (Rickert *et al.*, 2016) in all sites.

**Organochlorine Pesticides Distribution in *Oreochromis niloticus* Tissues:** Some of the OCP detected in this study are ubiquitous in both surface water and fish tissues whereas others like p,p-DDD, heptachlor epoxide, endosulfan I and endosulfan II were not detected in surface water. Furthermore, the

levels of OCP detected were found higher in the fish tissues compared to those in the water. Similar results have been reported by many authors. Gbeddy *et al.* (2015) reported higher levels of OCP in fish samples than in water and sediment samples from Volta Lake, Ghana. Higher o,p'DDT was recorded in fish than in sediments and water from the State of Campeche, Mexico (Hinojosa-Garro *et al.*, 2016). Veljanoska-Sarafiloska *et al.* (2013) also recorded higher OCP concentrations in the fish tissues than in water samples from the littoral zone of Lake Prespa in the Republic of Macedonia. The OCP compounds are hydrophobic, and thus have high affinities for lipids, and fish tissues are lipid-rich. Moreover, the higher levels depends on some parameters such as feeding behaviour, habitat, fat content, age, water-solubility, degree of ionization, stability, and molecular structure and affinity of the compound in the tissues of organisms (Zhao *et al.*, 2009; Eqani *et al.*, 2013; Pazou *et al.*, 2013). According to Campbell *et al.* (2000) and Eqani *et al.* (2013) bioaccumulation of OCP in fish can occur in two ways: aqueous uptake of hydrophilic compounds across the gill, and dietary uptake through the gastrointestinal membrane. Unfortunately, there is no MRL defined for the levels of OCP in the internal organs of fish. Nevertheless, in fish tissues,  $\Sigma$ Endosulfan were above the MRL of 200  $\mu\text{gkg}^{-1}$  recommended by CAC (2019) in all locations. Similarly to endosulfan,  $\Sigma$ BHC was above the MRL of 10  $\mu\text{gkg}^{-1}$  recommended by CAC (2019) in all locations. On the other hand, dieldrin and aldrin were below the MRL of 200  $\mu\text{gkg}^{-1}$ , in the same way,  $\Sigma$ DDT was below 5000  $\mu\text{gkg}^{-1}$  recommended by CAC (2019) in all locations. Despite, its presence in all tissues and all locations, heptachlor residues never exceeded the MRL established by FAO (2016). Methoxychlor residues were also far below the MRL established by the Public Health Service of the US Department of Health and Human Services (ATSDR, 2002).

The highest OCP residue level of 334.78  $\mu\text{gkg}^{-1}$  in this study was recorded in the liver tissue of *O. niloticus*. A similar, result was obtained in the Thamirabarani River system of Southern Peninsular, India, where OCP residues

in the liver were higher than those in gills and muscle (Arisekar *et al.*, 2018). All the OCP residues detected in the fish liver were also found in fish muscle but relatively lower. Liver tissue can, therefore, serve as a "marker" of the presence of OCP in the fish. However, all detected OCP residues present in fish gills were higher compared to those in fish muscle except for endosulfan sulphate. Yang *et al.* (2007) reported higher residue levels in fish from high mountain lakes and the Lhasa River in the Tibetan Plateau of China. The high concentration of OCP was also recorded in tilapia gills tissue in Volta Lake, Ghana (Gbeddy *et al.*, 2015). According to Gbeddy *et al.* (2015) the gills serve as a major channel for the absorption and distribution of OCP residues to various fatty tissues in fish.

The distribution of the amount of OCP in different locations might be attributed to several factors. All detected OCPs were found at Aguégúés and in high quantity when compared to Dangbo and Adjohoun. The explanation for the high amount of the OCP at Aguégúés which is downstream and mouth of Ouémé River may be due to the slow water current at this point, high malaria vector control activities and migration of OCP-contaminated fishes from either Cotonou Lagoon or Porto-Novo Lagoon. Pazou *et al.* (2013) have reported a higher concentration of OCP in fishes and other aquatic organisms obtained from Cotonou Lagoon.

**Human Health Risk:** Environmental Protection Agency (EPA) defined human health risk assessment as the process to estimate the nature and probability of adverse health effects in humans exposed to chemicals in contaminated environmental media, now or in the future (USEPA, 2022). Risk assessment for OCP is estimated using parameters viz; estimated daily intake (EDI), target hazard quotient (THQ) and hazard index (HI) (USEPA, 1987). Based on these parameters, the local population health risk from the consumption of fish from Lower Ouémé Delta using the OCP concentration from the muscle samples was evaluated. All EDI of OCP residues exceeded the acceptable daily intake (ADI) recommended by various agencies, except  $\Sigma$ Endosulfan which was below the recommended value.

In the present study, the THQ values for all OCP residues were below 1, which means there is no potential non-carcinogenic health risk from the ingestion of a single OCP residue through the consumption of fish. In a study from South Africa, the concentration of OCP in *Hydrocynus vittatus* muscle exceeded MRL in edible fat as set by the European Union (Gerber *et al.*, 2016). The health risk assessment also showed that the levels of OCP pose very high cancer risks to the local populations consuming the tigerfish, with as high as 2 in 10 increased risk factors (Gerber *et al.*, 2016). In another study from Sri Lanka, Jinadasa and Jayasinghe (2018) reported that the hazard ratio (HR > 1) for heptachlor exoepoxide-isomer B for *Anguilla* sp., while all other calculated OCP values were below 1. Those values (except HR > 1 scenario) are suggesting that the lifetime cancer risk associated with the consumption of studied fish species is greater than one in one million. Meanwhile, HR was almost close to 1 for  $\alpha$ -BHC in *Anguilla* sp. The hazard ratios calculated to assess the carcinogenic risk, in a study on the human health risk of organochlorine pesticides in water, sediments, and fish from the Chenab River, Pakistan indicated that the values for  $\Sigma$ DDT and aldrin HR were greater than one (HR > 1), indicating the probability of carcinogenic risk occurrence of one in million populations due to contaminated fish consumption (Riaz *et al.*, 2018).

**Conclusions:** From this study, the physicochemical of the water at all stations was within the normal range established by various parties (WHO, CCME and ANZECC). In this study, out of 17 OCP analysed, seven were detected in water (p,p-DDE, heptachlor, aldrin, dieldrin, endosulfan sulphate and methoxychlor and D-BHC), whereas, only p,p-DDT and endrin were not detected in fish tissues. Pesticide residues in water were largely lower than residues in the fish tissues. Endosulfan and its metabolites were the most abundant pesticides, with endosulfan sulphate being the predominant metabolite. Besides, endosulfan sulphate pesticide residues were detected in all the samples. The higher concentration of these pesticides in the aquatic environment could be

attributed to the current use of these OCP by the farmers to control the pests along the river. Geographically, the mean concentrations of the OCP reported in both water and fish tissues downstream (Aguégués) were found to be higher than those from Adjohoun and Dangbo. This could be attributed to various anthropogenic activities involving the use of OCP in the area. This study revealed that OCP residues in the surface water were above the MRL established by WHO in all locations, except for endosulfan. On the other hand, in fish tissues,  $\Sigma$ Endosulfan and  $\Sigma$ BHCs were above the MRL recommended by CAC, while, others were below their recommended value in all locations. Considering, the mean concentrations of the different contaminants in *O. niloticus* muscles the estimated EDI of all OCP residues exceeded the acceptable daily intake (ADI) recommended by WHO/FAO except  $\Sigma$ Endosulfan which is below the recommended value. This may be the main source of ingestion of these pollutants by humans. Further, the mean concentrations of OCP residues in *O. niloticus* flesh does not pose a non-carcinogenic health hazard to human that consumes this fish since their THQ and hazard index (HI) estimated were below one in all cases. But, the consumers of the whole *O. niloticus* are exposed to the non-carcinogenic health hazard because the THQ and hazard index (HI) of gills and liver estimated were above one. Consumers of *O. niloticus* from Ouémé River are advised to discard the liver and the gills. Conclusively, routine monitoring of OCP residues in the study area is necessary for the prevention, control and reduction of environmental pollution, to minimize health risks to humans. As such, the need to sensitize farmers on safe pesticide use is crucial to reduce the levels of pesticide residues in water and fish from the study area.

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