



Assessment of Organochlorines Pesticides Residues in Water, Fish and Sediment Samples from Wurbo Lake, Bali, Taraba State, Nigeria

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Abstract:

Organochlorines pesticides (OCPs) have been used worldwide, particularly in many African countries as in Nigeria for the control of pests. OCPs are characterized by their bioaccumulation in the environment, especially in the food chain, where they find their way into the human body. The research was aim to estimate the residual concentrations of different OCPs in water, fish and sediment from Kundi Lake to find out the extent of pesticide contamination and accumulation. The water sample was subjected to liquid-liquid extraction method while the fish and the sediment samples were subjected to soxhlet extraction. The extracts were later analysed for

organochlorines pesticide residues using Gas-Chromatography coupled with mass spectrometer (GC-MS). The average length, average weight and condition factor of the studied fish species were measured. The *Mormyrus rume* had the highest average length of 17.12 ± 0.05 cm, and average weight of 195.36 ± 0.05 g follow by *Clarias gariepinus* which had the average length of 16.83 ± 0.01 cm, and average weight of 189.43 ± 0.03 g. *Tilapia zilli* was the least species of fish sample with a length of 12.75 ± 0.03 cm and average weight of 102.21 ± 0.02 g. Also, all the fish species sampled in this present study had condition factors >1 . Based on the results obtained from all samples, it was found that the levels of organochlorine pesticides residues in water were generally below MRL as compared with FAO/WHO (0.5ppm). In fish samples, highest concentration of pesticide residues in *Mormyrus rume* follow by *Clarias gariepinus*, while the *Tilapia zilli* recorded low concentration of the pesticide residues. The Endosulfan II recorded high concentration of 4.68 ppm in *Mormyrus rume* follow by Endrin which recorded the concentration of 2.75 ppm, Aldrin 1.78 ppm and Atrazine 1.72 ppm all in *Mormyrus rume*. In *Clarias gariepinus*, Simazine 1.56 ppm follows by Endosulfan I 1.28 ppm while In *Tilapia zilli*, Endosulfan II 1.16 ppm follows by Atrazine 0.91 ppm and Beta Lindane 0.81 ppm. The results indicate that the area is contaminated with high levels of these pesticides in fish and sediment which may pose a great danger to the environment. Regular monitoring is therefore required to control the levels of pesticide residues in the water bodies.

Keywords: Water, Fish, Sediment, Organochlorine, Pesticide

Introduction

Pesticides include herbicides for destroying weeds and unwanted vegetation, insecticides for controlling insects, fungicides used for preventing the growth of molds and mildew and any other substances with the similar usage

(Randhawa et al., 2016). Pesticides use has increased worldwide, particularly in its use to salvage the food supply to the ever increasing global population. Although it is undisputed that pesticides are essential in modern agricultural activities. There is also growing concern about possible environmental contamination from



agrochemicals, industries, and household and rain water runoff from agricultural systems. (Thurman *et al.*, 2000). Agricultural practices are the largest consumer of pesticides in controlling various pests and unwanted weeds in Bali, Taraba State, Nigeria. Moreover, pesticides are also used in public health to control vector-borne diseases which includes malaria as well as unwanted plants such as grass and weeds in ornamental landscaping, parks and gardens. They are also useful in suppressing or avoiding the proliferation of insects, pests, bacteria, fungi, and algae in electrical equipment, refrigerators, paint, carpets, paper, cardboard, and food packaging materials (Gilden *et al.*, 2010). Various farming and fishing activities are taking place near the bank of Wurbo Lake year in year out could bring about water quality problems and disruption in fish (Akoto *et al.*, 2013). Organochlorines (OCPs) are chlorinated organic compounds used predominantly as insecticides. They are mainly classified into three categories; namely diphenylaliphatics such as DDT and its metabolite, cyclodienes which include Aldrin, Dieldrin, Endrin, heptachlors, Endosulfan, and Endosulfan sulphate (Nur-Banu and Semra, 2004). Organochlorines pesticides (OCPs) are persistent organic pollutants which have caused global concern as toxic environmental contaminants (Covacia *et al.*, 2005). OCPs are ubiquitous, hydrophobic and lipophilic contaminants with the potential for bioaccumulation in aquatic organisms especially fish, through food chain transfer (Afful *et al.*, 2010).

The occurrence of pesticides residue, especially organochlorines (OCs) in the environment is a great worry due to their tendency for long-range transport (Tongo *et al.*, 2019). Also their capacity to bio accumulate in food chain poses a threat to human health and the environment (Getenga *et al.*, 2004; Chau, 2005). The potential health implications of OCP exposures have been observed in increasing cases of cancer, chronic kidney diseases, sterility in both males and females, endocrine disorders, neurological and behavioural disorders (Abhilash & Singh, 2009). Fish can absorb these pesticides directly from water or by ingesting contaminated food (Teklit,

2016) and could pose potential human health hazards when consumed (Fianko *et al.*, 2011). Varying levels of organochlorines residues have been reported in fish (Fianko *et al.*, 2011; Tongo *et al.*, 2014; Ezemonye *et al.*, 2015) with different fish species varying greatly both in their susceptibility to OCPs and in their ability to store residues in their tissues (Johnson *et al.* 1973). Accumulation of pesticides in these organisms has become a serious public health issue worldwide (Okrikata *et al.*, 2022). Fish are used extensively for environmental monitoring because they concentrate pollutants directly from water and diet, thus enabling the assessment of transfer of pollutants through the food web (Bruggeman, 1982; Cox, 2002; Jeyakumar *et al.*, 2014).

Fish occupy different habitats in the ecosystem and have different feeding behaviours, thereby exhibiting different profile of accumulation of contaminants such as pesticides. For example, benthic fish species are considered more prone to contamination (Wasswa and Kiremire, 2004; Wei *et al.*, 2014) as they tend to accumulate sediments bound contaminants than pelagic fish (Perry *et al.*, 1996; Ccancapa *et al.*, 2016). When large animals feed on these contaminated organisms, the toxins are taken into their bodies moving up the food chain in increasing concentration in a process known as bio magnifications (Ujjania *et al.*, 2012). The accumulation of pesticides in the sediments is because of some physicochemical parameters such as P^H which have direct influence in the solubility of these pesticides in water (Rigotto *et al.*, 2013). Sediments also serve as an important sinks and remobilization of contaminants in aquatic system. The sediments act as secondary contamination source after water in the ecosystem and are the principal reservoirs of environmental pesticides representing a source from which residues can be released to the atmosphere, ground water and taken up by living organisms (Vega, 2005).

The used of modern pesticides in agriculture and public health is dated back to the 19th century when the German Scientists Ziedler synthesise the dichlorophenyltrichloroethane (DDT) as the first important synthetic organic pesticides in

1873. About 28 years ago, it was also estimated that each year about 500 million people were affected by water-borne or associated diseases and as many as 10 million of these die (Abera, 2011). Recently WHO reports that about 80 % of all human illnesses in the developing world are caused by biological contaminations (Essumang and Chokky, 2009).

Materials and Method

Study Site

The study was carried out at Wurbo Lake, Bali Local Government Area of Taraba State, Nigeria. Bali lies between latitude 7°46' N and 7°54' N of the equator and longitude 10°30' E and 11°00' E of the prime meridian (Topographic sheet, 1968). It is found in dry guinea savannah. It is among the largest local Government in Taraba State, with an estimated land area of 11,540 km². Based on the results of the 2006 National Population Census, Bali local Government had a population of about 211,024 persons (NPC, 2006). It has a tropical climate marked by two seasons; dry and rainy seasons. The rainy season starts around April and ends November occasionally, with 1350 – 1500 mm rainfall annually. The dry season is from December to March. The major ethnic groups in the area include; Jibu, Tiv, Chamba, Fulani and Itchen etc. The major occupation of the inhabitants is farming, fishing and nomadism. Their water sources for domestic and agricultural uses are River, Lake, ponds and wells.

Sample Collection

Water sample was collected from the locations within a depth of 20 cm into three separate pre clean 1 L plastic containers (thoroughly cleaned with soap and rinsed with acetone) and covered with screwed caps from different locations at appropriate depth and turbulent midstream positions of water bodies. These include the water inlet, centre and the outlet (spillway). The samples were preserved after collection in ice to minimize degradation of pesticide, stored at 4 °C until extraction. Sediment sample was taken from positions where a fine-texture substrate

deposition takes place. The upper 2cm of the bed sediment was collected with a Teflon coated spoon, stored in aluminium containers at -20 °C in the laboratory until analysis. The minimum of three fish species samples were collected. Fish identified and selected for this project are tilapia (*Tilapia zilli*), Catfish (*Clarias gariepinus*), and Mormyrids (*Mormyrus rume*).

Extraction of Pesticide Residues in Water Samples

The extraction sediment samples was done using the method described by Frimpong *et al.* (2013), with slight modification adopted from Okoya *et al.*, (2013) and Fosu-Mensah *et al.* (2016). The water sample was filtered using WHATMAN filter paper, 100 mL portion was drawn from the filtered water sample and transferred into 1 L capacity separating flasks with 30 mL of saturated sodium chloride solution (NaCl) added and mixed thoroughly. The mixture was shaken vigorously for 5 min and allowed to settle. After complete separation, the organic phase was drained into a 250 ml conical flask while the aqueous phase was re-extracted twice with 50 ml of n-hexane. The organic layers put together and dried over anhydrous sodium sulphate through filter papers into 50 mL round bottom flasks. The extracts from the water samples were then concentrated to about 2 mL on rotary vacuum evaporators and prepared for silica clean up (Joseph *et al.*, 2019).

Extraction of Pesticide Residues in Sediments

The extraction sediment samples was done using the method described by Frimpong *et al.* (2013), with slight modification adopted from Okoya *et al.*, (2013) and Fosu-Mensah *et al.* (2016). 10 g of the representative sediment sample was weighed and transferred into 250 mL separating flasks, 150 mL of acetonitrile was then added to each of the samples in the flasks and ultra-sonicated for 5 min. The flask was closed tightly having added an additional 10 mL of acetonitrile. Thereafter, the sample was placed on a mechanical shaker to shake continuously for 30 min at 300 mot/min. The content was then separated into layers when allowed to stand for 10 min. 10 mL of the

supernatants and dried over 2 g anhydrous magnesium sulphates through filter paper into 50 mL round bottom flasks. The extract was reduced to about 2 mL using rotary film evaporator and made ready for clean-up (Akan *et al.* 2015; Joseph *et al.*, 2019).

Extraction of Pesticide Residues in Fish

The extraction fish samples was done using the method described by Frimpong *et al.* (2013), with slight modification adopted from Okoya *et al.*, (2013) and Fosu-Mensah *et al.* (2016)., for the extraction of pesticides from the fish samples, ten (10.0) g of homogenized fish sample was weighed into a 150ml conical flask followed by the addition of 20 g and 5 g of anhydrous sodium sulphate and sodium hydrogen carbonate respectively. A mixture, 100ml of 1:1 (v/v) ethyl acetate/dichloromethane was transferred into the 20g fish samples and mixed thoroughly by shaking the conical flask while corked. Again, 20g of anhydrous sodium sulphate was added to the content of the conical flask followed by 20g of sodium hydrogen carbonate. The conical flask was corked tightly and the mixture shaken thoroughly for 10min. The content was allowed to stand for 3hrs. The organic layer was decanted into a 200ml round bottom flask and evaporated using the rotary evaporator at 40°C. The pesticide in the rotary flask was dissolved and collected with 2ml of ethyl acetate and transferred into a 2ml vial and ready for the clean-up (Akan *et al.* 2015; Joseph *et al.*, 2019).

Sample Clean-Up

According to methods described by Okoya *et al.*, (2013) and Fosu-Mensah *et al.* (2016), the clean-up of the extracted samples was done using chromatographic columns packed with silica gel which have 2 g layer of anhydrous Na₂SO₄ on top and conditioned with the extracting solvent. Approximately 2.5 g of activated silica gel was weighed and parked into a glass column which has been plugged with glass wool and 1.0 g of anhydrous sodium sulphate. About 10 ml n-hexane was used to wet and rinse the column. The extract was then transferred into the column and eluted with 20 mL portions of hexane/acetone mixtures. The elutes was

collected into a round bottomed flask and then concentrated to dryness. The residues was then dissolved in 2 mL of ethyl acetate and placed in a GC vial for gas chromatograph analysis (Akan *et al.* 2015; Joseph *et al.*, 2019).

GC Analysis

The GC analysis of the organochlorines pesticide residues was conducted using a model 2010 Shimadzu GC equipped with an EC. Separation was done on an SGE BPX-5 of 60 m capillary column with 0.25 mm internal diameter and 0.25 µm film thicknesses, equipped with 1 m retention gap. The oven temperature was programmed as follows: initial temperature was set at 90 °C for 3 min and ramped at 30 °C/min to 200 °C for 15 min and then to 265 °C at a rate of 5 °C/min for 5 min then to 275 °C at the rate of 3 °C/min and allowed to stay for 15 min. The injector setting is a pulsed splitless mode with a temperature of 250 °C at a pressure of 1.441 bar. Pulsed pressure was 4.5 bar, pulsed time 1.5 min, purge flow of 55.4 mL/min with a purge time of 1.4 min. The detector temperature was 300 °C. Nitrogen was used as carrier gas at a flow rate of 30 mL/min. A Varian CP-3800 GC equipped with a Combi PAL Auto sampler was used to measure levels of the pesticide residues. The column used was a 30 m × 0.25 mm internal diameter fused silica capillary coated with VF-1701 (0.25 µm film). The oven temperature was programmed as follows: initial temperature was set at 65 °C for 2 min and ramped at 25 °C/min to 210 °C for 6 min and then to 230 °C at 20 °C/min and allowed to stay for 20 min. The injector setting is a pulsed splitless mode at a temperature of 230 °C. The detector temperature was 250 °C in “constant makeup flow” mode. Helium gas was used as carrier gas at a flow rate of 2 mL/min.

Result and Discussion

Results

Table 1 below is the morphometric characteristics of the fish species collected from the lake.

Table1. The Morphometric Characteristics, Average Length, Average Weight and Condition Factor of the Fish Species

Collection Sites	English Name	Scientific Name	Sample Size	Average Weight (g)	Ave Length (cm)	K – value
Wurbo Lake	Tilapia	<i>Tilapia Z</i>	3	102.21 ±0.02	12.75±0.03	4.17
	Cat Fish	<i>C. gariepinus</i>	3	189.43±0.03	16.82±0.01	4.38
	Mormyrids	<i>Mormyrus rume</i>	3	195.33±0.05	17.12±0.04	3.91

The figures 1 to 5 below are the results of the pesticide residues analysis in water, sediment and

fish samples collected from Kundi lakes. The results are measured in concentration (in ppm).

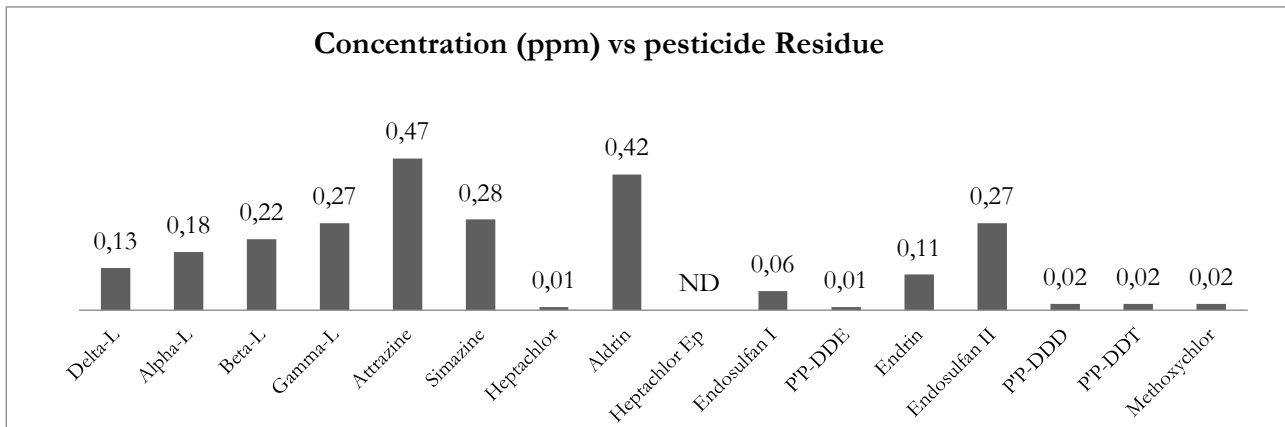


Figure 1. Concentration of pesticide residues in water

Note: The concentrations were in an increasing order as Atrazine > Aldrin > Simazine > Endosulfan II > Gamma- Lindane > Beta-Lindane.

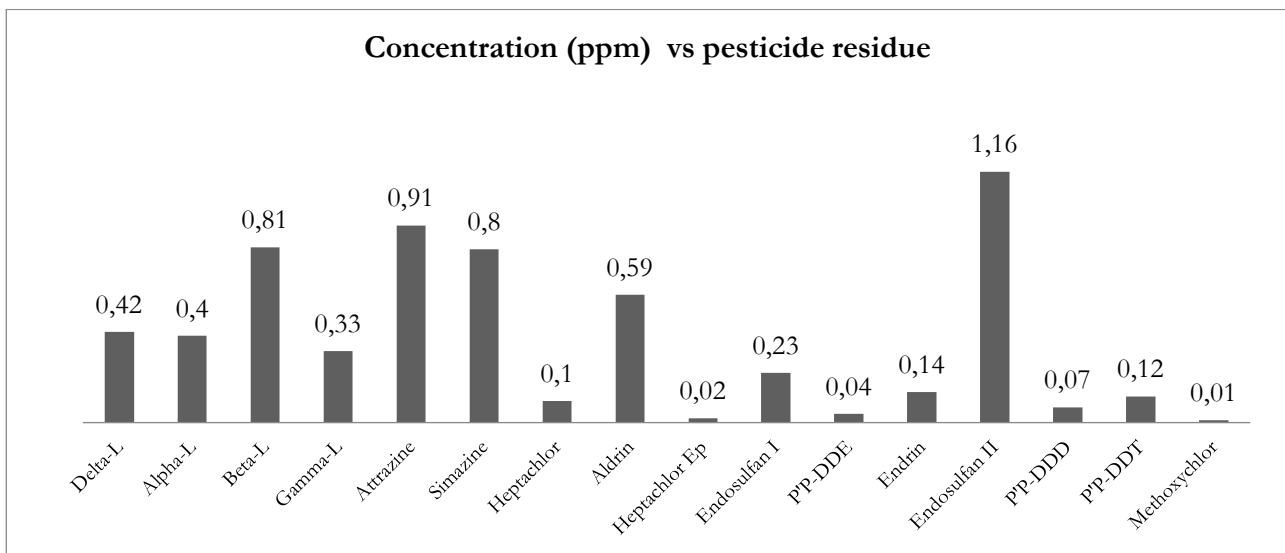


Figure 2. Concentration of pesticide residues in Tilapia Zilli

Note: The concentrations were in an increasing order as Endosulfan II > Atrazine > Beta-Lindane > Simazine > Aldrin.

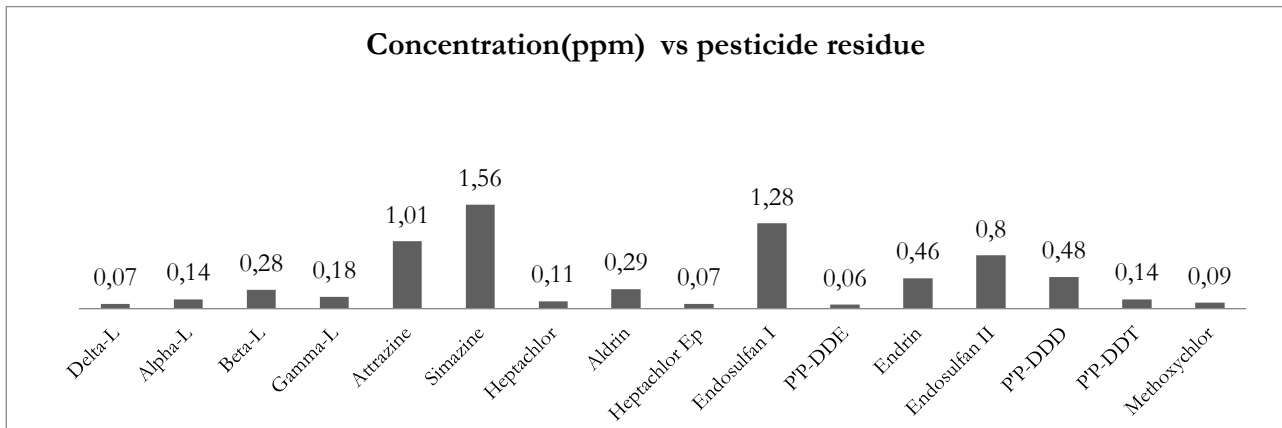


Figure 3. Concentration of pesticide residues in Clarias gariepinus

Note: The concentrations were in an increasing order as Simazine > Endosulfan I > Atrazine > Endosulfan II > Endrin.

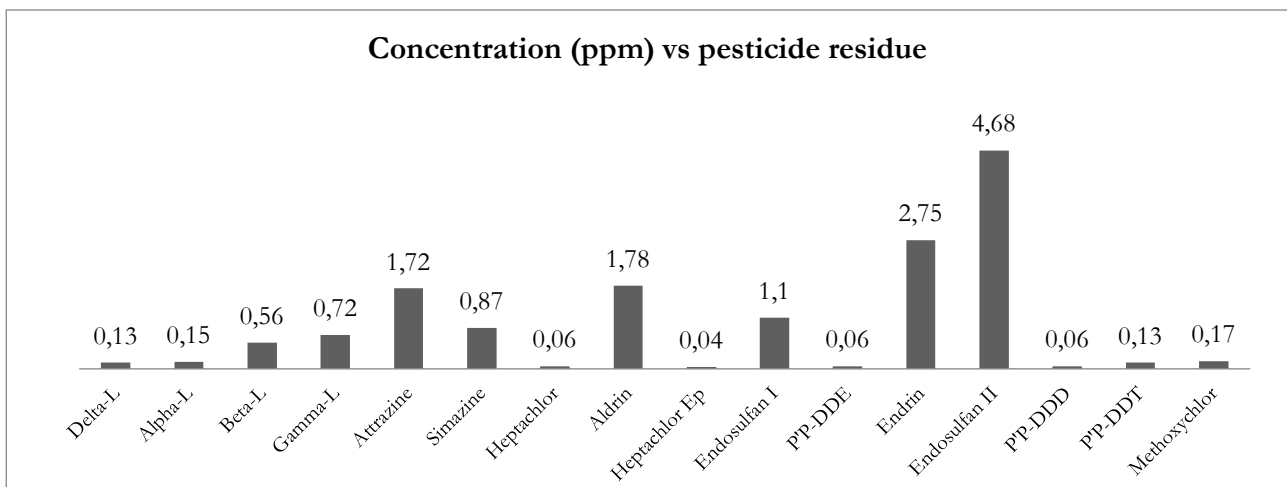


Figure 4. Concentration of pesticide residues in Mormyrus rume

Note: The concentrations were in an increasing order as Endosulfan II > Endrin > Atrazine > Endosulfan I > Simazine.

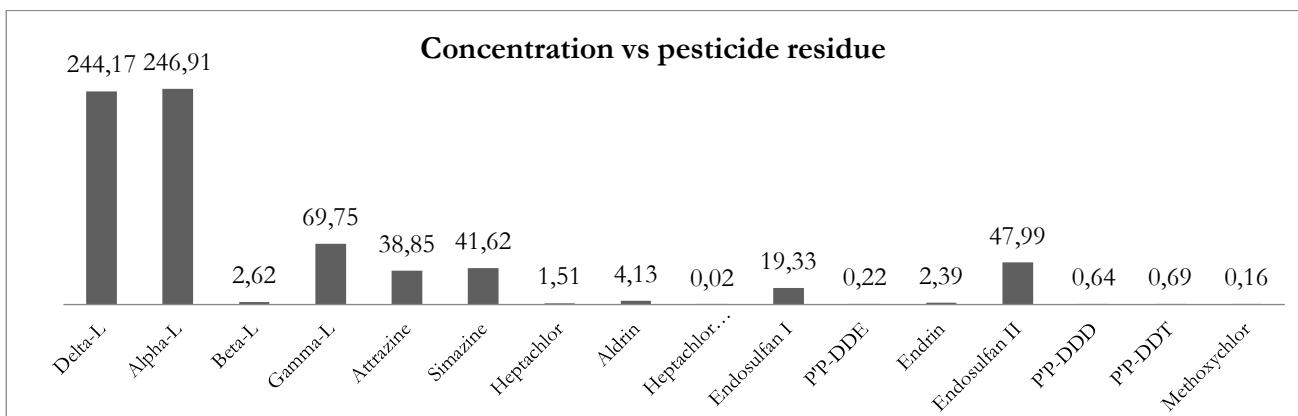


Figure 5. Concentration of pesticide residues in Sediment

Note: The concentrations were in an increasing order as Alpha Lindane > Delta Lindane > Gamma Lindane > Endosulfan II > Atrazine > Simazine.

Discussion

The average length, average weight and condition factor of the studied fish species were measured. The *Clarias gariepinus* *Mormyrus rume* had the highest average length of 17.12 ± 0.04 cm, and average weight of 195.33 ± 0.05 g follow by *Clarias gariepinus* which had the average length of 16.82 ± 0.01 cm, and average weight of 189.43 ± 0.03 g. *Tilapia zilli* was the least species of fish sample with a length of 12.75 ± 0.03 cm and average weight of 102.21 ± 0.02 g. The *Clarias gariepinus* had highest condition factor of 4.38, follow by *Tilapia zilli* which recorded the K-factor of 4.17 while the *Mormyrus rume* had the least K- factor of 3.91. The condition factor (K) gives information on the physiological condition of fish in relation to its welfare (Ujjania *et al.*, 2012). Ayelaja *et al.*, (2014) reported that fishes with a low condition index are presumably believed to have experienced adverse physical environment or insufficient nutrition (Ujjania *et al.*, 2012). The K value range of 2.9 to 4.8 was recommended as suitable for matured fresh water fish. all of the fish species sampled in this present study had condition factors > 1 , and were within the normal ranges as recommended by (Swati, 2015), who stated that condition factor greater or equal to one is good, indicating a good level of feeding, and proper environmental condition.

Based on the result of the analysis of the OCP residue on the water samples collected as indicated in figure 1, the Atrazine (0.47 ppm) was found to be higher in, follow by Aldrin (0.42 ppm) and Simazine (0.29 ppm). The lowest pesticide residues was recorded in p,p'-DDD, p,p'-DDT, p,p'-DDE, Heptachlor, and Methoxychlor. The Heptachlor epoxide was not detected in the water sample collected. The result also indicted that the levels of pesticide residues in water are generally lower because these pesticides are lipophilic and less soluble in water (Fosu-Mensah *et al.*, 2016). Also, the concentration of pesticide residue in water was below maximum residue limit (MRL) as compared with (Essumang & Chokky, 2009; FAO/WHO 2009 and 2010) where the pesticide

residue limit ranges from 0.0001 to 0.5 ppm. In a similar study by Kuranchie-Mensah *et al.* (2011) found that over 80 % of pesticides detected in water samples were below the MRLs by FAO/WHO (2009). Tongo *et al.* (2019) also reported a very low concentration of pesticides residues in water (0.0006 to 0.001 ppm) in the water samples collected from Warri River. Another study by Modibbo *et al.*, (2019) also found that the pesticides residues in water range from 0.15 to 0.92 ppm from river Njuwa, Adamawa state.

The result of the fish analysis shows that the concentrations of OCP residues in fish were found to be higher than that of the water sample. All the sixteen (16) organochlorines pesticides analysed were found to presence in the fish samples collected. Based on the result obtained, it was also observed that these concentrations recorded high in *Mormyrus rume* follow by *Clarias gariepinus*, while the *Tilapia zilli* recorded low concentration of the pesticide residues. The Endosulfan II recorded high concentration of 4.68 ppm in *Mormyrus rume* follow by Endrin which recorded the concentration of 2.75 ppm, Aldrin (1.78 ppm) and Atrazine (1.72 ppm) all in *Mormyrus rume* in figures 4 above. In *Clarias gariepinus*, Simazine (1.56 ppm) follows by Endosulfan I (1.28 ppm) in figure 3 above. These values were above the maximum residue limits (MRL) by FAO/WHO (2009 and 2010). The lowest pesticide residues was recorded in p,p'-DDD p,p'-DDE, p,p'-DDD, Methoxychlor and Heptachlor epoxide. Also, the high concentration of OCP observed in *Mormyrus rume* and *Clarias gariepinus* might be attributed to the feeding mode of the fish (Fianko *et al.*, 2011). This result is corroborated by (Biego *et al.*, 2010) who related to habitation and feeding habit of *Clarias gariepinus* and *Mormyrus rume* to an increased concentration of pesticide residues compared with other fish species. UNEPA (2007), equally adds that pesticides accumulation in was due to their lipid content; this implies that due to the high lipid content in *Clarias gariepinus*, and *Mormyrus rume* more pesticide residues tend to be trapped in their lipid stores.

The current study discovered high levels of pesticides residues in fish samples collected and this result also agree with Kuranchie-Mensah *et al.* (2011) who found widespread of pesticides residues in fish samples (1.78 to 2,38 ppm) from the Volta Lake in Ghana. The high levels pesticides were as a result of excessive use of pesticide in the farming activities taking place in those areas. Similar studies was carried out by Devi *et al.*, (2013), who reported relatively higher concentration of pesticides in fish tissues (1.43 to 2.93 ppm) as was detected in this study. Another finding by Tongo *et al.*, (2019) where very low concentrations of pesticides residues which ranges from 0.0022 to 0.0048 ppm for *Tilapia zilli* and 0.0046 to 0.0049 ppm for *Clarias gariepinus*.

The level of OCP residues in sediment analysed was higher than that of both the water and fish samples. All the sixteen (16) organochlorine pesticides analysed were detected in the sediment sample collected as indicated in figure 5 above. Alpha-Lindane recorded very high concentration of 246.91 ppm follow by Delta Lindane which recorded the concentration of 244.17 ppm, Gamma Lindane recorded the concentration of 69.75 ppm and Endosulfan II recorded the concentration of 47.99 ppm. The lowest pesticide residue was recorded only in p,p'-DDE, Methoxychlor and Heptachlor epoxide which were below the maximum residue limits (MRL) by FAO/WHO (2010). The concentrations of OCP residues in the sediment obtained in this study were higher when compared with the finding obtained by Akan (2013) reported the concentration of OCP in the range of 19.87 to 25.59ppm. In similar investigations with high OCP residues in the range from 2.16 to 567.49 ppm was reported by Malik *et al.*, (2008) from Gomti River, India. However, these OCPs residues were measured high in sediment samples. Sediments serve as reservoir for pollutants like pesticides and heavy metals. Pesticides are also less soluble in water (Fosu-Mensah *et al.*, 2016). The lake sediments act as a sink for the persistent contaminants, whose resuspension during the lake's mixing may increase pesticide bioavailability and accumulation in the fish (Bhandari, *et al.*, 2020).

Pesticide pollution to the lake is therefore, likely to pose a danger to both aquatic organisms and humans (Mavura & Wangila, 2014).

Conclusion

This study has reveal concentrations of organochlorine pesticides residues in water, sediments and three species of fish from Wurbo Lake was successfully investigated. The result indicated that the pesticides residues were recorded low in water as compared to that of fish and sediments which was also below the WHO/FAO values. It was also observed that these concentrations recorded high in *Mormyrus rume*, follow by *Clarias gariepinus*, while the *Tilapia zilli* recorded low concentration of the pesticide residues. The high levels of these pesticides may pose a great danger when these fish are being consumed over time. Regular monitoring is therefore required to control the levels of pesticide residues in the water bodies.

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