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# Identification and Quantification of Organochlorine Pesticides Residues in Water, Sediment and Fish Species from Central Mayokam and Suntai Rivers of Central Taraba State, Nigeria

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## *Abstract*

Pesticides use has increased worldwide, particularly due to the low yield of food production as a result of harm caused by pests and unwanted weeds. As a result, residues of these chemical contaminate the environment. Water, sediment and fish (*Tilapia zilli*, *Clarias gariepinus*, *Mormyrus rume*) samples were collected from Mayokam and Suntai river for the determination of levels of organochlorine pesticide residues. The extraction process was performed using Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) method and GC-MS for separation and detection of pesticides residues. The morphometric and condition factor of the studied fish species were measured. All the fish species sampled in this present study had condition factors > 1. A total of fourteen (14) organochlorine pesticide residues were detected. From the results obtained, the pesticides residues were recorded low in water as compared to that of fish and sediments. Based on the result of the water sample, Beta-lindane recorded higher concentration of 0.39 ppm from Mayokam, follow by Endosulfan II, 0.33 ppm from Suntai and Aldrin, 0.16 ppm from Mayokam River. In the fish samples, Endosulfan II recorded high concentration of 6.02 ppm in *Mormyrus rume*, 2.56 ppm in *Clarias gariepinus* and 2.03 in *Tilapia zilli* from Suntai river follow by Aldrin which recorded the concentration of 2.57 ppm in *Mormyrus rume* and 1.43 ppm in *Tilapia zilli* also from Suntai river. Endrin recorded 2.56 ppm in *Clarias gariepinus* from Mayokam river. Furthermore, about 65% of these residues in fish and sediment were above the MRL values set by World Health Organisation and Food and Agricultural Organisation

(WHO/FAO). The results indicate that there is a high level of contamination of these pesticides in fish 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.

**Key word:** Organochlorine, pesticide, Contamination, Environment, QuEChERS, GC-MS

## *Introduction*

Pesticides use has increased worldwide, particularly in the effort to meet the world demand on food supply to the ever increasing global population (Akoto *et al*., 2013; Mshelia *et al*., 2022). Therefore, large scale agricultural production gains can only be achieved by using adequate pesticides in controlling various pests and unwanted weeds (Parra-Arroyo et al., 2022). They are further used on crops post-harvest to prolong storage life and improve quality and in public health to control vector-borne diseases as well as unwanted plants such as grass and weeds in ornamental landscaping, parks and gardens (Gilden *et al*., 2010). The rapid increase in the cost of farm labour, pressure to intensify land productivity as well as greater availability of these chemicals in the markets are also some of the factors driving the increase use of the pesticides (Opuni *et al*., 2021; G. Wang *et al*., 2022). Despite the good results of using pesticides in agricultural and public health, their use is usually accompanied with harmful environmental and public health effect (Blasco *et al.,* 2011; Cieslik *et al*., 2011; Eunice & Francis, 2024; Parra-Arroyo et al., 2022; Thurman *et al*., 2000). Pesticides enter and pollute any component of the environment in a number of ways, including application through agricultural activities, industrial plants and municipal sewage treatment plant, accidental spillage or through the unauthorized dumping of pesticide products or their containers (Akdogan *et al.,* 2014 & Cox, 2002; Kaushal *et al*., 2021; Lofrano *et al*., 2020).

Pesticides hold a unique position among environmental contaminants due to their high biological activity and toxicity (Chang, 2018). Although some pesticides are selective in their mode of operation, even though their selectivity is only limited to test animals or weeds. According to Llorent-Martínez *et al*., (2011), more than 98% of the insecticides and 95% of the herbicides sprayed do not reach their final destination, which are the target species. Slow degradation of theses pesticides especially organochlorine in the environment can lead to contamination and endangering the aquatic life in water, soil, air, crops, and plants and indirectly to human (Cieslik *et al*., 2011; Hamilton & Crossley, 2004). In addition, pesticide use reduces biodiversity, contributes to pollinator decline (Wells, 2007), destroy habitat (especially for birds) and threatens endangered species (Millet & Spoolman, 2004). Researches have shown that each year, about 2.5 million tons of pesticides are released and 50% of them penetrate to soil, water and bodies of living

creatures (Rao & Northup, 2009; Teklit, 2016). Furthermore, pesticide is considered as the second causes of mortalities after infectious diseases in the developing countries (Zakari *et al*., 2017). Recently, deaths of over 18 people occurred in Ode Irele, Ondo State, Nigeria, as a result of pesticide poisoning (Eunice & Francis, 2024). Additionally, there were reports of children being poisoned in Zhejiang, China (Yimaer et al., 2017).

Organochlorine pesticides (OCPs) are considered as one of the most important group of persistent organic pollutants which have caused global concern as toxic environmental contaminants (Awe et al., 2022; Covacia *et al*., 2005). They are mainly classified into three categories; namely diphenylaliphatics such as DDT and its metabolites, cyclodienes which include aldrin, dieldrin, heptachlor, endosulfan and endosulfan sulfate (Nur-Banu & Semra, 2004). These compounds resist degradation and remain in the environment for years (yadav *et al*., 2015). They are also noted for their long half-lives and their potential to bioaccumulate in aquatic organisms especially fish, through food chain transfer (Afful *et al*., 2010). There has been increasing evidence that human exposure to OCPs is linked to some health problems (Joseph *et al.*2019). Health impacts can range from acute, such as headaches, dizziness, and nausea to chronic like cancer, immunosuppression, reproductive and developmental disorders (Garabrant *et al*., 1992; Kaur *et al.,* 2019; Parra-Arroyo et al., 2022; Terry, 2012). Recently World health Organisation reports that about 80 % of all human illnesses in the developing world are related to biological contaminations (FAO/WHO, 2009 &2010).

Contamination of water bodies is a major concern for fish and other aquatic organisms which are the major sources of protein (Essumang *et al.,* 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). Furthermore, contaminant founds in fish is a reflection of the state of pollution in surrounding environments (Guo *et al*., 2008; Lan Franchi *et al.,* 2006; Mshelia *et al*., 2022). Therefore, 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 (Das *et al*., 2002 & Lan franchi *et al*., 2006). The sediments also 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 taking up by living organisms (Walley *et al.,* 2006 & Akan *et al.,* 2013).

Taraba State, specifically Bali in the North-East Nigeria, is known for its agricultural activities where the used pesticide has increased significantly in the recent time without looking at the environmental contamination it may cause in such areas. Also, the pesticide handlers (sellers, applicators and farmers) are largely not knowledgeable about the composition and nature of the pesticides they handle and the use of monitoring tools to assess pesticides in the environment is still in its early stages. Furthermore, there have been a few researches focusing on pesticides residues, despite their significance and high toxicity. Therefore, the objective of this study was to identify individual organochlorine pesticide residues and examined the extent of contamination in water, fish and sediment samples.

**Study Site**

The study was carried out at Mayokam and Suntai rivers of Bali Local Government Area of Taraba State, Nigeria. Mayokam lies between latitude 8°14 N and 52° N of the equator and longitude 11° 03 E and 05° E of the prime meridian. Suntai lies between latitude 7°54 N and 30° N of the equator and longitude 10° 22 E and 49° E of the prime meridian. The two locations are found in dry guinea savannah and are located within Bali which is the largest local Government in Taraba State, with an estimated land area of 11,540 $km^2$. Suntai and Mayokam have 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 occupation of the inhabitants is farming, fishing and nomadism.

**Water Sample Collection**

Water sample was collected from the locations within a depth of 20 cm into three separate pre clean 1 L plastic containers from different locations at appropriate depth and turbulent midstream positions of water bodies. These include the water inlet, center and the outlet (spillway). Prior to labeling, the samples was wrapped in aluminum foil and preserved in ice box at a temperature of 4±2° C to minimize degradation of pesticide and thereafter conveyed to the laboratory.

**Sediment Sample Collection**

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 and effort was made to eliminate any stones, roots, debris, foreign objects and shells from the sample. The measured quantity of the sample was placed into suitable polyethylene bags, wrapped in aluminum foil labeled and stored in a cooler ice box at a temperature of 4±2° C while in the field, and thereafter conveyed to the laboratory until analysis.

**Fish Sample Collection**

The fish samples (n = 4 per each specie) were collected from two (2) rivers. Fish identified and selected for this project are tilapia (*Tilapia zilli*), Catfish *(Clarias gariepinus*), and Mormyrids (*Mormyrus rume*). Species were among the commonly consumed fishes from the rivers. The samples were collected randomly from fishermen catches at the landing sites between 9:00 am – 11:00 am. Only visually healthy, undamaged fish were collected to minimize confounding effects of disease or injury on pesticide accumulation. The sample was wrapped in aluminum foil, stored in a cooler ice box at a temperature of 4±2 C while in the field, and thereafter conveyed to the laboratory for preservation at -20 $^0$ C prior to analysis. The measurements of the total length (cm) of each fish were taking from the tip of snout (mouth close) to the end of caudal fin using meter rule, body weight (g) was measured using electronic digital balance and the condition factor of individual fish sampled were calculated

and recorded. The condition factor was calculated using the formula describe by Ujjania *et al*. (2012).

Condition Factor, $K = 100W/L^3$ (1)

Where *W* is weight in gram and *L* is total length in cm.

**Analytical Quality Assurance**

Appropriate quality assurance procedures and precautions were taken to ensure the authenticity of the results. Samples were carefully handled to avoid cross-contamination. Glass-wares were properly cleaned and deionized water was used throughout the study. All the reagents used were of analytical grade**.** The quality of the analysis of pesticide residues was enhanced through solvent blank and spikes. The solvent blanks were used to check for interference from reagent, while the spike samples were used to determine the recovery which is an indication of method performance and accuracy.

**Sample preparation**

Before extraction of pesticide residues, the water sample was filtered using 0.45 µm fibre glass filters (manufactured by Whatman) to eliminate any suspended particles. The sediment sample undergoes the process of air drying and subsequent sieving using a 250 µm stainless steel mechanical shaker. The fish samples were thawed and cleaned with distilled water. Muscle tissues were dissected, minced into smaller pieces and homogenized (Ayeloja *et al.,* 2014). A modified Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) method was used for extraction and Gas Chromatography- Mass spectrometer (GC-MS) was used for detection and separation of pesticide residues.

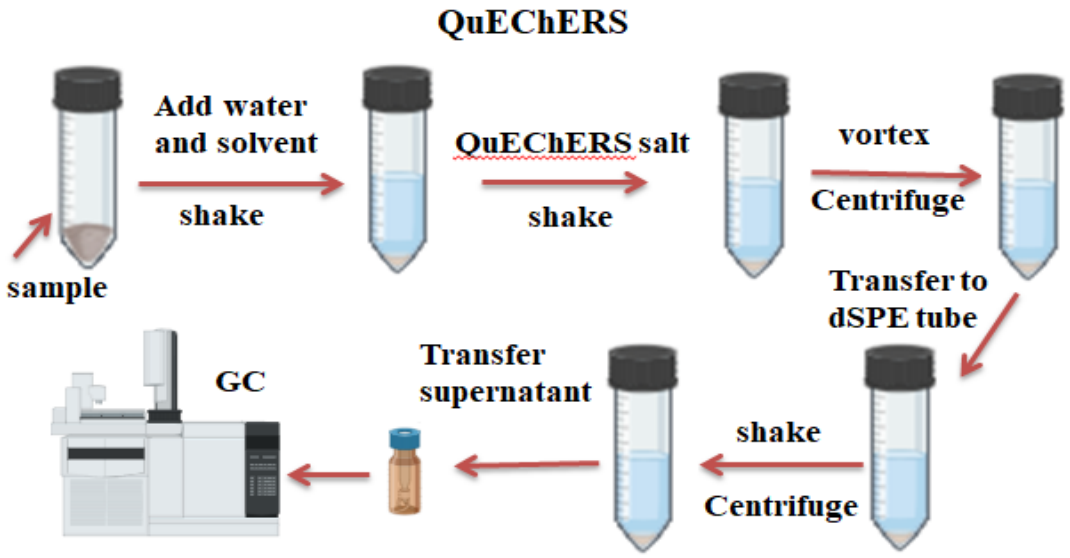



**Figure 1;** Schematic diagram of Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS)

**QuEChERS Extraction of Pesticide Residues in water**

A modified QuEChERS method was followed in sample preparation. 10 ml water samples were placed in a 15 ml centrifuge tube and 2.5 ml dichloromethane was added and shaken vigorously. Tubes were capped and vortexed for 3 min before adding 4 g MgSO4, 1.5g Sodium acetate and 1 g NaCl were added. The tubes were votexed vigorously for 1 min and centrifuged for 10 min at 7000 rpm. One milliliter of the supernatant was transferred into a 2 mL vial containing 500 mg MgSO. The vials were shaken and subsequently centrifuged. The extract was transferred into a PTFE capped vial (obtained from Agilent GCMS) for GC–MS analysis.

**QuEChERS Extraction of Pesticide Residues in fish**

Five (5 g) fish sample was mixed with 10 ml acetonitrile in a 15 ml centrifuge tube. The mixture was shaken vigorously. 4 g activated anhydrous $MgSO_4$, 1.0 g activated NaCl were added and shaken for 10 min with vortex mixer at 30 rpm on rotospin. The extract was centrifuged for 10 min at 8,000 rpm and then 1 ml supernatant of extract was cleaned with the mixture of 50 mg PSA, 150 mg anhydrous $MgSO_4$ and 10 mg activated charcoal (application of activated charcoal in the stage of clean up to remove pigments from matrix). The extract was again shaken for 10 min at 30 rpm on rotospin and centrifuged for 10 min at 8,000 rpm. The supernatant was collected in GC vial for the analysis.

**QuEChERS Extraction of Pesticide Residues in sediment**

A 5 g of sediment sample was placed in a 15 ml centrifuge tube and 10 ml acetonitrile was added. The mixture was shaken vigorously. Further, 1.5 g trisodium citrate dihydrate, 4 g anhydrous magnesium sulphate, and 1 g sodium chloride were added. The mixture was hand-shaken for 1 minute using vortex mixer and then centrifuged for 10 min at 7000 rpm. Afterwards, a 7.5 ml aliquot of the supernatant was transferred to a centrifuge tube (15 ml) containing 0.75 g anhydrous magnesium sulphate, 0.5 g C18 and 0.125 g PSA. The tube was vortexed and centrifuged. The aliquot of the supernatant was transferred into a glass test tube and allowed to evaporate to nearly dryness under a stream of nitrogen and the residue was re-dissolved in 1.5 ml dichloromethane prior to its injection into the GC-MS system.

**Determination of Pesticides Residues in Fish, Sediment and water**

The GC analysis of the organophosphate 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 VF1701 (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.

**Statistical Analysis**

The statistical analysis of mean, error and Standard deviation for the data obtained was performed using IBS SPSS Statistic 31.

**Result**

Table I and 2 below are the morphometric measurement of the fish species collected from Mayokam and Suntai Rivers. These measurements were carried out to ensure that the fish analysed are matured since at these weights, the species are prone to accumulating quantities of contaminants such as pesticide residues in their tissues over a period of time. This can offer a precise depiction of the possible hazards associated with consuming fish that may harbour pesticide residues which can accumulate in their tissues from water, sediments, and food particles.

**Table1: The morphometric measurement; average length, average weight and condition factor of the fish species from Mayokam River.**

| Collection Sites | English Name | Scientific Name | Sample Size | Average Weight (g) | Ave Length (cm) | K – value |
|---|---|---|---|---|---|---|
| **Mayokam** | Tilapia | *Tilapia Z* | 4 | 102.71 ±1.54 | 13.30±0.81 | 4.31 |
| | Cat Fish | *C. gariepinus* | 4 | 203.12±2.00 | 16.51±1.48 | 3.97 |
| | Mormyrids | *Mormyrus rume* | 4 | 209.48± 1.15 | 17.15±0.94 | 4.46 |

**Table2: The morphometric measurement; average length, average weight and condition factor of the fish species from Suntai River.**

| Collection Sites | English Name | Scientific Name | Sample Size | Average Weight (g) | Ave Length (cm) | K – value |
|---|---|---|---|---|---|---|
| **Suntai** | Tilapia | *Tilapia Z* | 4 | 97.63 ±0.54 | 13.65±0.57 | 4.42 |
| | Cat Fish | *C. gariepinus* | 4 | 179.36±0.96 | 16.59±0.66 | 3.87 |
| | Mormyrids | *Mormyrus rume* | 4 | 196.75±1.31 | 16.46±0.15 | 4.37 |

The highest condition factor was observed in the *Mormyrus rume* (4.46) from Mayokam river (Table 1) follow by *Tilapia Zilli* (4.42) from Suntai River (Table 2). The least condition factor was observed in *Clarias gariepinus* (3.87) from Suntai River (Table 2).
Tables 3 to 6 below Show the concentration of the organochlorine pesticides residues in water, fish and sediment samples collected from Mayokam and Suntai.

**Table 3; Result of concentration of pesticide residues in water from Mayokam and Suntai River.**

| | **Mayokam River** | | **Suntai River** | | | | |
|---|---|---|---|---|---|---|---|
| **Pesticide Residues** | **RT (min)** | **Conc (ppm)** | **RT (min)** | **Conc (ppm)** | **Mean Conc (ppm)** | | **STD (±)** |
| **Delta-lindane** | 5.37 | 0.02 | 5.353 | 0.06 | 0.040 | | 0.003 |
| **Alpha-lindane** | 5.828 | 0.13 | 5.862 | 0.04 | 0.085 | | 0.064 |
| **Beta-lindane** | 6.051 | 0.39 | 6.034 | 0.1 | 0.245 | | 0.205 |
| **Gamma-lindane** | 6.577 | 0.05 | 6.56 | 0.07 | 0.060 | | 0.014 |
| **Heptachlor** | 7.098 | 0.01 | 7.041 | 0.01 | 0.010 | | 0 |
| **Aldrin** | 7.315 | 0.16 | 7.315 | 0.15 | 0.155 | | 0.007 |
| **Heptachlor epoxide** | 0 | ND | 0 | ND | 0 | | 0 |
| **Endosulfan I** | 8.717 | 0.03 | 8.632 | 0.03 | 0.030 | | 0 |
| **p,p'-DDE** | 8.98 | 0 | 9.181 | 0 | 0 | | 0 |
| **Endrin** | 9.467 | 0.03 | 9.467 | 0.05 | 0.025 | | 0.035 |
| **Endosulfan II** | 9.804 | 0.8 | 9.776 | 0.33 | 0.565 | | 0.332 |
| **p,p'-DDD** | 9.89 | 0.01 | 9.896 | 0.05 | 0.030 | | 0.028 |
| **p,p'-DDT** | 10.474 | 0.01 | 10.48 | 0.01 | 0.010 | | 0 |
| **Methoxychlor** | 11.48 | 0.01 | 11.502 | 0.01 | 0.010 | | 0 |

From above, the concentrations were in an increasing order as Beta-lindane > Endosulfan II > Aldrin > Alpha-lindane > Gamma-lindane > Delta-lindane > Endrin.

**Table 4; Result of concentration of pesticides residues in *Tilapia Zilli*, *Clarias gariepinus* and *Mormyrus rume* from Mayokam.**

| | **Mayokam River** | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | ***Tilapia Zilli*** | | ***Clarias gariepinus*** | | ***Mormyrus rume*** | | **Mean con** | **STD** |
| **Pesticide Residues** | **RT (min)** | **Con (ppm)** | **RT (min)** | **Con (ppm)** | **RT (min)** | **Con (ppm)** | **(ppm)** | **(±)** |
| **Delta-lindane** | 5.343 | 0.19 | 5.336 | 0.3 | 5.353 | 0.14 | 0.21 | 0.082 |
| **Alpha-lindane** | 5.845 | 0.22 | 5.862 | 0.44 | 5.485 | 0.16 | 0.273 | 0.147 |
| **Beta-lindane** | 6.039 | 0.31 | 6.034 | 0.83 | 6.034 | 0.29 | 0.477 | 0.306 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Gamma-lindane** | 6.566 | 0.8 | 6.583 | 0.18 | 6.566 | 0.1 | 0.36 | 0.383 |
| **Heptachlor** | 6.76 | 0.06 | 6.783 | 0.08 | 6.772 | 0.03 | 0.057 | 0.025 |
| **Aldrin** | 7.321 | 0.8 | 7.333 | 0.34 | 7.298 | 0.27 | 0.47 | 0.288 |
| **Heptachlor epoxide** | 7.985 | 0.07 | 8.019 | 0.07 | 7.986 | 0.05 | 0.063 | 0.012 |
| **Endosulfan I** | 8.637 | 1.12 | 8.62 | 1.11 | 8.649 | 0.81 | 1.013 | 0.176 |
| **p,p'-DDE** | 9.055 | 0.05 | 9.038 | 0.03 | 9.078 | 0.03 | 0.037 | 0.012 |
| **Endrin** | 9.444 | 0.22 | 9.45 | 2.56 | 9.473 | 0.75 | 1.177 | 1.227 |
| **Endosulfan II** | 9.776 | 1.72 | 9.787 | 1.69 | 9.799 | 0.69 | 1.367 | 0.586 |
| **p,p'-DDD** | 9.89 | 0.03 | 9.907 | 0.15 | 9.913 | 0.09 | 0.09 | 0.06 |
| **p,p'-DDT** | 10.491 | 0.02 | 10.502 | 0.13 | 10.497 | 0.14 | 0.097 | 0.067 |
| **Methoxychlor** | 11.492 | 0.05 | 11.492 | 0.13 | 11.504 | 0.09 | 0.09 | 0.04 |

From above, the concentrations were in an increasing order as Endrin > Endosulfan II > Endosulfan I > Beta-lindane > Alpha-lindane > Aldrin > Endrin > Delta-lindane.

**Table 5; Result of concentration of pesticides residues in *Tilapia Zilli*, *Clarias gariepinus* and *Mormyrus rume* from Suntai River.**

| | **Suntai River** | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | ***Tilapia Zilli*** | | ***Clarias gariepinus*** | | ***Mormyrus rume*** | | | | |
| **Pesticide Residues** | **RT (min)** | **Con (ppm)** | **RT (min)** | **Con (ppm)** | **RT (min)** | **Con (ppm)** | **Mean (ppm)** | **Con** | **STD (±)** |
| **Delta-lindane** | 5.353 | 0.14 | 5.347 | 0.14 | 5.359 | 0.05 | 0.11 | | 0.052 |
| **Alpha-lindane** | 5.862 | 1.23 | 5.856 | 0.36 | 5.856 | 0.15 | 0.58 | | 0.573 |
| **Beta-lindane** | 6.057 | 0.77 | 6.028 | 0.22 | 6.057 | 1.24 | 0.743 | | 0.511 |
| **Gamma-lindane** | 6.589 | 0.42 | 6.571 | 0.4 | 6.566 | 0.58 | 0.467 | | 0.099 |
| **Heptachlor** | 6.772 | 0.07 | 6.755 | 0.02 | 6.738 | 0.05 | 0.047 | | 0.025 |
| **Aldrin** | 7.377 | 1.43 | 7.304 | 0.84 | 7.333 | 2.57 | 1.613 | | 0.879 |
| **Heptachlor epoxide** | 8.985 | 0.05 | 7.996 | 0.03 | 8.008 | 0.08 | 0.053 | | 0.025 |
| **Endosulfan I** | 8.603 | 0.33 | 8.631 | 0.14 | 8.62 | 0.71 | 0.393 | | 0.29 |
| **p,p'-DDE** | 9.078 | 0.01 | 9.078 | 0.01 | 9.061 | 0.02 | 0.013 | | 0.006 |
| **Endrin** | 9.455 | 0.08 | 9.461 | 0.18 | 9.461 | 1.11 | 0.457 | | 0.568 |
| **Endosulfan II** | 9.793 | 2.03 | 9.793 | 2.56 | 9.782 | 6.02 | 3.537 | | 2.167 |
| **p,p'-DDD** | 9.903 | 0.07 | 9.902 | 0.17 | 9.942 | 0.08 | 0.107 | | 0.055 |
| **p,p'-DDT** | 10.503 | 0.06 | 10.491 | 0.13 | 10.503 | 0.02 | 0.07 | | 0.056 |
| **Methoxychlor** | 11.493 | 0.04 | 11.51 | 0.02 | 11.487 | 0.06 | 0.04 | | 0.02 |

From above, the concentrations were in an increasing order as Endosulfan II > Aldrin > Beta-lindane > Alpha-lindane > Endrin > Endosulfan I > Gamma-lindane.

**Table 6; Result of concentration of pesticide residues in sediment from Mayokam and Suntai River.**

| | Mayokam River | | Suntai River | | | | |
|---|---|---|---|---|---|---|---|
| **Pesticide Residues** | **RT (min)** | **Conc (ppm)** | **RT (min)** | **Conc (ppm)** | **Mean (ppm)** | **Conc** | **STD (±)** |
| **Delta-lindane** | 5.37 | 0.34 | 5.364 | 0.23 | 0.285 | | 0.078 |
| **Alpha-lindane** | 5.862 | 4.89 | 5.845 | 0.59 | 2.740 | | 3.041 |
| **Beta-lindane** | 6.022 | 2.84 | 6.034 | 0.65 | 1.745 | | 1.549 |
| **Gamma-lindane** | 6.577 | 0.63 | 6.56 | 0.29 | 0.460 | | 0.24 |
| **Heptachlor** | 6.789 | 0.15 | 6.789 | 0.15 | 0.150 | | 0 |
| **Aldrin** | 7.289 | 0.68 | 7.304 | 0.43 | 0.555 | | 0.177 |
| **Heptachlor epoxide** | 7.955 | 0.03 | 8.122 | 0.01 | 0.345 | | 0.474 |
| **Endosulfan I** | 8.654 | 3.58 | 8.637 | 0.15 | 1.865 | | 2.425 |
| **p,p'-DDE** | 9.078 | 0.02 | 9.06 | 0.01 | 0.015 | | 0.007 |
| **Endrin** | 9.467 | 0.99 | 9.467 | 0.17 | 0.580 | | 0.579 |
| **Endosulfan II** | 9.787 | 1.1 | 9.799 | 0.23 | 0.665 | | 0.615 |
| **p,p'-DDD** | 9.919 | 0.32 | 9.896 | 0.04 | 0.180 | | 0.198 |
| **p,p'-DDT** | 10.502 | 0.17 | 10.485 | 0.05 | 0.110 | | 0.084 |
| **Methoxychlor** | 11.504 | 0.03 | 11.492 | 0.02 | 0.025 | | 0.007 |

From above, the concentrations were in an increasing order as Alpha-lindane > Endosulfan I > Beta-lindane > Endosulfan II > Endrin > Alpha-lindane > Gamma-lindane > Delta-lindane.

**Morphometric measurement and condition factor**

The morphometric measurement such as average length, average weight and condition factor of the studied fish species was measured. The *Mormyrus rume had* the highest average length of 17.15±0.94 cm, and average weight of 209.48 ± 1.15 g follows by *Clarias gariepinus* which had the average length of 16.51 ± 1.48 cm and average weight of 203.12 ± 2.00 g both from Mayokam River (Table 1). *Tilapia zilli* was the least species of fish sample with a length of 13.63 ± 0.57 cm and average weight of 97.63 ±0.54 g from Suntai River (Table 2). All 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. The condition factor (K) gives information on the physiological condition of fish in relation to its welfare (Ujjania *et al.,* 2012). The K value range of 2.9 to 4.8 was recommended as suitable for matured fresh water fish. Ayeloja *et al* (2014) reported that fishes with a low condition index are presumably believed to have experienced adverse physical environment or insufficient nutrition which may result in low accumulation of contaminants as these

chemicals are lipophilic in nature. In this study, the *Mormyrus rume* had the highest condition factor of 4.46 from Mayokam river (Table 1) follow by *Tilapia zilli* which recorded the K-factor of 4.42 from Suntai river (Table 2) while the *Clarias gariepinus* had the least K- factor of 3.87 also from Suntai river (Table 2). The high k value of the studied fish showed that the fish are matured enough for experimental analysis.

**Pesticide Residues in water Samples**

In this study, a total of fourteen (14) organochlorine pesticide (OCPs) residues were analyzed which include Delta-lindane, Alpha-Lindane, Beta-Lindane, Gamma-Lindane, Heptachlor, Endrin, Aldrin, Heptachlor Epoxide, Endosulfan I, Endosulfan II, P'P-DDE, P'P-DDD, P'P-DDT and Methoxychlor. Based on the result of the water sample, Beta-lindane (0.39 ppm) showed higher concentration from Mayokam, follow by Endosulfan II (0.33 ppm) from Suntai and Aldrin (0.16 ppm) from Mayokam river (Table 3). The lowest pesticide residues recorded were Endosulfan I, Endrin, p,p'-DDD, p,p'-DDT, Heptachlor, and Methoxychlor. These values were below the maximum residue limit (MRL) as compared with FAO/WHO (2009 and 2010) where the pesticide residue limit ranges from 0.0001 to 0.5 ppm. The Heptachlor epoxide and p,p'-DDE were not detected in the water sample collected. 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, Delta, Nigeria. Another study by Modibbo *et al.,* (2019) also found the relatively low pesticides residues in water range from 0.15 to 0.92 ppm from river Njuwa, Adamawa, Nigeria. The low levels of OCPs residues in water were due to the fact that organochlorines are scarcely soluble in water (Fosu-Mensah *et al*., 2016; Imo *et al*., 2013).

**Pesticide Residues in Fish Samples**

Fourteen (14) organochlorine pesticide (OCPs) residues were also analyzed and detected in the fish samples collected. The results of the fish analysis shows that the concentration of OCPs detected showed slightly higher levels compared to those in water sample (Table 4 and 5). Furthermore, all the fourteen (14) organochlorine pesticides analyzed were detected in the fish samples collected. It was found that the levels of organophosphates pesticides residues in fish samples were higher in *Mormyrus rume* follow by *Clarias gariepinus* and *Tilapia zilli*. The Endosulfan II recorded high concentration of 6.02 ppm in *Mormyrus rume*, 2.56 ppm in *Clarias gariepinus* and 2.03 ppm in *Tilapia zilli* from Suntai river follow by Aldrin which recorded the concentration of 2.57 ppm in *Mormyrus rume* and 1.43 in *Tilapia zilli* also from Suntai river (Table 5). Endrin recorded 2.56 ppm in *Clarias gariepinus* from Mayokam river (Table 4). Furthermore, Endosulfan I recorded the concentration of 1.12 ppm in *Tilapia zilli*, 1.11 ppm in *Clarias gariepinus* and 0.81 ppm in *Mormyrus rume* from Suntai river (Table 5). These values were above the maximum residue limits (MRL) by FAO/WHO (2009 and 2010). The p,p'-DDE, p,p'-DDD, Methoxychlor, Heptachlor and Heptachlor epoxide recorded lowest

concentration. The high concentration of OCPs observed in *Mormyrus rume* and *Clarias gariepinus* might be attributed to the feeding mode of the fish (Fianko *et al.,* 2011). Furthermore, Biego *et al*., (2010) states that the high concentration of pesticides residues in *Clarias gariepinus* and *Mormyrus rume* compared with other fish species was as a result of their feeding habit and high lipid content in them as pesticides can easily be trapped in their lipid stores (UNEPA, 2007). The current study agrees with the finding by Kuranchie-Mensah *et al*. (2011) who recorded widespread of pesticides residues in fish samples (1.78 to 2.38 ppm) from the Volta Lake in Ghana. 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) who reported 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*.

**Pesticide Residues in Sediment Samples**

Fourteen (14) organochlorine pesticide (OCPs) residues were analyzed and detected in the sediment samples. The level of OCPs residues in sediment analysed was also higher than that of the water. Also, all the fourteen (14) organochlorine pesticides analyzed were detected in the sediment samples collected (Table 6). Alpha-Lindane recorded very high concentration of 4.89 ppm follow by Endosulfan 1 which recorded the concentration of 3.58 ppm and Beta-lindane 2.84 ppm all from Mayokam river (Table 6). The lowest pesticide residues recorded were p,p'-DDE, Heptachlor epoxide, and Methoxychlor which below the maximum residue limit recommended by FAO/WHO (2009 and 2010). The current study agrees with the finding by Akan (2013) who reported the concentration of OCPs in the range of 19.87 to 25.59 ppm. In similar investigations, high levels OCPs residues in the range of 2.16 to 567.49 ppm were reported by Malik *et al*., (2009) from Gomti River, India. However, high levels of OCPs in fish and sediment was anticipated as these chemicals are hydrophobic in nature and therefore they are prone to concentrating in fatty tissues of fishes and sediment. Furthermore, the 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).

**Conclusion**

With increasing in population followed by increasing demand of food, the use of pesticide has also increased significantly in the development countries like Nigeria. Furthermore, this study is the first to document a detailed analysis of OCPs occurrences in water, fish and sediment in the area by providing various levels of these contaminants. This research has identified the presence of persistent, bio-accumulative and toxic pesticide residues in water, fish and sediment at levels that raise public health concerns. From the above results obtained, a total of fourteen (14) organochlorine pesticide (OCPs) residues were detected. The pesticide residues were recorded low in water as compared to that of fish and sediments. The concentrations of

these residues recorded high in *Mormyrus rume*, follow by *Clarias gariepinus* and *Tilapia zilli*. Also, about 65% of these residues in fish and sediment were above the MRL values set by WHO/FAO. The prolonged uptake of these pesticides through food chain can be stored in the human body and may cause severe health hazards. The presence of these suspected pesticide residues are alarming for the people living in this area. Therefore, regular monitoring is required to control the levels of pesticide residues in the water bodies to in order to reduce environmental pollution. Farmers should also be sensitized on the safe use and danger of pesticides. Furthermore, they should be encouraged to use different types of alternate measures instead of pesticides to protect the crop losses due to pests attack.

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**Disclosure of conflict of interest**

The authors declare that they have no conflict of interest.

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