
MODIFY QUECHER'S EXTRACTION METHOD FOR THE DETERMINATION OF ORGANOPHOSPHATE PESTICIDE RESIDUES IN FISH SAMPLES COLLECTED FROM WURBO AND YELWA LAKES OF BALI, TARABA STATE, NIGERIA

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Abstract

Organophosphate pesticides (OPPs) have been used worldwide, particularly in many African countries especially in Nigeria for the control of pests and weeds for agricultural purposes. Despite the positive outcomes of using pesticides in agriculture, their usage often results in various harmful environmental and health effects on water systems and aquatic organisms. Pesticides, due to their toxicity and impact on biological components, can be termed as biocides—that is, compounds capable of harming all forms of living organisms, including their intended targets. This research was aimed at determining the residual levels of organophosphates pesticides residues in fish species from Wurbo and Yelwa lakes to find out the extent of contamination in the area. The extraction process was performed using QuEChERS method. The extracts were later analyzed for organophosphates pesticide residues using GC-MS. The result showed that the levels of organophosphates pesticides residues in fish samples were higher in *Mormyrus rume* follow by *Clarias gariepinus* while the *Tilapia zilli* recorded low concentration. The total of five organophosphate were recorded.

Chlorpyrifos recorded high concentration of 1026.46 ppm in *Mormyrus rume* from Wurbo lake and 691.92 ppm from Yelwa lake. In *Clarias gariepinus*, Chlorpyrifos recorded the concentration of 584.45 ppm in Wurbo and 359.41 ppm from Yelwa lake. Ronel recorded levels of 42.53 in *Clarias gariepinus* and 5.56 ppm in *Mormyrus rume* from Wurbo. Phosphorodithioic acid recorded the concentration of 8.08 ppm in *Clarias gariepinus* and 4.84 ppm in *Mormyrus rume* from Wurbo lake. These values were above the maximum residue limits (MRL) by FAO/WHO, (2009, 2010 & 2021), ranges from 0.001 to 0.5ppm. 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.

Keywords: Fish, Quenchers, Organophosphate, Residue, Wurbo, Yelwa

Introduction

Pesticides play an important role in the control of wide range of human and livestock disease vectors, (Knobler *et al.*,2003 & Lanfranchi *et al.*,2006) and in the increase of crop yields. Despite the positive outcomes of using pesticides in agriculture, their usage often results in various harmful environmental and health effects on water systems and aquatic organisms. Pesticides, due to their toxicity and impact on biological components, can be termed as biocides—that is, compounds capable of harming all forms of living organisms, including their intended targets. The use of pesticides in agricultural practice has increased in Nigeria since 1950, leading to environmental contamination (Chang, 2018; Haruna and Maitera, 2023 & Thurman *et al.*, 2000). Globally, the United Nations has noted that approximately 200,000 people die annually due to toxic pesticide exposure, highlighting the need for stricter worldwide regulatory measures for pesticides, which are originally intended for pest control and weed management during farming processes (Isah *et al.*, 2021; Rifai, 2017).

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). Several types of pesticides have been used in the last decades such as organochlorines, organophosphate, carbamates, pyrethrin and pyrethroids (Jayaraj *et al.*, 2016 & Kaur *et al.*, 2019). Organochlorine pesticides (OCPs) were commonly used as insecticides between the 1940s and 1970s (Cox, 2002) until they were banned at the Stockholm Convention on Persistent Organic Pollutants (POPs) by the United Nations ((UNEP, 2001 & 2009). This action was due to their long-range transport, slow degradation and persistence in the environment for longer period (Ennaceur *et al.*, 2008; Tongo *et al.*, 2019 & Okrikata *et al.*, 2022). Also, their capacity to bio accumulates in food chain poses a threat to human health and the environment (Chau, 2005; Getenga *et al.*, 2004 & Maksymiv, 2015). After the ban of organochlorine, organophosphate pesticides (OPPs) became the most widely used pesticides accounting for an estimated 34% of world-wide insecticide sales (Eze *et al.*, 2018 & Singh and Singh, 2005). This is because they degrade faster and are generally not persistent in the environment but remains in the water for long periods without breaking down into other compounds (Maton *et al.*, 2016 & Fosu-Mensah *et al.*, 2016).

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). They are spread in the air, soil, water and can be taken up by plants and animals (Mavura & Wangila, 2014 & Bhandari *et al.*, 2020). The movement of pesticides in the environment depends on the physical and chemical properties of the pesticides (such as volatility, water solubility and half-life) as well as environmental conditions (Geronimo *et al.*, 2014). Contamination of water bodies for example is a major concern for fish and other aquatic organisms which are major sources of protein (Esumang & Chokky, 2009). 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 (Das *et al.*, 2002 & Lanfranchi *et al.*, 2006). 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 (UNEP, 2007). 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 (Walley *et al.*, 2006 & Akan *et al.*, 2013). There has been increasing evidence that human exposure to OPPs 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 prolonged impairment of cognitive functions, including memory and attention (Terry, 2012 & Kaur *et al.*, 2019). Recently WHO reports that about 80 % of all human illnesses in the developing world are caused by biological contaminations (FAO/WHO, 2009 &2010).

In Taraba State, Bali specifically, North-East Nigeria, the use of monitoring tools to assess pesticides in the environment is still in its early stages. There have been few research specifically focusing on organophosphate pesticides, despite their significance and high toxicity. Therefore, the objectives of this study were to identify individual organophosphate pesticide residues and examined the extent of contamination in fish samples.

Materials and Method

Study Site

The study was carried out at Wurbo and Yelwa Lakes of 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°03'0 E and 11°00' E of the prime meridian. 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 occupation of the inhabitants is farming, fishing and nomadism.

Sample Collection

Fish identified and selected for this project were tilapia (*Tilapia zilli*), Catfish (*Clarias gariepinus*), and Mormyrids (*Mormyrus rume*). Species were among the commonly consumed fishes from the lakes. The fish were collected according to the standard procedure by USFDA, (2001). Samples were collected randomly from fishermen catches at the landing sites between 9:00am – 11:00am. 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 recorded. Fish samples were thawed, cleaned with distilled water and scales sloughed off. Muscle tissues was dissected, minced into smaller pieces and homogenized (Ayeloja *et al.*, 2014)

Extraction of Pesticide Residues in Fish

The Quick-Easy-Cheap-Effective-Rugged-Safe (QuEChERS) method was employed in this analysis. Ten (10.0) g of powder fish sample was weighed into a 100 ml conical flask followed by the addition of 10 ml of water. The sample was homogenized and kept for 40 min. 10 ml of acetonitrile containing 0.5% acetic acid and immediately the QuEChERS salt (6 g MgSO₄ + 1.5 g NaAcetate) were added, shake and vortex for 2 min and centrifuge at 5000 rpm for 5 min. The mixture was allowed to stand for 2 min for phase separation. 6 ml of the upper extract of acetonitrile was drawn for further clean up. The extract was transfer to the dSPE tube containing clean up mixture (1200 mg anhydrous MgSO₄, 400 mg PSA and 400 mg C18), and

vortex for 3 min. the mixture was again centrifuged at 5000 rpm for 5 min. the supernatant was collected and filtered through a 0.2 μ m PTFE membrane filter. 2.0 μ l of the clean extract was injected into GC/MS for analysis.

GC Analysis

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 μ m 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 \times 0.25 mm internal diameter fused silica capillary coated with VF1701 (0.25 μ m 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

Result

Tables I and 2 below is the morphometric characteristics of the fish species collected from Wurbo and Yelwa Lakes of Bali Local Government Area of Taraba State.

Table 1: The morphometric measurement; average length, average weight and condition factor of the fish species from Wurbo.

Collection Sites	English Name	Scientific Name	Sample Size	Average Weight (g)	Ave (cm)	Length	K - value
Wurbo Lake	Tilapia	<i>Tilapia Z</i>	4	99.67 \pm 0.07	12.78 \pm 0.13	4.17	
	Cat Fish	<i>C. gariepinus</i>	3	199.78 \pm 0.03	16.58 \pm 0.13	4.38	
	Mormyrids	<i>Mormyrus rume</i>	4	215.37 \pm 0.11	16.98 \pm 0.29	3.91	

Table 2: The morphometric measurement; average length, average weight and condition factor of the fish species from Yelwa.

Collection Sites	English Name	Scientific Name	Sample Size	Average Weight (g)	Ave (cm)	Length	K - value
Yelwa Lake	Tilapia	<i>Tilapia Z</i>	4	101.42 \pm 0.04	13.16 \pm 0.23	4.45	
	Cat Fish	<i>C. gariepinus</i>	3	203.19 \pm 0.05	16.89 \pm 0.11	4.22	
	Mormyrids	<i>Mormyrus rume</i>	3	185.89 \pm 0.03	17.19 \pm 0.17	3.66	

Figures 1 to 3 below Show the concentration of the organophosphate pesticides residues in fish samples collected from Wurbo and Yelwa Lakes.

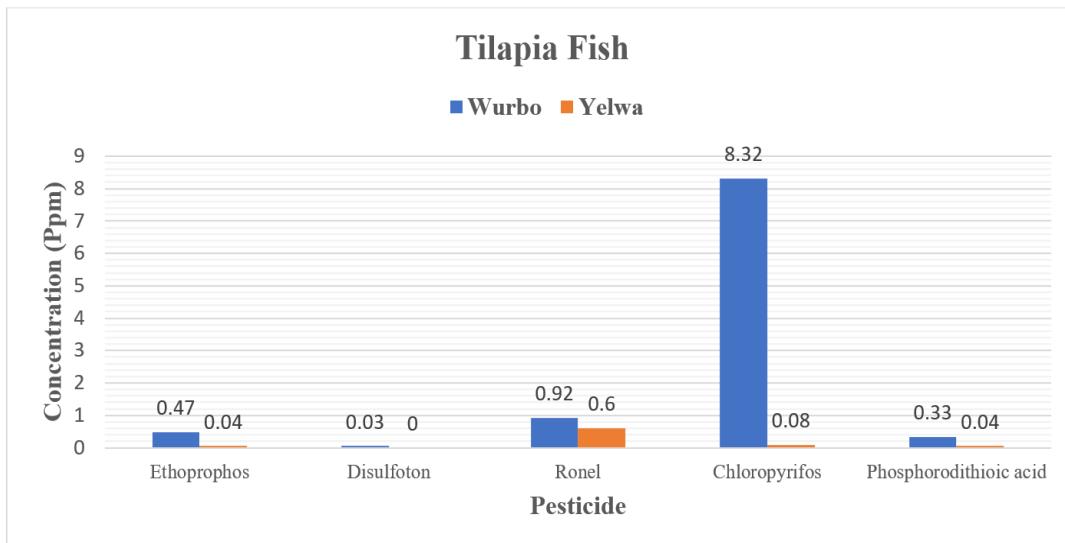


Figure 1; Show the concentration of the organophosphate pesticide residues in *Tilapia zilli*

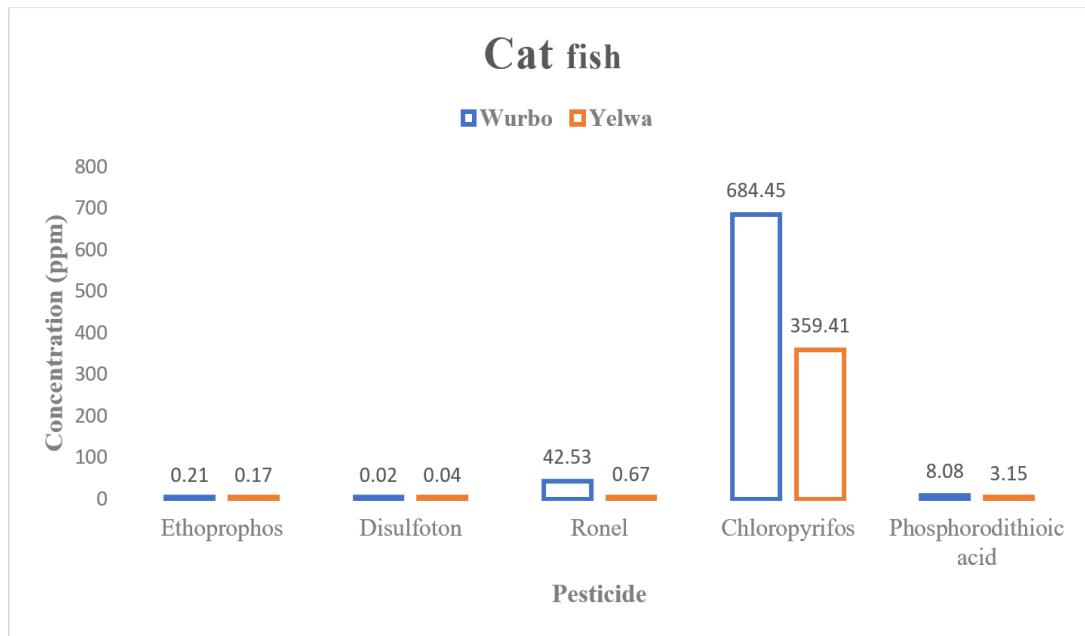


Figure 2; Show the concentration of the organophosphate pesticide residues in *Clarias gariepinus*

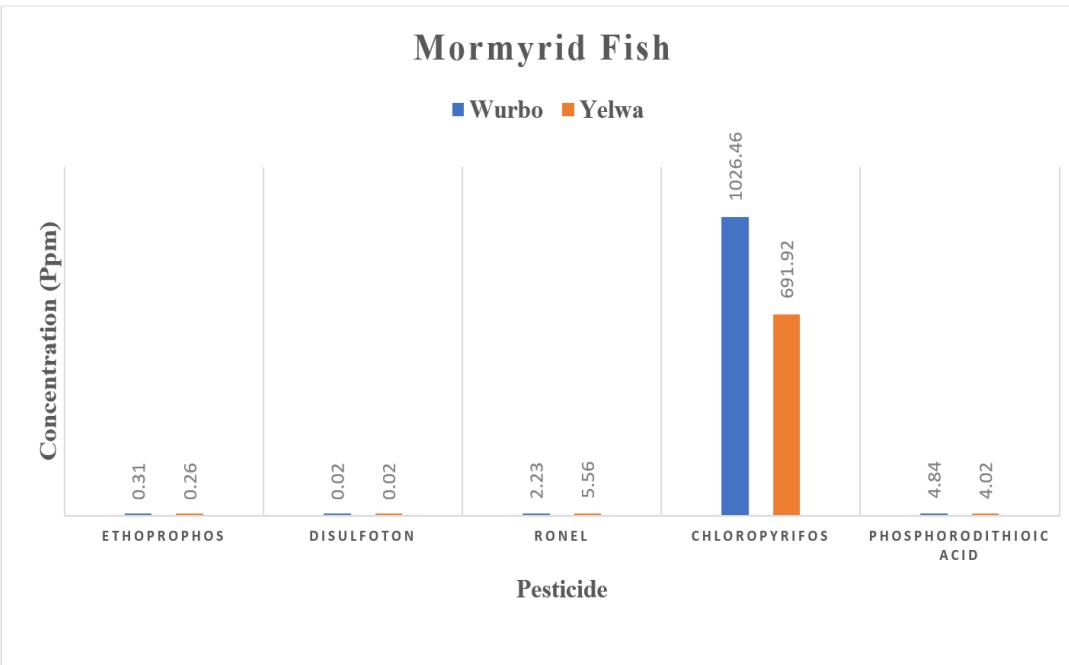


Figure 3; Show the concentration of the organophosphate pesticide residues in *Mormyrus rume*

Discussion

The morphometric measurement such as average length, average weight and condition factor of the studied fish species were measured. The *Mormyrus rume* had the highest average length of 16.98 ± 0.29 cm, and average weight of 215.37 ± 0.11 g follows by *Clarias gariepinus* which had the average length of 16.89 ± 0.11 cm, and average weight of 203.19 ± 0.05 g. *Tilapia zilli* was the least species of fish sample with a length of 12.78 ± 0.03 cm and average weight of 99.67 ± 0.07 g. 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. The K value range of 2.9 to 4.8 was recommended as suitable for matured fresh water fish. Ayeloa *et al* (2014) reported that fishes with a low condition index are presumably believed to have experienced adverse physical environment or insufficient nutrition.

Organophosphate pesticides are one of the widely used pesticides by most farmers in those areas because of their low cost and also the wide range of uses (Vatandoost *et al.*, 2016). The concentration of organophosphate pesticide residues in the lakes and rivers was dependent on various factors, which include application of each pesticide, nearness of farmlands to the water bodies, breakdown ability of these pesticides, absorption potential of the soil, water temperature, clay content and pH (Abedi-Koupai *et al.*, 2011). For this reason, the occurrence of pesticide residues of organophosphate in the fish samples from Wurbo and Yelwa lakes was an indicative of possible contamination of the lakes through agricultural activities around the areas. In this study, the presence of organophosphate pesticide residues at all sampled location can be traced to direct pesticide spray over farmlands or as a result of runoff from long-distance farmlands (Muhamad *et al.*, 2010), and these substances have been observed to be harmful to aquatic life.

Based on the results obtained from all samples, it was found that the levels of organophosphates pesticides residues in fish samples were higher in

Mormyrus rume follow by *Clarias gariepinus* while the *Tilapia zilli* recorded low concentration of the pesticide residues. The total of five organophosphate were recorded in this study which include; Disulfoton, Ethoprophos, Ronel, Chlorpyrifos and Phosphorodithioic acid. The Chlorpyrifos was found to be higher follow by Ronel and Phosphorodithioic acid. Ethoprophos and Disulfoton was recorded very low in all the three fish species. Chlorpyrifos recorded high concentration of 1026.46 ppm in *Mormyrus rume* from Wurbo lake and 691.92 ppm from Yelwa lake. In *Clarias gariepinus*, Chlorpyrifos recorded the concentration of 584.45 ppm in Wurbo and 359.41 ppm from Yelwa lake. Ronel recorded levels of 42.53ppm in *Clarias gariepinus* and 5.56ppm in *Mormyrus rume* from Wurbo. Phosphorodithioic acid recorded the concentration of 8.08 ppm in *Clarias gariepinus* and 4.84 ppm in *Mormyrus rume* from Wurbo lake. These values were above the maximum residue limits (MRL) by FAO/WHO, (2009, 2010 & 2021), ranges from 0.001 to 0.5ppm.

Similar study 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*. Recent studies by Unice, (2024) reported lower concentration in the range of 0.02 to 0.42 ppm of these OPP in the water in afro tropical stream flowing through farmlands in Niger which was also lower than those detected in this study. The high concentration of OPP 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 fish 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. Pesticide pollution to the lake is therefore, likely to pose a danger to both aquatic organisms and humans (Mavura & Wangila, 2014). The study observed several organophosphate pesticides like chlorpyrifos that have been banned in countries like the members of the European Union (EU 2019), even though the concentration was recorded very high.

Conclusion

This study has revealed concentrations of organophosphates pesticides residues in three species of fish from Wurbo and Yelwa lakes. It was found that the levels of organophosphate pesticides residues were higher in *Mormyrus rume* followed by *Clarias gariepinus* while the *Tilapia zilli* recorded low concentration of the pesticide residues. The Chlorpyrifos was observed to be higher followed by Ronel and Phosphorodithioic acid. Ethoprophos and Disulfoton were very low in all the three fish species. The result indicated that the pesticides residues were above the WHO/FAO values. 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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References

Abedi-Koupai, J., Eslamian, S.S., Zareian, M.J., 2011. Measurement and modelling of water requirement and crop coefficient for cucumber, tomato and pepper using micro lysimeter in greenhouse. *Journal of Science and Technology of Greenhouse Culture*. 2(7), 51-64.

Akan, J., I., Jabiya, Z. Mohd. F.I., Abdurhman, (2013). Organic Pesticide Residues in Vegetable and Soil, Sample from Alau Dam and Gmgulong Areas Borno State, Nigeria, *International Jour of environment Monitoring and analysis*1(2); 58-65.

Akdogan A, Hol A, Divrikli U, Elci L, Campus I (2014). Determination of pesticides in soil by 143mechanical stirring-assisted extraction coupled with gas chromatography-mass spectrometry. *Anal Lett*;47(4):675-88. 2.

Akoto O, Azuure AA, Adotey KD (2015) . Pesticide residues in water, sediment and fish from Tono Reservoir and their health risk implications. *SpringerPlus*;5(1):1-11

Ayeloa, A. A., George, F. O. A., Shorinmade, A. Y., Jimoh, W. A., Afolabi, Q. O. and Olawepo, K. D. (2014). Heavy Metal Concentration in Selected Fish Species from EleyeleReservoir Ibadan, Oyo State, South-West Nigeria. *Africa Journal. Of Environmental Science and tech.*, 8(7): 422 – 427.

Bhandari G, Atreya K, Scheepers PT, Geissen V (2020). Concentration and distribution of pesticide residues in soil: Non-dietary human health risk assessment. *Chemosphere*; 126594

Biego GHM, Yao KD, Ezoua P, KouadioLP, (2010). Assessment of Organochlorine Pesticides Residues in fish sold in Abidjan Markets and fishing sites. *At. J. food* 144 *Agric. Nutr.* 10:3.

Chau KW (2005), Characterization of transboundary POP contamination in aquatic ecosystems of Pearl River delta. *Mar Pollut Bull*51:960–965.

Chang GR, (2018). Persistent organochlorine pesticides in aquatic environments and fishes in Taiwan and their risk assessment. *Environmental Science and Pollution Research*;25(8):7699-7708. <https://doi.org/10.1007/s11356-017-1110-z>.

Cox, J.R. (2002). Pesticide residue analysis facilities: experiences from the Natural Resource Institute's Support Program. In: Hanak, E., Boutrif, E., Fabre, P., Pinero, M. (Scientific Editors) *Food safety management in developing countries, Proceedings International Workshop*, CIRADFAO. Montpellier, France.

Das B, Khan YSA, Das P, Shaheen SM (2002), Organochlorine pesticide residues in catfish, *Tachysurus thalassinus* (Ruppell, 1835) from the South Patches of the Bay of Bengal. *Environ Pollut* 120:255–259.

Devi NL, Chakraborty PQ, Zhang SG (2013). Selected Organochlorine Pesticides (OCPs) In Surface Soils from Three Major States from the North Eastern part of India. *Bull. Environ. Sd. Res.* 2:1.

Eunice O. Ikayaja, (2024). Organophosphate pesticide residue impact on water quality and changes in macroinvertebrate community in an Afrotropical stream flowing through farmlands. *Environmental Monitoring Assessment*; <https://doi.org/10.21203/rs.3.rs-3969787/v1>

Essumang DK, Chokky L (2009), Pesticide residues in the water and fish (Lagoon tilapia) samples from Lagoons in Lagoons in Ghana. *Bull ChemSoc Ethiopia* 23(1):19–27.

Ennaceur S, Gandoura N, Driss MR (2008). Distribution of polychlorinated biphenyls and organochlorine pesticides in human breast milk from various locations in Tunisia: levels of contamination, influencing factors, and infant risk assessment. *Environmental Research*;108(1):86-93. <https://doi.org/10.1016/j.envres.2008.05.005>.

Eze JN, Ndu IK, Edelu BO (2018). Teenage organophosphate insecticide poisoning: An ugly trend in Enugu, Nigeria. *Journal of Community Medicine and Primary Health Care*;30(1):99-108.

FAO/WHO (2009) Food standard programmes. In: Codex Alimentarius Commission, vol 9 (4), pp 149–158.

FAO/WHO (2010) Pesticide residues in food and feed. Acceptable daily intake. Codex Alimentarius Commission. FAO/ WHO Food Standards,

FAO/WHO (2021) Pesticide residues in food and feed. Acceptable daily intake; Codex Alimentarius Commission. FAO/ WHO Food Standards, Rome

Fianko R. J, Augustine D, Samuel TL, Paul OY, Eric TG, Theodosia A, Augustine F (2011) Health Risk Associated with pesticide Contamination of fish from the Densu River Basin in Ghana, *j. Environ. Protection* 2: 115-123.

Fosu-Mensah BY, Okoffo ED, Darko G, Gordon C (2016). Organophosphorus pesticide residues in soils and drinking water sources from cocoa producing areas in Ghana. *Environ Syst Res*;5(1):1-12.

Geronimo. De, Aparício E, VC, Barbaro S, Portocarrero R, Jaime S, Costa JL (2014). Presence of pesticides in surface water from four sub-basins in Argentina. *Chemosphere* 107: 423-431.

Getenga ZM, Kengara FO, Wandiga SO (2004) Determination of organochlorine pesticides in soil and water from river Nyando drainage system within Lake Victoria Basin, Kenya. *Bull Environ Contam Toxicol* 72: 335-343.

Haruna, I., Maitera, O.N., 2023. Determination of organochlorine pesticides residues in water, fish and sediment samples from River Tella, Gassol, Taraba State, Nigeria. *International Journal of Scholarly Research in Science and Technology*. 02(01), 019-027. <https://doi.org/10.56781/ijssrt.2023.2.1.0014>

Isah, H. M., Raimi, M. O., Sawyerr, H. O., 2021. Probabilistic Assessment of Self-Reported Symptoms on Farmers Health: A Case Study in Kano State for Kura Local Government Area of Nigeria. Available at SSRN: <https://ssrn.com/abstract=3925849>

Jayaraj R, Megha P, Sreedev P, (2016). Organochlorine pesticides, their toxic effects on living organisms and their fate in the environment. *Interdisciplinary toxicology*;9(3-4):90-100. <https://doi.org/10.1515/intox-2016-0012>.

Joseph Clement Akan, Lawan Bukar Inuwa, Zaynab Muhammad Chellube, Musa Muhammad Mahmud, Fanna Inna Abdulrahman (2019). Assessment of the Levels of Herbicide 151 Residues in Fish Samples from Alau Dam, Maiduguri, Borno, State, Nigeria. *International Journal of Environmental Chemistry*. Vol. 3, No. 2, pp. 53-58. doi: 10.11648/j.ijec.20190302.11.

Kaur R, Mavi GK, Raghav S, (2019). Pesticides classification and its impact on environment. *Int J Curr Microbiol Appl Sci*;8(3):1889-1897.

Knobler SL, Lemon SM, Najafi M, et al. (2003) The resistance phenomenon in microbes and infectious disease vectors: implications for human health and strategies for containment: workshop summary. Washington(DC): National Academies Press (US).

Lanfranchi AL, Menone ML, Miglioranza KSB and Janiot LJ (2006), striped weakfish (*Cynoscion guatucupa*): a biomonitor of organochlorine pesticides in estuarine and Near-coastal zones. *Mar Pollut Bull* 52:74-80.

Maksymiv I, (2015). Pesticides: benefits and hazards. *Journal of Vasyly Stefanyk Precarpathian National University*;2(1):7076. <https://doi.org/10.15330/jpnu.2.1.70-76>.

Maton SM, Dodo JD, Nesla RA, Ali AY (2016). Environmental impact of pesticides usage on farmlands in Nigeria. International Journal of Innovative Research Development;5(4): 311-317.

Mavura WJ, Wangila PT (2014) Distribution of Pesticide Residues in Various Lake Matrices: Water, Sediment, FishAnd Algae, The Case of Lake Nakuru, Kenya. The African network for chemical analysis of pesticides, Arusha.

Okrikata, E., Agere, H., Malu, S. P., Olusesan, A. I., & Ahmed, M. (2022). Determination and Health risk assessment of pesticide residues in watermelon (*citrullus lanatus*) fruit Samples in wukari, Nigeria. The Bioscientist Journal, 10(1), 4154

Randhawa, M., Abid, Q., Anjum, F., Chaudhary, A., Sajid, M., & Khalil, A.A. (2016). Organochlorine pesticide residues in okra and brinjal collected from peri-urban areas of big cities of Punjab-Pakistan. Pakistan Journal of Agricultural Sciences, 53, 425-430.

Rifai, N. (2017). Tietz textbook of clinical chemistry and molecular diagnostics-e-book. Elsevier Health Sciences

UNEP (2007), Voluteer Lake Monitoring, A methods Manual. Office of water US Environmental Protection Agency, Washinton DC.

United Nations Environment Programme (UNEP, 2001). Final act of the conference of plenipotentiaries on the Stockholm Convention on Persistent Organic Pollutants. UNEP/POPS/CONF/4.

United Nations Environment Programme (UNEP, 2009). Stockholm Convention on Persistent Organic Pollutants (POPs) in Stockholm Convention on Persistent Organic Pollutants (POPs).

Singh J, Singh DK (2005). Dehydrogenase and phosphomonoesterase activities in groundnut (*Arachis hypogaea L.*) field after diazinon, imidacloprid and lindane treatments. Chemosphere;60(1):32-42. <https://doi.org/10.1016/j.chemosphere.2004.11.096>.

Terry Jr AV, (2012;). Functional consequences of repeated organophosphate exposure: potential non-cholinergic mechanisms. Pharmacol & ther. 134(3):355-365.

Thurman, E. M., Bastian, K. C., & Mollhagen, T. (2000). Occurrence of cotton herbicides and insecticides in playa lakes of the High Plains of West Texas. The Science of the total environment, 248(2-3), 189–200. [https://doi.org/10.1016/s0048-9697\(99\)005422](https://doi.org/10.1016/s0048-9697(99)005422).

Tongo, I., Ewere, E. & Ezemonye, L. (2019). Organochlorine pesticide residues in fish (*Alestes Baremoze* and *Synodontis bastianii*) from Warri River, Nigeria: Levels and human Exposure assessment. Sokoto Journal of Veterinary Sciences.

Walley, F. Tailoy and Lupwagi, P. (2006). Herbicides effects on pulse crop modulation and Nitrogen Fixation Teach 2006 proceeding pp, 121-123.