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Pesticide Residue Analysis in Selected Food Crops in Bwari Area of Abuja, Nigeria

Salome Sagi Kolo [1, *], Sunday Adebayo Kolawole [1], Noela Chinyelu Igwemmar [1], Mary Sunday Dauda [1] and Rebecca Wusa Ndana [2]

[1] *Department of Chemistry, Faculty of Science, University of Abuja, Nigeria.*
[2] *Department of Biological Sciences, Faculty of Science, University of Abuja, Nigeria.*

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Abstract

High yields in food crops and prevention of post-harvest losses are some agricultural practices which have led to a wide application of agrochemicals like organochlorine pesticides (OCPs). This study analyzed the presence and concentrations of twenty OCP residues in food crops selected from communities in Bwari area council of the Federal Capital Territory (FCT). Experimental values gotten were compared to Maximum Residue Limit (MRL) to ascertain that they are within acceptable limits. The stored food crop samples; maize (Zea mays), guinea corn (Sorghum bicolor) and cowpea (Vigna unguiculata) were collected from storages of households from the study site. Samples were prepared using QuEChERS method of preparation and analysis carried out using the Gas Chromatography – Mass Spectrometry (GC-MS). Results from this study showed OCP residue presence in most food crops, at varying concentrations. Despite the presence of OCPs, their mean concentration values were at levels below MRL. However, Zuma Yellow Maize (ZYM) had the presence of p,p’-DDE (a metabolite of DDT) with mean concentration value of 146.38 µg/kg. This was far above MRL of 50 µg/kg for p,p’-DDE. Thus, ZYM showed health risk index of 337.513 which is greater than 100, indicating a higher health risk to consumers. Routine monitoring of food crops should be carried out frequently to ensure food safety and security. Furthermore, farmers should also be educated on the effects of pesticides, as actions are taken on the mis-use of pesticides.

Keywords: Organochlorine pesticides; Maize; Guinea corn; Cowpea; QuEChERS; Gas Chromatography-Mass Spectrometry

1. Introduction

Agricultural practices with the intention to have high yields in food crop production have led to the wide application of agrochemicals like organochlorine pesticides (OCPs). This is one way that the second Sustainable Development Goals (SDGs) on food security; which aims to end hunger, achieve food security, improve nutrition, and promote sustainable agriculture - is achieved. The increasing demand for food supply has had a direct relationship with the steady rise in human population (Sruthi *et al.,* 2017).

In recent times, the world’s population has been estimated to be about 8.5 billion people by the year 2030 with annual growth rate put at 1.2% (Smith and Gregory, 2013). According to the United Nations (UN), 95% of this increase is likely to be experienced in developing countries of the world and mostly in Sub-Sahara Africa (Atangana, 2022). Therefore, to meet up with the second goal of the SDGs, this has encouraged the unreserved use of pesticides, especially as it pertains to post-harvest losses.

The Food and Agriculture Organization (FAO) of the United Nations (UN), have said that, ‘pesticides are chemical compounds that are used to control pests, including insects, rodents, fungi, larvae and unwanted plants (weeds) in order

* Corresponding author: Salome Sagi Kolo. Email: salometerfaabraham@gmail.com

to improve the yield of food crops' (FAO, 2021). They are substances or any mixture of substances which have biological or chemical components intended for controlling, repelling or even destroying pests, thereby regulating plant's growth (World Health Organization, 2023a).

Some organochlorine insecticidal pesticides are DDT, Lindane and Chlordane. The usage of pesticides is widely distributed in the world ranging from agricultural fields, homes, parks, and schools. These pesticides often find their way further, into food crops commonly consumed by man (Kasozi *et al.,* 2006; Oluwole and Cheke, 2009).

The impact of pest and diseases have always been a setback in agriculture and its practices. This has led to crop damage, reduction in food availability and consequently, increase in food prices (Sruthi *et al.,* 2017). For instance, the recurrence of *Tuta absoluta*, popularly known as "Tomato Ebola", points to the country's inability to handle recurring food crop diseases which has challenged many farmlands. This "Tomato Ebola" in Nigeria is a vivid demonstration of the importance of pesticides in modern day agricultural practices (Sruthi *et al.,* 2017). For without the use of pesticides, unimaginable losses of food crops would occur, while farmers helplessly look on.

In the past, the Nigerian news has reported on a number of cases of food poisoning and fatalities in various parts of the nation, including Cross River, Taraba, and Gombe States (Mazlan *et al.,* 2017). Furthermore, because of high levels of contamination, the European Union (EU) prohibited some agricultural food exports from Nigeria (Akande *et al.,* 2023).

The Nigerian Agricultural Quarantine Service (NANQS) has reported that plant pests account for about 50% of food crop loss (Dextra International, 2019; Ugwu *et al.,* 2022). As a measure to curtail food crop loss and damage, farmers in Nigeria and most part of the world at large, have employed the use of pesticides for many decades (Asogwa and Dongo 2009).

Though pesticides are very important in food crop production and storage today, they are very poisonous by nature when they get into food crops and constitute one of the most hazardous groups of contaminants to human health, fauna and the environment at large (Alengebawy *et al.,* 2021). Food crops whose main nutrients come from persistent and continuous development of soil nutrients, have an awesome capacity of retaining nutrients and other components from the soil (Kolawole *et al.,* 2022). The thriving of such plants in contaminated soil is an indication of a potential hazard and overwhelming contamination retention by plants is of serious human health risks; as through the food chain, even pesticide residues enter the human body (Kolawole *et al.,* 2022).

When people come in contact with large quantities of pesticides, it may cause severe poisoning or long-term health effects, cancer inclusive, and adverse effects on reproduction. Pesticides, particularly in food crops, have become the leading cause of death by self-poisoning, particularly in low- and middle-income countries (Eddleston, 2020). To protect food producers and consumers from the adverse effects of pesticides, the World Health Organization (WHO) reviews evidence and have developed internationally-accepted maximum residue limits for pesticides (Donkor *et al.,* 2016). The use of some of these banned organochlorine pesticides in farming activities, storage of food crops and around the house, has exposed our ecosystem to varying degrees of contamination due to the proliferation of different types of organochlorine pesticides used in cultivation and storage of food crops. As these pesticides are often used on food crops both on farmlands and in storages, its continuous spread in the environment becomes inevitable. It is therefore necessary to ascertain the distribution, behavior and fate of these compounds in various food crops (grains) and put in place strict regulations and control. This study therefore determined organochlorine pesticide residues in selected food crop in communities in Bwari Area Council of the Federal Capital Territory (FCT), Abuja – Nigeria. These areas were selected as a result of much farming activities ongoing in the areas which contributes to a significant amount of food crops in that environment. Also, there is limited data on organochlorine pesticide residues in the study area. The results from this study will serve as an awareness to the farmers, education for the consumers, a guide in policy-making for regulatory bodies, contribution to better handling of food crops to ensure food security and safety; and also, good insight to agricultural productivity and by extension, safeguarding the environment.

2. Materials and method

2.1. Study Area

The study area is along Duste - Bwari road in the North- Eastern part of Abuja. This study was conducted on 2 communities and their extension areas latitude 9° 17' 5" North of the Equator and longitude 7° 25' 23" East of the Greenwich Meridian, with sunrise at 07:16:48 and sunset at 19:54:52 (Imhanfidon *et al.,* 2023). It is sited about 26 kilometres away from Abuja city and 10 kilometres away from Bwari town (Sunday, 2021); where there are so much farming activities on-going as a major occupation of the inhabitants. This district has a tropical climate with the characteristic dry and wet seasons. Dry season begins from November to February while the wet season is from March to October. The mean annual rainfall is between 125 cm and 175 cm while the annual temperature is about 28 ^{o}C (Sunday, 2021).

2.2. Experimental design

Samples were carefully sampled and unwanted materials picked out from them. They were homogenized so as to get uniform particles and further sieved. Food crop samples were subjected to QuEChERS method of extraction and clean-up. These were further subjected to Gas – Chromatography (GC-MS) analysis and final results analyzed using Microsoft Excel (MS Excel) and Statistical Package for Social Sciences (SPSS).

2.3. Data Collection

This research work was aimed at analyzing organochlorine pesticide (OCP) residues in selected food crop samples of some communities in Bwari Area Council of the Federal Capital Territory (FCT), Abuja. The study sites were visited not less than three times, so as to get familiar with the terrain. Focus group discussions were held with community heads and leaders, after which interviews were scheduled at a later time.

2.4. Food Crop Samples Collection

Guinea corn (brown and white); Maize (yellow and white); Cowpea (brown and white) were obtained from ten (10) storage bags each. Sub-samples were collected using a grain probe from various locations of each bag. These were composited to produce representative samples. Samples were put in Ziploc bags, labelled appropriately as shown on table 1.

Table 1 Showing Collected Food Crop Samples and their Laboratory Code

Sample ID	Sample Description	Lab. CODE
1	Gaba Brown Guinea corn sample	GBGC
2	Gaba White Guinea corn sample	GWGC
3	Gaba Yellow Maize sample	GYM
4	Gaba White Maize sample	GWM
5	Gaba Brown Cowpea sample	GBCP
6	Gaba White Cowpea sample	GWCP
7	Zuma Brown Guinea corn sample	ZBGC
8	Zuma White Guinea corn sample	ZWGC
9	Zuma Yellow Maize sample	ZYM
10	Zuma White Maize sample	ZWM
11	Zuma Brown Cowpea sample	ZBCP
12	Zuma White Cowpea sample	ZWCP

These were taken to the laboratory and stored in the freezer at 4 ^{o}C for further analysis.

2.5. Preparation of Food Crop Samples

Foreign matters (like stones and admixtures) were sorted out from the samples. Thereafter, samples were pulverized using Master Chef blender model MC – BL1970 to ensure proper comminution and homogeneity. The pulverized samples were sieved through 1.0 mm size sieve mesh to obtain uniform particle size. They were put in air-tight Ziploc bags, labelled accordingly and stored in the freezer at 4 ^{o}C until needed for further analysis.

2.6. Reagents Used for Analysis

In this study, all chemicals and reagents that were used for the analysis were of analytical grade (AR). Anhydrous Magnesium sulfate (98 % Sigma-Aldrich, US), Acetonitrile (99.5 % Thermo-Scientific Chemicals, US), Glacial acetic acid

(99.5% Welychem Co. Ltd, China), Anhydrous Sodium citrate (99 % Hawkins Inc., US), Sodium chloride (95 % Hawkins Inc., US), pure Acetone 99.5 % (Sigma-Aldrich, US).

Extraction and Clean-up of Samples: The samples were extracted using multi-residue pesticide analysis technique, which involves the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method of sample preparation (OAOC 2007.01 procedure). This was further cleaned up using Primary Secondary Amine (PSA).

Analytical Method: The analytes were stored in GC vails and sent for GC-MS analysis. Samples were collected and analyzed in triplicates after which the data was analyzed using Microsoft Excel and Statistical Package for Social Sciences (SPSS).

Extraction of Organochlorine Pesticides in Food Crop Samples: 10 g of finely grounded sample was placed in a 50 mL polypropylene centrifuge tube with 10 mL water (H_2O) added to it. To this mixture was added 15 mL acetonitrile (CH_3CN) and the mixture was vortexed vigorously for 5 minutes. Furthermore, 0.5 g disodium hydrogen citrate sesquihydrate ($C_6H_8Na_2O_8$), 1 g trisodium citrate dihydrate ($C_6H_5Na_3O_7.2H_2O$) (buffers), 4 g anhydrous magnesium sulphate ($MgSO_4$) and 1 g sodium chloride (NaCl) (for salting-out) were added. The mixture was vortex immediately for another five (5) minutes then centrifuged at 4500 rpm for five (5) minutes. The extract was then separated from the precipitates by simple decantation.

QuEChERS Clean-Up of Sample Extract: Using the AOAC 2007.01 procedure, 1 ml aliquot of the extract from extraction and partitioning steps was transferred into a polypropylene centrifuge tube containing 100 mg anhydrous magnesium sulphate ($MgSO_4$), 75 mg graphite carbon black (C_{18}) sorbent (to remove plant pigment), and 20 mg Primary Secondary Amine (PSA) sorbent (to remove excess water). The tube was again vortexed for 0.5 minute and centrifuged at 4500 rpm for 2 minutes. An aliquot of the supernatant was transferred into a glass test tube and acidified by adding 15 µL of 5 % (v/v) formic acid in acetonitrile per mL of extract for subsequent injection into the Gas Chromatography.

2.7. Instrumental analysis

GC-MS: Agilent HP-5-60 Gas Chromatography-Mass Spectrometry was used to determine the concentration of organochlorine in the samples (Zhang, 2019). The instrument column was 30 m × 0.25 mm x 0.25 µm film thickness. It was operated at 250 ^{o}C and attached to a Gas Chromatograph (6890N Agilent technologies) and a Mass Selective Detector (Agilent 5975B). 1 µL samples was injected into the machine in a spitless mode and the initial column temperature was maintained at 100 ^{o}C for 2 minutes and then increased to 180 ^{o}C at a rate of 15 ^{o}C per minute. Thereafter it was ramped up to 250 ^{o}C at a rate of 3 ^{o}C per minute, where it was held for 9 minutes. The carrier gas used was helium with a flow rate of 1.0 $mLmin^{-1}$. The operation mode of the mass spectrometer was electron impact ionization with the use of automatic gain control. The storage window was programmed at full scan mode in the range of m/z 200 – 500, and the Selected Ion Monitoring (SIM) mode was employed in acquiring data by Agilent ChemStation software.

Sample Analysis for Organochlorine Pesticides Residues: Internal standard technique was used to analyze the food crop extracts (Hiatt, 2010). The organochlorine standards containing a mixture of 14 organochlorine compounds of high purity (alpha BHC, beta-BHC, Lindane, delta-BHC, Heptachlor, Aldrin, Heptachlor – epoxide, Endosulfan I, p,p'-DDE, Dieldrin, Endrin, Endosulfan II, p,p'-DDT, and Endosulfan sulphate) were prepared at concentrations ranging from 0.100 to 2.000 ppm, with Anthracene, PCB-153, and PF-38 added as Internal Standards. The modern Shimadzu GC-MS QP-2010 was used to analyze the standards. Also, calibration curve for each compound was prepared automatically (Kadokami, 2013). Food crop sample extracts from the clean-up were then analyzed under the same conditions as for the standards, while Selective Ion Mode (SIM) had m/z values ranging from 65 to 274.

Recovery Studies: The efficiency of the method was validated with recovery studies. Fortification of reference materials without pesticide residues with four organochlorine compounds at two concentration levels 0.1 mg/kg and 1.0 mg/kg were carried out in triplicate and the same method of extraction and clean-up was followed. Other quality assurance measures applied in the laboratory included rigorous contamination control procedures (washing and cleaning procedures), monitoring of blank levels of solvents and analysis of procedural blanks. The samples were then analyzed with GC-MS with percentage recovery (Richter *et al.,* 2020) calculated using equation (1):

$$\%\, Recovery = \frac{Residue\ concentration}{fortification\ concentration} * 100 \qquad (1)$$

Where fortification concentration is the same as spike concentration.

Quality Assurance: Glass wares and tools that were used during laboratory analysis were thoroughly washed with detergent and running water. This was followed by rinsing with distilled water and then proper drying. These were further rinsed with acetone before they were used in the laboratory. Chemicals and reagents used were all of analytical grade and obtained from BDH and Sigma and Co. They were all used in accordance to given instructions. Instruments were calibrated and test-run before they were used.

2.8. Statistical analysis

The results obtained from this study were analyzed using Microsoft Excel. The correlation and level of statistical significance were also estimated using ANOVA (analysis of variance). The concentrations of organochlorine pesticide residues in guinea corn, maize, and cowpea samples in the study area were then compared with the Maximum Residue Limits (MRLs) by European Union (Carrère *et al.,* 2018).

3. Results and Discussion

Organochlorine pesticide (OCP) residue GC-MS standard chromatograms for all food crop samples from Gaba and Zuma communities were sharp, clear and distinct, indicating that the samples were properly prepared (extracted and cleaned up) before GC-MS analysis was carried out on them. Also, the right analytical equipment was used which presented less noise during analysis.

The mean concentration values for all food crop samples from the communities were calculated and designated appropriately. Also, the percentage distributions of the various OCP residues in each of the samples were calculated. The retention time (RT) and Q-values were also calculated accordingly.

3.1. OCP Residue Concentrations as Observed in Food Crop Samples

Gaba Guinea Corn Samples: The mean concentration value (MCV) of organochlorine pesticides in Gaba brown guinea corn sample (GBGC) ranged from 0.03 ± 0.01 µg/kg to 11.43 ± 1.29 µg/kg and that of white guinea corn sample (GWGC) ranged from 0.03 ± 0.01 µg/kg to 49.20 ± 1.69 µg/kg. Mean concentration value for GWGC; p,p'-DDE (49.20 µg/kg was the most abundant organochlorine pesticide residue, followed by γ-chlordane (5.51 µg/kg) and γ-BHC (2.80 µg/kg), respectively.

Figure 1 shows the chart of mean concentrations of various pesticides of interest in guinea corn samples from Gaba communities.

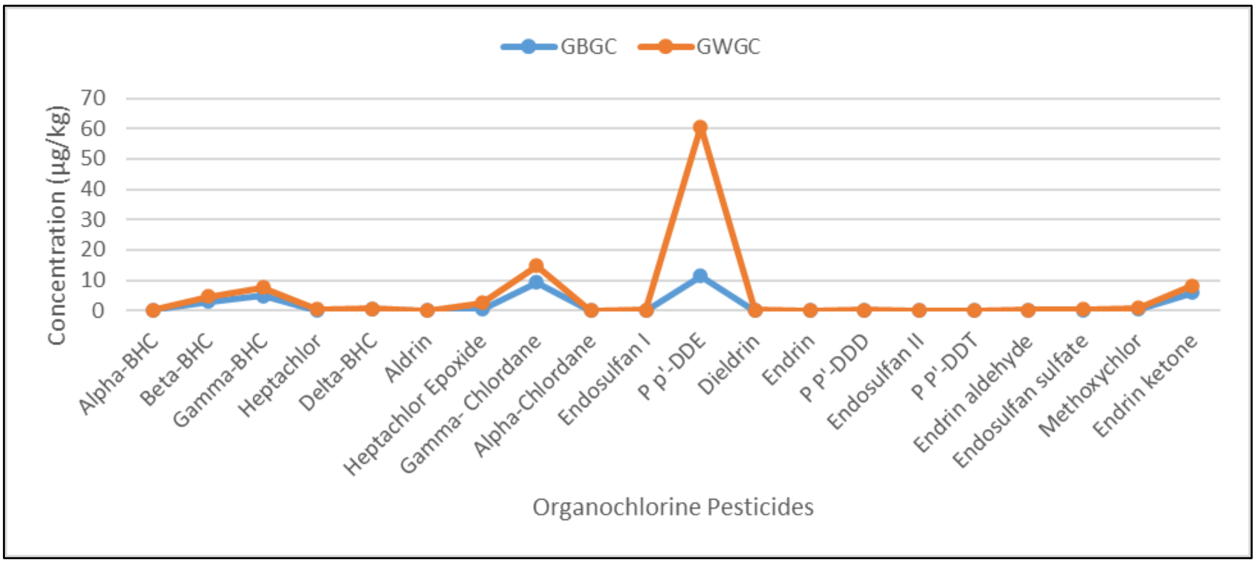

Figure 1 Graphical Representation of OCPs of Gaba Guinea corn Samples

The results showed that most organochlorine pesticide residues in the samples were below maximum residue limits (MRLs) set by the European Union.

These values were within EU/CODEX/FAO Maximum Permissible Limits (MRLs) of 50 µg/kg for all metabolite products of DDT and ADI limit of 20 µg/kg for p,p'-DDD and p,p'-DDT while 5 µg/kg was for Methoxychlor. However, p,p'-DDE contamination violated the acceptable ADI value of 20 µg/kg recommended by EU and FEPA (World Health Organization, 2023a). The concentration of DDT metabolites present in GBGC and GWGC were less than those reported by Oshatunberu *et al.* (2022), that monitored organochlorine pesticide residue contamination of cereals sold in selected markets in South West of Nigeria. Another report by Anzene *et al.* (2014), studied organochlorine pesticide residue analysis of post-harvest cereal grains in Nasarawa State, Nigeria. Oshatunberu *et al.* (2022) reported that the levels of p,p'-DDE residues in millet grains from markets of Ado-Ekiti (212 µg/kg), Ibadan (210 µg/kg), Osun (46 µg/kg) and Ondo (127 µg/kg) were higher that the MRL recommended by both the FAO/WHO as well as the EU. Also, in a study by Kolo *et al.* (2025), that determined levels of organochlorine pesticide residues in selected farmlands soil samples in Bwari – Abuja, found p,p'-DDE pesticides in Gaba farmlands soil sample to be within the MRL recommended by EU/CODEX/FAO (World Health Organization, 2023a), while γ-Chlordane and γ-BHC were above the 10 µg/kg set by the European Union (EU).

Though, the levels of DDT metabolites reported in this study were within EU/CODEX/FAO Maximum Permissible Limits (MRLs), their presence in the samples suggests that there is a need to increase monitoring of pesticides in these food crops. Farmers can be educated, and awareness raised, of the dangers of unauthorized use and mis-use of pesticides, especially those that have been banned, which can harm Nigeria's agriculture industry's reputation as a whole. Hence, it has been reported that banned organochlorine pesticides bio-accumulates in food chain and high level exposure could result in nervous system dysfunction and liver damage (Akoto *et al.,* 2013).

Zuma Guinea Corn Samples: The mean concentration value (MCV) of organochlorine pesticides in Zuma brown guinea corn sample (ZBGC) ranged from 0.02 ± 0.01 µg/kg to 4.36 ± 0.02 µg/kg and that of white guinea corn sample (ZWGC) ranged from 0.02 ± 0.01 µg/kg to 3.89 ± 1.32 µg/kg.

Figure 2 shows the chart of concentrations of OCP residue variations, representing the distribution of pesticide residues in the guinea corn sample from Zuma communities.

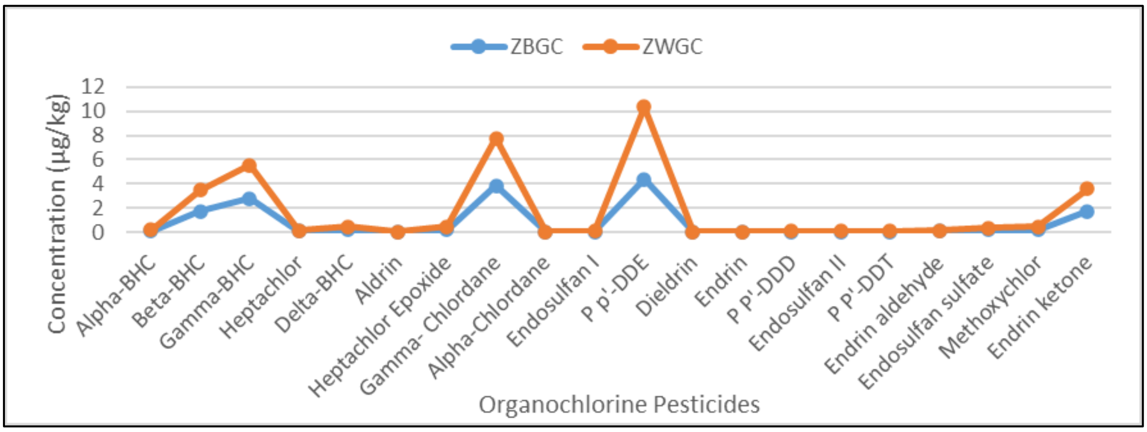

Figure 2 Graphical Representation of OCPs of Zuma Guinea corn Samples

The results showed that most organochlorine in the samples were below maximum permissible limits (MLRs) and acceptable dietary intake (ADI) set by the European Union.

Guinea corn samples from Zuma communities were found to have metabolite products of DDT. ZBGC was contaminated with p,p'-DDE (4.36 µg/kg), p,p'-DDD (0.08 µg/kg), p,p'-DDT (0.05 µg/kg) and methoxychlor (0.22 µg/kg). Also, ZWGC was found to have p,p'-DDE (5.98 µg/kg), p,p'-DDD (0.04 µg/kg), p,p'-DDT (0.04 µg/kg) and methoxychlor (0.23 µg/kg). These values were also within EU/FAO/WHO Maximum Permissible Limits (MLRs) of 50 µg/kg (Raimi, 2021) for all metabolite products of DDT and ADI limit of 20 µg/kg for p,p'-DDD and p,p'-DDT and 5 µg/kg for Methoxychlor (World Health Organization, 2023a). The concentration of DDT metabolite present in ZBGC and ZWGC were less than those reported by Oshatunberu *et al.* (2022) monitored organochlorine pesticides contamination of cereals sold in selected market in South West of Nigeria and also that reported by Anzene *et al.,* (2014) that studied organochlorine pesticide residues analysis of post-harvest cereal grains in Nasarawa State, Nigeria. Oshatunberu *et al.* (2022) reported that the levels of p,p'-DDE residues in millet grains from markets of Ado-Ekiti (212 µg/kg), Ibadan (210 µg/kg), Osun (46 µg/kg) and Ondo (127 µg/kg) were higher that the MRL recommended by both the FAO/WHO as well as the EU. Also, Kolo *et al.* (2025), determining levels of organochlorine pesticide residues in selected farmlands soil samples in Bwari – Abuja, reported that p,p'-DDE pesticides in Gaba farmlands soil samples was found to be within the MRL recommended by EU/CODEX/FAO (World Health Organization, 2023a). Though γ-Chlordane and γ-BHC were found to be less than the standards in this study, their concentrations were higher than the Adequate Daily Intake (ADI) recommended. Which is to say that, their presence in guinea corn samples revealed that the pesticides, despite their ban, are still in use by farmers of those communities.

In a similar study by Anzene *et al.* (2014), concentration of gamma-BHC (Lindane, Gammalin-20) (25 µg/kg) was found in post-harvest guinea corn samples in Nasarawa State, Nigeria. This value and that reported in this study were higher than EU/FAO maximum residue limit. Hence, it has been reported that lindane bioaccumulates in food chain and high level exposure had resulted in nervous system dysfunction, and liver damage (Akoto *et al.,* 2013).

Comparing concentrations of pesticides in guinea corn samples from the study sites;

ZWGC > ZBGC > GWGC > GBGC.

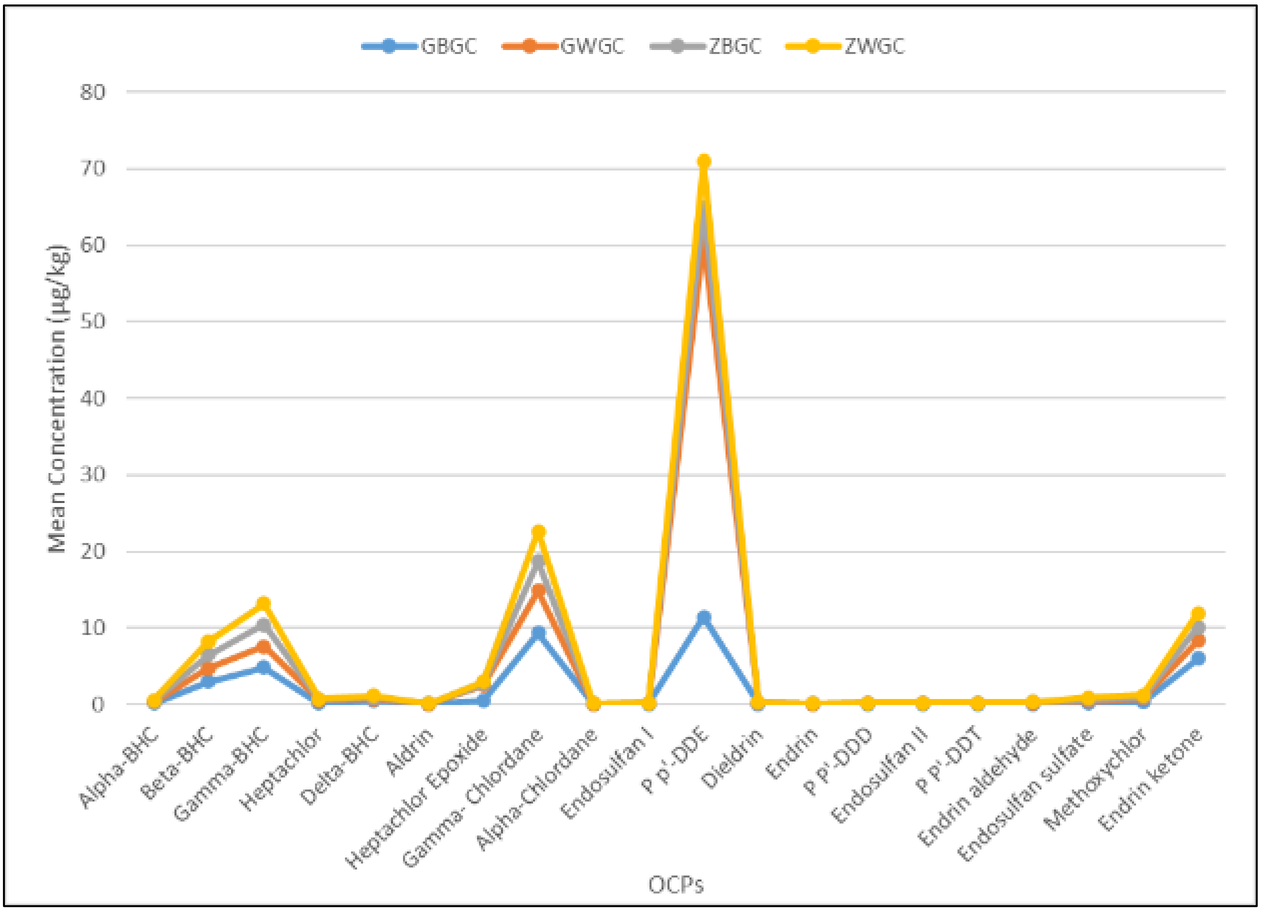

Figure 3 Graphical Representation of OCPs of Gaba and Zuma Guinea corn Samples

Gaba Maize Samples: The MCVs of organochlorine pesticides in GYM ranged from 0.03 ± 0.00 µg/kg to 26.34 ± 1.36 µg/kg and that of GWM ranged from 0.04 ± 0.02 µg/kg to 45.26 ± 1.13 µg/kg. Figure 4, shows the chart of concentrations of pesticide residue variations in maize samples from Gaba communities.

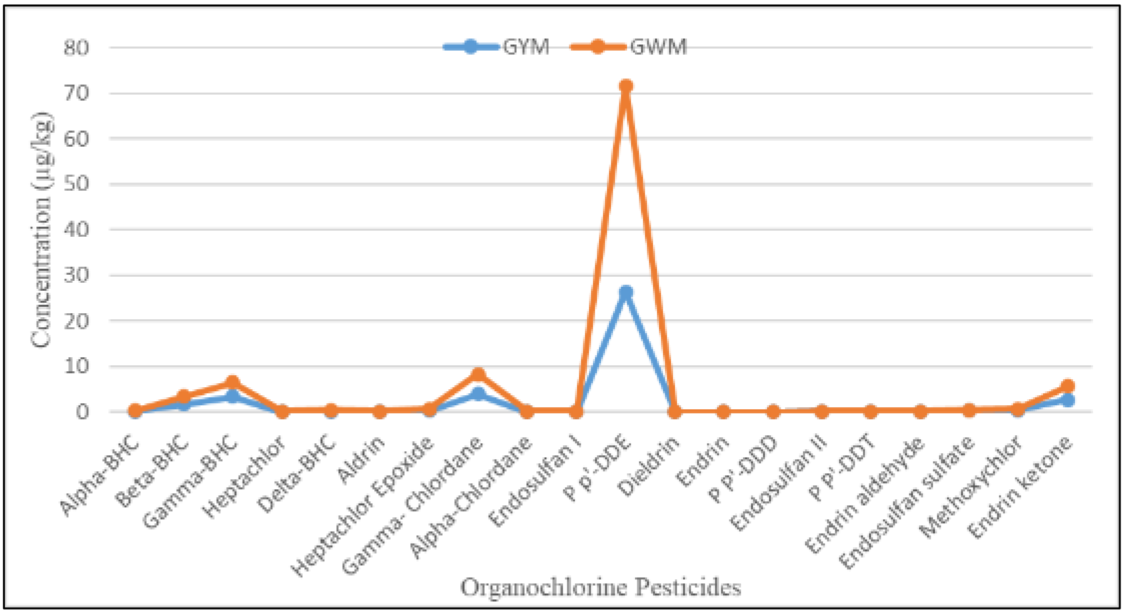

Figure 4 Graphical Representation of OCPs of Gaba Maize Samples

The results showed that most OCPs in the samples were below MRLs while a few were slightly above acceptable dietary intake (ADI) set by the European Union. Maize samples from Gaba communities were found to have metabolite products of DDT. The GYM had p,p'-DDE (26.34 µg/kg), p,p'-DDD (0.04 µg/kg), p,p'-DDT (0.11 µg/kg) and Methoxychlor (0.43 µg/kg) while GWM was with p,p'-DDE (45.26 µg/kg), p,p'-DDD (0.06 µg/kg), p,p'-DDT (0.04 µg/kg) and Methoxychlor (0.30 µg/kg). These values were within EU//FAO/WHO Maximum permissible Limits (MRLs) of 50 µg/kg (Raimi, 2021) for all metabolite products of DDT but higher than ADI limit of 20 µg/kg for p,p'-DDE, p,p'-DDD and p,p'-DDT. (World Health Organization, 2023a).

The concentration of DDT metabolites present in GYM and GWM were less than those reported by Oshatunberu *et al.* (2022) that monitored organochlorine pesticides contamination of cereals sold in selected markets in South West of Nigeria and that reported by Anzene *et al.* (2014) that studied organochlorine pesticide residue analysis of post-harvest cereal grains in Nasarawa State, Nigeria. Sosan *et al.* (2020), while accessing the dietary risk assessment of organochlorine pesticide residues in maize-based complimentary breakfast food products in Nigeria, reported the presence of p,p'-DDE (89 µg/kg); p,p'-DDT (282 µg/kg); and Methoxychlor (12 µg/kg).

Another study of organochlorine pesticide residues in cereals in Nigerian markets revealed that maize sold had mean p,p'-DDE contamination of 23 µg/kg and total DDT contamination of 66 µg/kg (Osibanjo and Adeyeye, 1995). Though, the levels of DDT metabolites reported in this study were within EU/CODEX/FAO Maximum Permissible Limits (MRLs), it would pose no health risks to consumers at the moment. Caution should therefore be taken that it is within limits and does not go beyond the permissible limits.

Zuma Maize Samples: The MCVs of organochlorine pesticide residues in ZYM ranged from 0.03 ± 0.01 µg/kg to 146.38 ± 1.13 µg/kg and that of ZWM ranged from 0.02 ± 0.01 µg/kg to 5.08 ± 0.70 µg/kg. Figure 5, shows the chart of concentrations of pesticide residue variations in maize samples from Zuma communities.

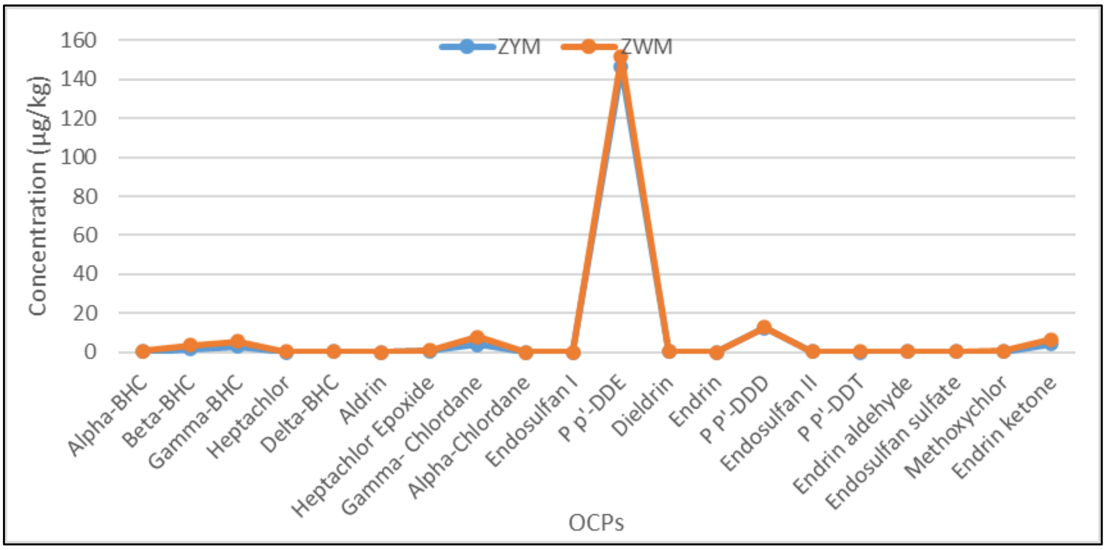

Figure 5 Graphical Representation of OCPs of Zuma Maize Samples

The results showed that most OCP residues in the tested samples were below maximum permissible limits (MLRs) and acceptable dietary intake (ADI) set by the European Union (EU).

Maize samples from Zuma communities were found to be contaminated with metabolite products of DDT. ZYM was contaminated with p,p'-DDE (146.38 µg/kg), p,p'-DDD (12.54 µg/kg) and p,p'-DDT (0.06 µg/kg). Also, ZWM was contaminated with p,p'-DDE (5.08 µg/kg), p,p'-DDD (0.08 µg/kg) and p,p'-DDT (0.07 µg/kg). These values were within EU/FAO/WHO Maximum Permissible Limits (MLRs) of 50 µg/kg (Raimi, 2021) for all metabolic products of DDT except for p,p'-DDE in ZYM. ADI of 20 µg/kg for p,p'-DDD and p,p'-DDT also met maximum standards (World Health Organization, 2023a).

The concentration of DDT metabolites present in ZYM and ZWM were similar to those reported by Oshatunberu *et al.* (2022) that monitored organochlorine pesticides contamination of cereals sold in selected markets in South West of Nigeria, and that reported by Anzene *et al.,* (2014) that studied organochlorine pesticide residues of post-harvest grains in Nasarawa State, Nigeria. Sosan *et al.* (2020) while accessing the dietary risk assessment of organochlorine pesticide residues in maize-based complimentary breakfast food products in Nigeria, reported the presence of p,p'-DDE (89 µg/kg) and p,p'-DDT (282 µg/kg).

Concentration of BHC isomers detected in the samples were less than the maximum permissible limits of both EU and FEPA except for the concentration of gamma-BHC (Lindane) which was higher than 0.3 µg/kg/body weight ADI limit set by EU/FAO (World Health Organization, 2023b).

When concentrations of all maize samples are compared: ZWM > ZYM > GWM > GYM.

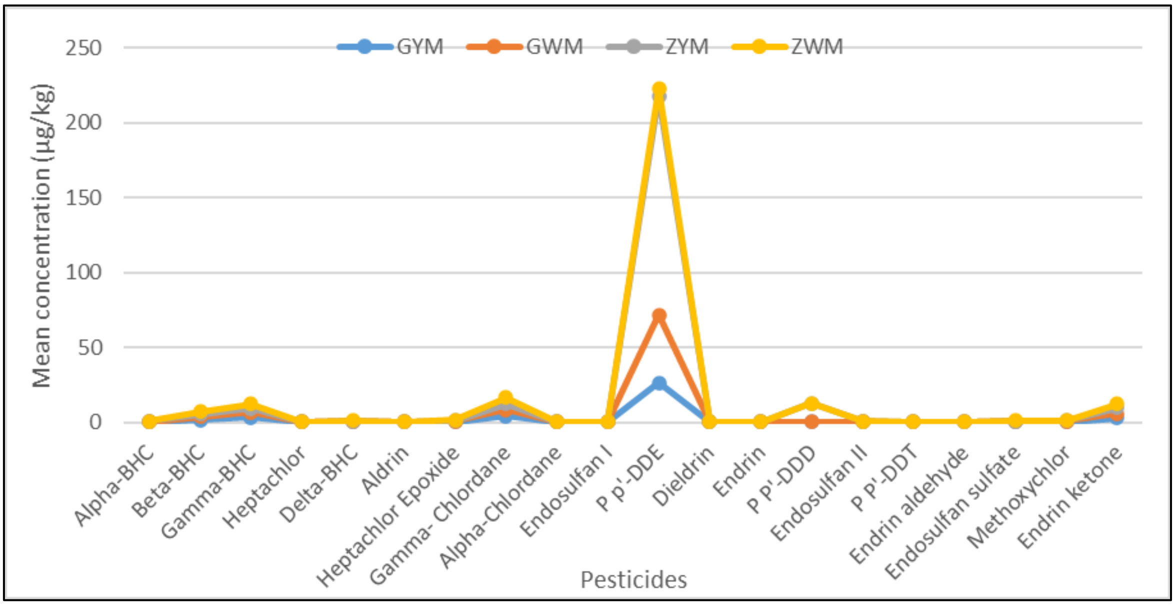

Figure 6 Graphical Representation of OCP residues in Gaba and Zuma Maize Samples

Gaba Cowpea Samples: The MCV of organochlorine pesticides in GBCP ranged from 0.03 ± 0.00 µg/kg to 4.31 ± 1.78 µg/kg and that of GWCP ranged from 0.02 ± 0.01 µg/kg to 4.73 ± 1.20 µg/kg.

Figure 7 shows the chart concentrations of pesticide residue variations in cowpea samples from Gaba communities.

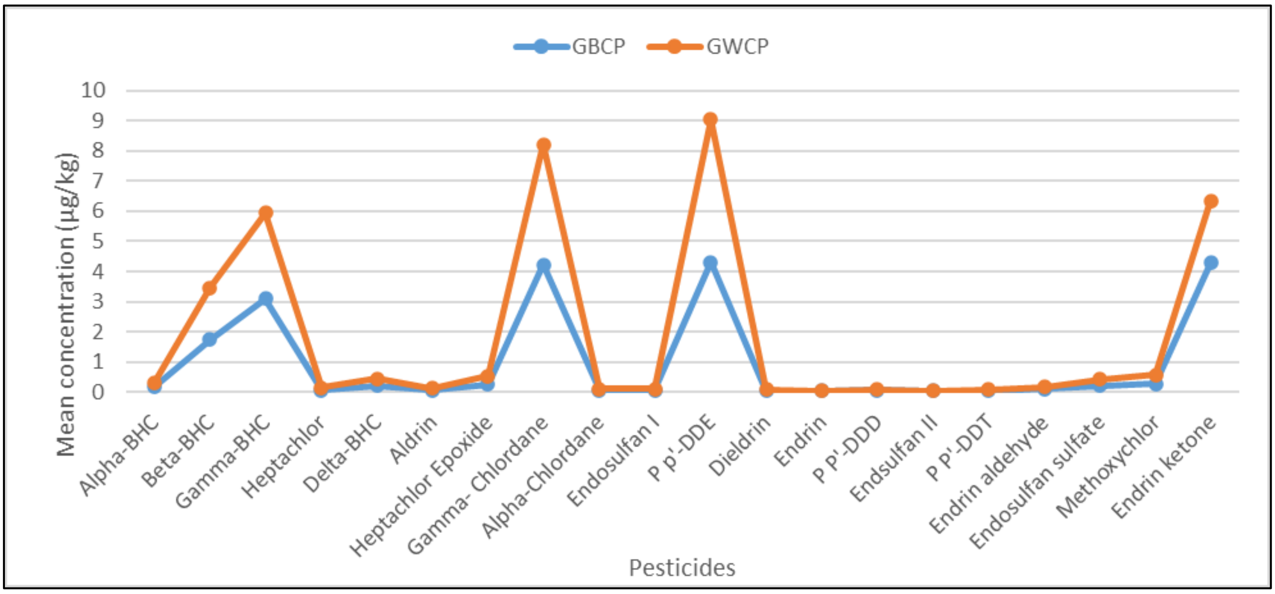

Figure 7 Graphical Representation of OCPs of Gaba Cowpea Samples

The results showed that most organochlorine residues in the tested samples were beyond maximum permissible limits (MLRs) and acceptable dietary intake (ADI) set by the European Union.

Cowpea samples from Gaba communities were found to have metabolite products of DDT. GBCP was contaminated with p,p'-DDE (4.31 µg/kg), p,p'-DDD (0.06 µg/kg), and p,p'-DDT (0.05 µg/kg). Also, GWCP was contaminated with p,p'-DDE (473 µg/kg), p,p'-DDD (0.02 µg/kg), and p,p'-DDT (0.04 µg/kg). Though these values were within EU/FAO/WHO Maximum Permissible Limits (MRLs) of 50 µg/kg (Raimi, 2021) for all metabolite products of DDT and ADI of 20 µg/kg for p,p'-DDD and p,p'-DDT set by EU/WHO/FAO (World Health Organization, 2023a). The concentrations of DDT metabolite detected in cowpea samples in this study were similar to values reported by Otitoju and Lewis, for cowpea sold in Wukari markets, Taraba State, Nigeria (Otitoju and Lewis, 2021).

In another study, Odion *et al.* (2020), observed DDT in cowpea samples ranging from 0.5 µg/kg to 11.42 µg/kg in selected markets in the Southwest of Nigeria. Also, Anzene *et al.* (2014) studied organochlorine pesticide residue analysis of post-harvest grains in Nasarawa State, Nigeria and reported cowpea contamination with metabolites of DDT similar to those reported in this study.

Concentrations of BHC isomers detected in samples were less than the maximum permissible limits of both EU and FEPA except for the concentration of gamma-BHC (Lindane) which was higher than 0.3 µg/kg/body weight ADI limit set by EU/FAO (World Health Organization, 2023b). In a similar study by Fadina *et al.* (2021), concentration of HBC in cowpea samples from selected markets in Ibadan ranged from 0.7 µg/kg to 3.8 µg/kg.

Zuma Cowpea Samples: The MCVs of organochlorine pesticide residues in ZBCP ranged from 0.02 ± 0.00 µg/kg to 5.36 ± 0.30 µg/kg and that of ZWCP ranged from 0.03 ± 0.01 µg/kg to 4.71 ± 0.22 µg/kg. Figure 8 shows the chart concentrations of pesticide residue variations in cowpea samples from Zuma communities.

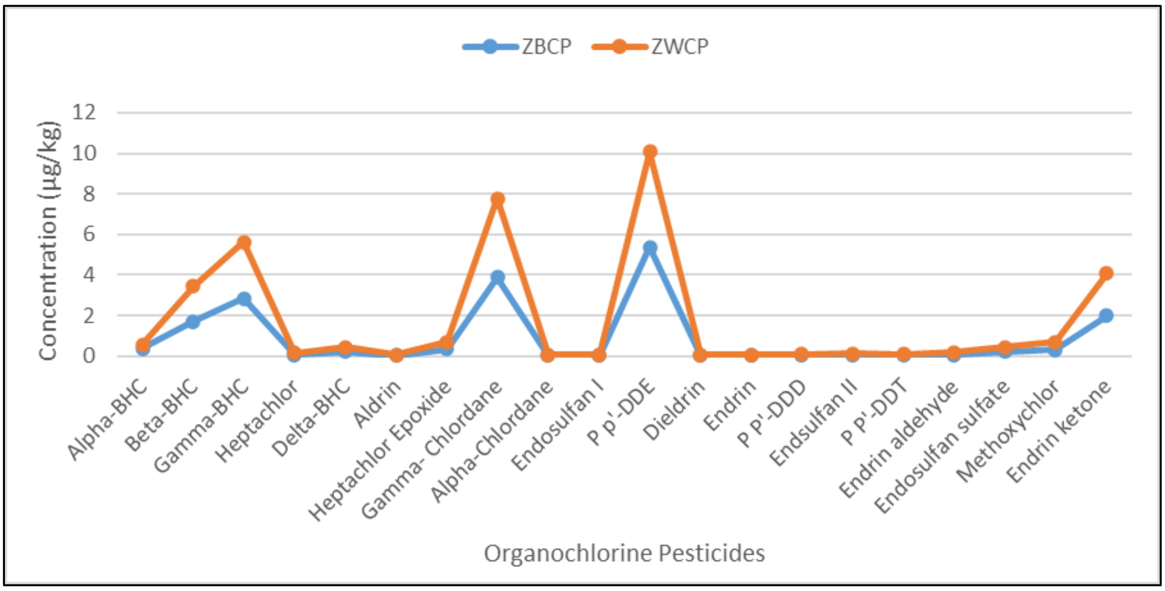

Figure 8 Graphical Representation of OCPs of Zuma Cowpea Samples

The results showed that most OCP residues in the studied samples were beyond maximum permissible limits (MRLs) and acceptable dietary intake (ADI) set by the European Union. Cowpea samples from Zuma communities were found to be contaminated with metabolite products of DDT. ZBCP was found to have p,p'-DDE (5.36 µg/kg), p,p'-DDD (0.05 µg/kg), p,p'-DDT (0.04 µg/kg) and methoxychlor (0.33 µg/kg). Also, ZWCP was observed to have p,p'-DDE (4.71 µg/kg), p,p'-DDD (0.05 µg/kg) and p,p'-DDT (0.07 µg/kg). These values were within EU/FAO/WHO Maximum Permissible Limits (MRLs) of 50 µg/kg (Raimi, 2021) for all metabolite products of DDT and ADI limit of 20 µg/kg for p,p'-DDD and p,p'-DDT and 5 µg/kg for Methoxychlor set by EU/WHO/FAO (World Health Organization, 2023a). In a related study, Fadina *et al.* (2021) reported cyclodienes concentration range of 16 – 46 µg/kg for beans samples obtained from selected markets in Ibadan.

Concentrations of cowpea samples from study sites shows; ZWCP > ZBCP > GWCP > GBCP.

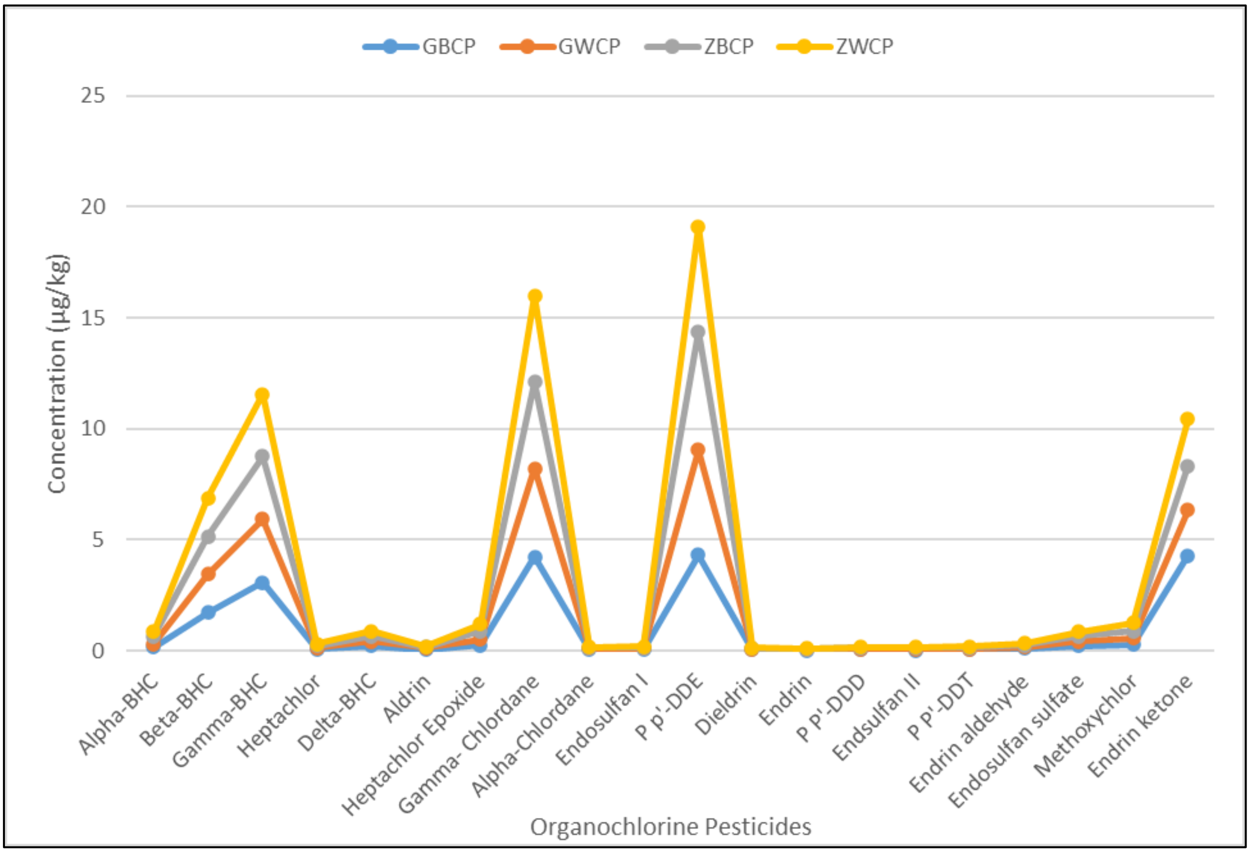

Figure 9 Graphical Representation of OCPs of Gaba and Zuma Cowpea samples

3.2 Health Risk Index for Food Crops

Health Risk Index is a tool used to assess the potential health risks associated with exposure to chemicals. This is often used in environmental and food safety concerns. This is carried out by comparing actual exposure levels to safe limits of those chemicals. It helps in the determination of negative health impact from exposure to chemicals; either through dermal contact, ingestion or inhalation. Health Risk Index is calculated with the ratio of actual exposure level to a reference dose or tolerable intake level for a particular chemical. It can be applied to food safety, environmental risk assessment or even workplace health.

The health risk index was therefore calculated using equation (2):

$$\text{Health Risk Index, } \left[HRI = \left\{\frac{EDI}{ADI}\right\} \times 100\right] \quad .... \quad (2)$$

Where,
HRI = Health Risk Index
EDI = Estimated Daily Intake
ADI = Adequate Daily Intake

$$\text{But, } EDI = \left[\frac{Cr}{bw} \times FIR\right] \quad ...\,.... \quad (3)$$

(where Cr is the Concentration of residue in sample, FIR is the Food Injection Rate and bw is the body weight).

The health risk index was calculated for all food crop samples. Gaba and Zuma maize samples would pose a health risk as it was found to be with Gamma-BHC and p,p'-DDE. Also, Zuma maize food crop samples were observed to have aldrin and gamma-Chlordane w ith values greater than 100. All food crop samples showed health risk index greater 100 for heptachlor epoxide except in Zuma cowpea sample. All samples showed values greater than 100 for Endrin ketone. Table 2 shows the table for health risk index of all food crops analyzed.

Table 2 Health Risk Index

Compound	Gaba Guinea corn	Zuma Guinea corn	Gaba Maize	Zuma Maize	Gaba Cowpea	Zuma Cowpea
Alpha-BHC	4.4388	10.0284	1.644	2.3016	2.1372	0.3288
Beta-BHC	1.096	0.822	1.096	1.37	0.822	0.274
Gamma-BHC	13.7	30.14	320.58	232.9	38.36	10.96
Heptachlor	32.88	24.66	32.88	41.1	16.44	8.22
Delta-BHC	0.1644	0.1644	0.3288	0.6576	0.1644	0.1644
Aldrin	41.1	16.44	24.66	156.18	90.42	0
Heptachlor Epoxide	320.58	353.46	279.48	345.24	197.28	49.32
Gamma- Chlordane	18.084	23.016	34.524	210.432	54.252	13.152
Alpha-Chlordane	3.288	4.932	8.22	23.016	13.152	1.644
Endosulfan I	0.3288	0.3288	0.822	1.1508	0.4932	0.1644
P p'-DDE	4.1922	9.5352	181.9497	337.5132	4.3155	1.2741
Dieldrin	65.76	16.44	65.76	65.76	41.1	24.66
Endrin	16.44	20.55	20.55	20.55	4.11	4.11
P P'-DDD	0.2466	0.2466	0.2055	0.3288	0.0411	0.1644
Endosulfan II	0.411	1.233	3.0825	0.6165	0.10275	0.71925
P P'-DDT	0.3699	0.1644	0.6987	0.1644	0.1233	0.2466
Endrin aldehyde	36.99	20.55	20.55	28.77	12.33	12.33
Endosulfan sulfate	1.233	1.781	3.151	1.918	0.959	0.548
Methoxychlor	5.754	4.7676	8.0556	3.7812	3.1236	1.1508
Endrin ketone	785.01	674.04	1237.11	1479.6	739.8	480.87

Values on table 1 which are 100 and above have high contaminant levels which would pose significantly negative health impact on its consumers. Whereas, values less than 100 are safe for human consumption. If allowed to increase, would pose a risk to consumers over time.

4. Conclusion

Man have battled the presence of pests for a very long time and how they cause post-harvest losses which have significantly impacted food safety and food security. It has caused contamination and eventually, degradation of food crops. This has necessitated the use of pesticides, most especially, the OCPs which do not only treat pests and prevent post-harvest losses, but impact the human health negatively.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare that no known conflict of interest or personal relationships that could have influenced the work reported in this paper.

Statement of ethical approval: The present research work does not contain any studies performed on animals/humans subjects by any of the authors.

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