

# Analysis of Pesticide Residues in Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) Obtained from Ogbadibo Local Government Area of Benue State

Onche Emmanuel\*, Eneji Shaibu Ishaq, Wuana Ahule Raymond

Department of Chemistry, Centre for Agrochemical Technology and Environmental Research (CATER), Joseph Sarwuan Tarka University, Makurdi, Nigeria

## Email address:

emmygen01@yahoo.com (Onche Emmanuel)

\*Corresponding author

## To cite this article:

Onche Emmanuel, Eneji Shaibu Ishaq, Wuana Ahule Raymond. Analysis of Pesticide Residues in Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) Obtained from Ogbadibo Local Government Area of Benue State. *Journal of Energy, Environmental & Chemical Engineering*. Vol. 8, No. 1, 2023, pp. 10-17. doi: 10.11648/j.jeece.20230801.12

Received: March 9, 2023; Accepted: March 28, 2023; Published: April 11, 2023

**Abstract:** The abuse of agrochemicals in agricultural practise in order to boost food production has generated environmental concern in terms of their residues. This study was carried out to determine pesticide residues of plantain and banana from Benue State, Nigeria due to unregulated applications of pesticides, leading to MRL non-compliances. QuEChERS and buffered ethyl acetate extraction methods were employed for diverse range of pesticides in plantain and banana, followed by selective identification and quantification of the residues using GC-MS technique. The method was evaluated for 23 organochlorine pesticides. The results of analysis showed that Plantain contains a wide range of pesticide residues with the highest BHC  $2.4 \times 10^{-5}$  ppb and Endosulfan 1  $0.2 \times 10^{-5}$  ppb, being the lowest in residues. Banana shows slight range with BHC  $1.6 \times 10^{-5}$  ppb, the highest and Methoxychlor  $0.6 \times 10^{-5}$  ppb, the lowest residues. This result showed that their limits of quantification were 0.1-10 µg/g, with recoveries of 70-120%. The method provided superior performance in terms of precision, and recovery of the spiked and incurred residues with similiar productivity. Generally, the findings revealed samples of plantain and banana showed some degree of contamination and it is advisable banned pesticides should not be sold in Benue State any more.

**Keywords:** Banana, GC-MS, Pesticides Residues, Plantain, QuEChERS

## 1. Introduction

Pesticides are extensively used in agricultural production to control pests, diseases, weeds and other plant pathogens that reduce yield and also extend the storage life of food commodities [14]. Nevertheless, the abuse of pesticides has resulted in a widespread distribution of residues in soil, water, air and crops.

There have been reports of poison cases and sudden deaths, attributed to the use and inappropriate storage of pesticides. The rising cases of food poisoning in Nigeria were linked to the misuse and abuse of agrochemicals and pesticides on grains and other agricultural products [27]. Pesticides in food materials are poorly monitored in Nigeria with no information available on the permissible levels of pesticides in food commodities. Hence, there is still a paucity of data on the

dietary intake of pesticides by the Nigerian population [22].

According to [21] the food commodities from Nigeria were banned from Europe till June 2016. The European Food Safety Authority had said that the rejected food commodities were found to contain between 0.03 mg/kg to 4.6 mg/kg of dichlorvos pesticide, when the acceptable maximum residue limit is 0.01 mg/kg. In Nigeria, most-deadly chemicals and organochlorine pesticides are used due to their being cheaper than newer and safer pesticides [9]. The research confirm the use of dichlorvos in a multiple response schedule discovered that aluminium phosphate tablets ranked 80 % for storage pesticide while dichlorvos was 60 %, DDT 35 % [20]. Others such as endosulfan, gamalin, carbofuran, carbendazim and permethrin were between 5-15 %. Furthermore, the study revealed the use of many restricted and obsolete pesticides within Benue state and in outrageous quantities which could

pose health challenge to consumers of these food products.

Several relevant bodies and organizations are taking the initiative to ensure safe use of pesticides in Nigeria. Government agencies such as National Environmental Standards and Regulations Enforcement Agency (NESREA), National Agency for Food and Drug Administration and Control (NAFDAC), the Cocoa Research Institute of Nigeria (CRIN), the Nigeria Stored Products Research Institute (NSPRI), are at the forefront of translating research findings to regulations and communicating these to the nation through various workshops. There are also private organizations such as the Pest Control Association of Nigeria (PECAN) and the West African Agricultural and Productivity Programme (WAAPP-Nigeria) working to ensure safe use of pesticides in Nigeria [17]. The aim of this study is to audit the commonly used pesticides for Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) in Ogbadigbo L. G. A., to extract the pesticide residues from Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) obtained from Ogbadigbo L. G. A. and to analyse the level of pesticide residues in Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) using GC-MS.

## 2. Materials and Methods

### 2.1. Reagents

The following analytical high grade chemicals were used. All organic solvents intended for extraction were GC-MS grade. Organophosphate and Organochlorine Pesticide containing a mixture of glyphosate Standards (AccuStandards), Analytical grade n-hexane (Aldrich), analytical grade acetone (Aldrich), anhydrous  $\text{MgSO}_4$  (Lobachemie, India), anhydrous Sodium acetate, Silica Gel (Lobachemie, India), distilled water, primary secondary amine (PSA), 1 % acetic acid in acetonitrile (v/v) and were used.

### 2.2. Equipment

Hewlett Packard (HP) 6890 GC/MS, equipped with dual injector and column, sample vials was used alongside gilson pipette, 200 mL beaker, spatula, weighing balance, blender (Kenwood), ultrasonic bath (Clean 120-HD), extraction tubes, rotary evaporator (Buchi R215), sintered glass column for liquid chromatography (2 mm diameter), (HP) 5 Column (Length; 30 mL).

### 2.3. Study Areas

Samples of Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) were purchased from open markets across Ogbadibo Local Government Area of Benue State. A map and brief description of the study area shown below in Figures 1.

Benue State lies within the lower River Benue trough in the middle belt region of Nigeria with Landmass of 34,059 square kilometers and a population of 4,253,641 in 2006 census. Agriculture is the mainstay of the economy engaging over 75% of the working population and its geographic coordinates are longitude 7° 47' and 10° 0' East, Latitude 6°

25' and 8° 8' North and it is characterized by an annual rainfall of 1250 -1500 mm.

### 2.4. Sample Collection

The sampling is focused on the Otukpa branch market as the end point of the food distribution in that locality. About 1 kg of each of the Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) was purchased randomly from three different sellers and composited to represent one sample of that Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) from the market at that visit. This process was conducted for all the samples of the Plantain (*Musa paradisiacal*) and Banana (*Musa acuminata*) in the markets in the month of August, 2021. The samples were code-named and stored in cellophane bags with tight covers to protect them from moisture and contamination. Samples were stored in a refrigerator at 4°C until further analysis.

### 2.5. Sample Preparation and Digestion

Sample preparation was achieved by washing the sample in a clean tap water to remove dirt. Each sample was homogenized by blending to smaller particle size using a blender (Kenwood), to obtain uniform representative samples.

Sonication extraction method was employed as described by [26] with slight modifications. Five grams of the plant material was soaked in 10 mL distilled water. Hexane and acetone (1:1) (20 mL) was added to the mixture and sonicated at 37°C for 20 minutes. The mixture was carefully decanted, and the clear supernatant concentrated to about 2 mL using the rotary evaporator.

### 2.6. Sample Clean-up

Sample extraction and clean-up was achieved by adopting the multi-residue pesticide analysis technique consisting of QuEChERS sample extraction method [15].

Twenty grams (20 g) of silica gel which was previously activated for at least 6 hours at 130°C (in a petri dish, loosely covered with foil) was used to pack the glass column. Two grams (2 g) of anhydrous sodium sulphate was also transferred to the column, then the column was conditioned with n-hexane (10 mL). The extract was eluted using n-hexane (20 mL). The eluent was then concentrated to 2 mL using the rotary evaporator. The sample extracts were then stored in the refrigerator at 4°C for gas chromatography-mass spectrometer analysis [2].

### 2.7. Calibration of the Instrument (Quality Control and Quality Assurance)

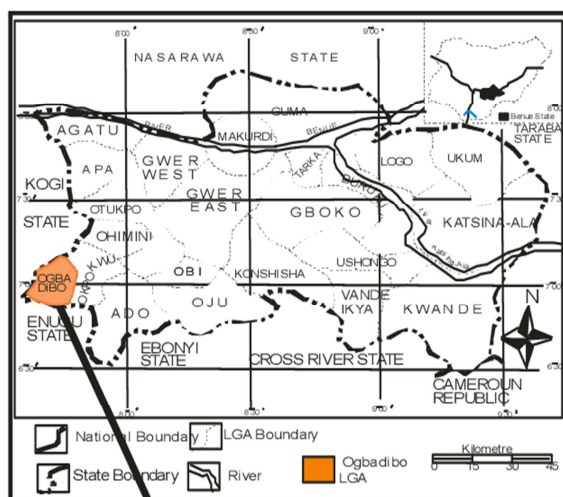
Quality control and quality assurance were considered to ensure accuracy and precision of results and the method. Five (5) point serial dilution calibration standards (0.001 - 2.50 ppm) were prepared from the stock and used to calibrate the GC-MS [19].

Glyphosate containing a mixture of 360g/L was used as the source and stock concentration standard. Afterwards, six (6) point serial dilution calibration standards (1.00, 5.00,

10.00, 15.00, 20.00, 25.00  $\mu\text{L/mL}$ ) were prepared from the sub-stock and used to calibrate the GC-MS. Limits of detection were based on the lowest concentration of the residues in each matrix that could be measured at the operating conditions of the GC which was 0.0001 mg/kg. Prior to calibration, the mass spectrometer (MS) was auto tuned to perfluorotributylamine (PFTBA) using already established criteria to check the abundance of  $m/z$  69, 219,

502 and other instrument optimal and sensitivity conditions. Determination of the levels of organochlorine pesticides in the sample was carried out using GC-MS by operating mass spectrometer detector (MSD) in selective ion monitoring (SIM) and Scan mode to ensure detection of the target constituents [23].

Sample analyses were performed using a Hewlett Packard (HP) 6890 GC/MS, equipped with dual injector and column.



Map of Benue State Showing Ogbadibo LGA

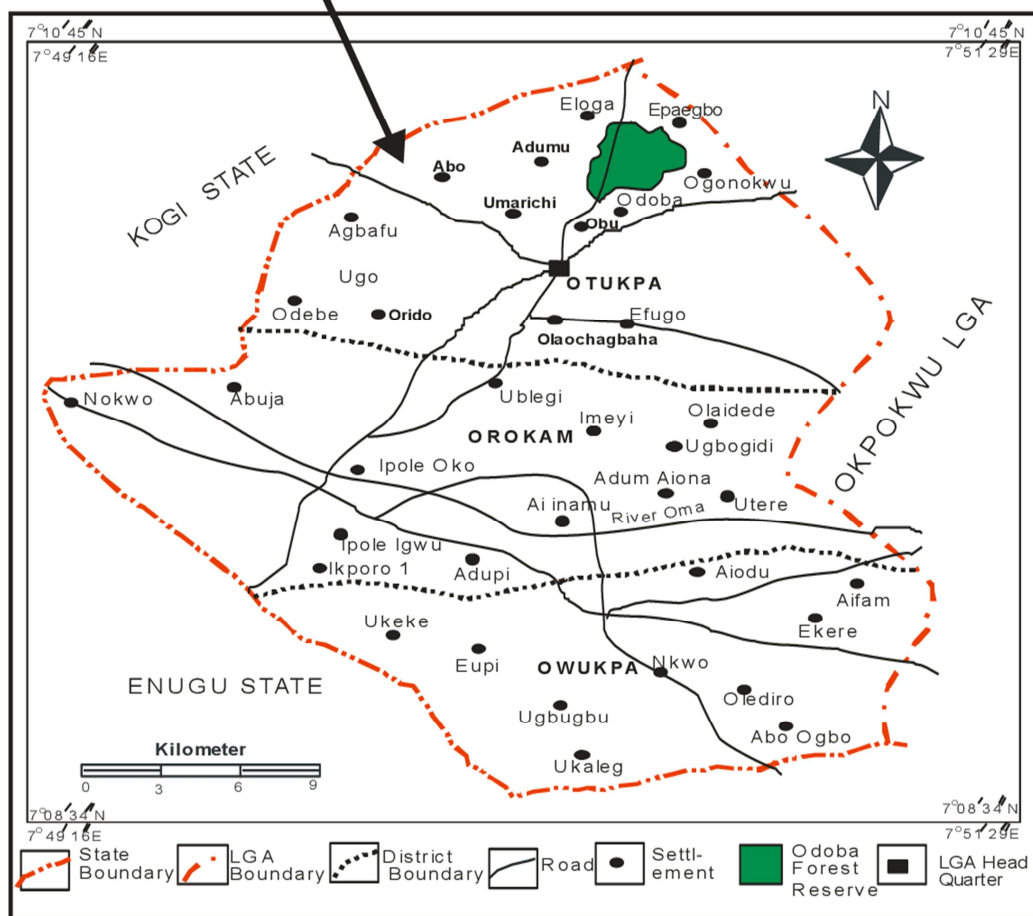


Figure 1. Map of Ogbadibo Local Government Area.

**Table 1.** HP 6890 Conditions used for the PAH Analysis.

Instruments	Conditions
Injector Temperature 2	80°C
Oven Temperature Program	130°C hold for 1 minute (min) 5°C/min to 300°C, 9 min at 300°C
Detector Temperature	320°C
Carrier Gas	Helium
Make-up Gas	Argon/Methane
Column Flow Rate	RTX-XLB2.0 milliliters/minute (mL/min); Rtx-CLPAH 1.0 mL/min
Amount Injected	1 microliter (uL)
Data System	HP Chem Station

## 2.8. Method of Identification

After calibration, the sample extracts was analysed for residues of OCPs and Glyphosate. The final extracts were directly employed for gas chromatography with mass spectrometric detection (GC-MS-SIM) for identification and determination with limits of detection 0.0001 mg/kg. The limits of determination for many residues can be as low as from 0.0003-0.01 mgkg<sup>-1</sup> [16].

### 2.8.1. Recovery Study

Recovery studies was undertaken to evaluate the efficiency of the extraction procedure used. The recovery of pesticides were achieved at three range concentrations in the matrix each, by using blank local milled rice samples in accordance with the EC guidelines [8]. About 2.0 g of each blank sample, after homogenization, were spiked by adding appropriate volumes of pesticide standard mixture in a solution of 1% acetic acid in acetonitrile (v/v). The spiked sample was left to stand for an hour (equilibrium times) then extracted, cleaned up and analyzed like the test samples. The recovery values expressed in percentages were calculated from the chromatograms as:

$$\% \text{ Recovery} = \frac{CS_2 - CS_1}{CS} \times 100 \quad (1)$$

where; CS<sub>1</sub>=Concentration of pesticide residues in original sample

CS<sub>2</sub>=Concentration of pesticide residues in spiked

CS=Concentration of added pesticide

### 2.8.2. Determination of Limit of Detection (LOD)

The limit of detection of the pesticide residues was determined by replicate chromatographic runs (6 times) of the least concentration of the pesticide standards and then multiply the standard deviation obtained by 3.

### 2.8.3. Determination of Limit of Quantification (LOQ)

The limit of quantification of the pesticide residues was determined by replicate chromatographic runs (6 times) of the least concentration of the pesticide standards and then multiplying the standards deviation by 10.

### 2.8.4. Identification and Quantification

Pesticide residues were identified and the retention times matched those of the standards and the relative abundances will be within 10% of those of the standards. Identified

pesticides were quantified using the external standards method of comparing sample peak areas with those of the pesticide standards under the same conditions. Each sample were analyzed three times and the mean values determined as:

$$\text{Pesticide content} = \text{As} \times \text{Wts} \times \text{vf} - \text{CF} \quad (2)$$

where; As = peak area of sample

Vf = final volume of clean extract

Wts= weight of sample extracted

CF =Calibration factor.

The calibration factor of each pesticide will be calculated as:

$$\text{CF} = \frac{\text{Peak Area of Standard} - \text{Total Amount of Standard injected.}}{\quad} \quad (3)$$

### 2.8.5. Dietary Estimate

$$\text{Dietary exposure} = \frac{\sum (A \times B)}{C \text{ (kg)}} \quad (4)$$

where; A= Residue

B= Food consumption per person per day

C= Body weight [28]

$$\text{Hazard Index (HI)} = \text{EDI/ADI} \quad (5)$$

Human exposure to pesticides is conventionally estimated based on monitoring of pesticide residues in environmental matrices and food. Using the mathematical equation (3.1) we can convert the determined concentrations of pesticide residues in these foods to Estimated Daily Intake.

## 3. Results and Discussion

### 3.1. Quality Control Parameters

The quality control parameters are presented in Tables 2 and 3. The LOQ ranged from 0.1×10<sup>-5</sup> ppb to 3.0×10<sup>-5</sup> ppb and LOD ranged from 0.01×10<sup>-6</sup> ppb to 0.1×10<sup>-6</sup> ppb indicating the high sensitivity of the gas chromatograph at the operating conditions. The mean and standard deviation were calculated from the detectable values, and values below the detectable limit were considered not detected (ND). The calibration curves of the analyzed pesticides presented good regression lines in the range of explored concentrations.

**Table 2.** Quality Control Parameters for Plantain.

Name of pesticide	Quality control parameters		
	Mean (10 <sup>-5</sup> ppb)	LOD (10 <sup>-6</sup> ppb)	LOQ (10 <sup>-5</sup> ppb)
BHC	2.4±0.3	0.9	3.0
Diazion	1.6±0.3	0.9	3.0
Aldrin	1.6±1.0	3.0	10.0
Mirex	2.0±0.1	0.3	1.0
Endosulfan I	0.2±0.07	0.21	0.7
DDT	2.2±0.3	0.9	3.0
Dimethoate	1.0±0.1	0.3	1.0
Cypermethrin	1.4±0.1	0.3	1.0
Deltamethrin	1.6±0.3	0.9	3.0

**Table 3.** Quality Control Parameters for Banana showing Mean Conc. LOD and LOQ.

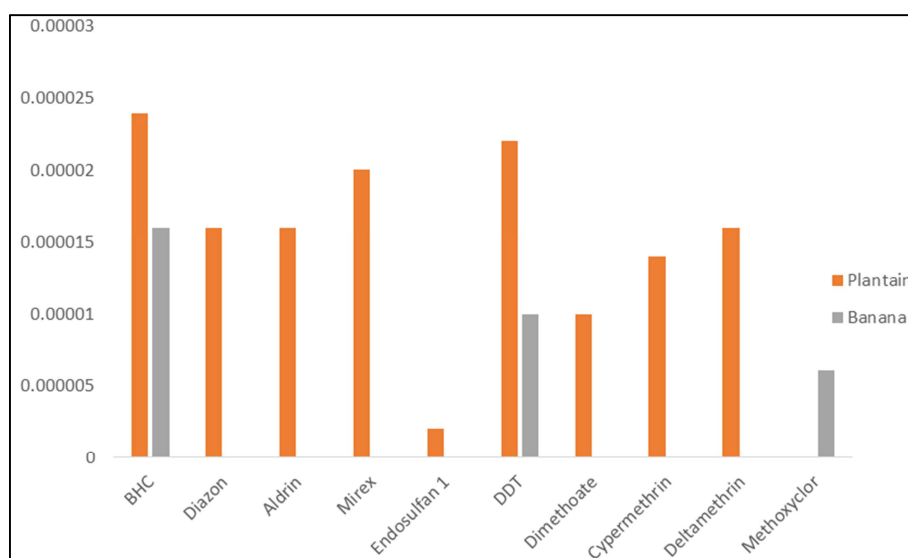
Name of pesticide	Quality control parameters		
	Mean ( $10^{-5}$ ppb)	LOD ( $10^{-6}$ ppb)	LOQ ( $10^{-5}$ ppb)
BHC	$1.6 \pm 0.1$	0.3	1.0
DDT	$1.0 \pm 0.1$	0.3	1.0
Methoxychlor	$0.6 \pm 0.2$	0.6	2.0

### 3.2. Level of Pesticide Residues in Plantain and Banana

The general descriptive results of pesticide residues as presented in Table 1 for Plantain shows that, twenty three (ocp's) commonly used pesticides in plantain and banana cultivation were determined. The frequently occurring residues were those of BHC, diazon, Aldrin, mirex,

endosulfan 1, DDT, dimethoate, cypermethrin, and deltamethrin, while dichlorvos, heptachlor, heptachlor epoxide, o-terphenyl, chlordane, chlorpyrifos, diedrin, endrin, endosulfan II, ddpv, parathion, methoxychlor, carbanyl was not detected in most of the plantain samples.

Table 2 for Banana shows that, twenty three (ocp's) commonly used pesticides in plantain and banana cultivation were determined. The frequently occurring residues were those of BHC, DDT and methoxychlor while dichlorvos, ethion, diazion, heptachlor, aldrin, heptachlor epoxide, mirex, endosulfan I, o-terphenyl, chlordane, chlorpyrifos, diedrin, endrin, endosulfan II, DDVP, parathion, dimethoate, cypermethrin, deltamethrin, carbonyl was not detected in most of the banana samples.



**Figure 2.** Mean Concentration of Plantain and Banana.

**BHC:** Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of BHC ranged from  $2.1 \times 10^{-5}$  to  $2.7 \times 10^{-5}$ . The LOD and LOQ were  $1.0 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $3 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain two sample and lowest in plantain one sample collected, as observed in the following pattern: Plantain 2 ( $2.7 \times 10^{-5}$ ) > Plantain three ( $2.4 \times 10^{-5}$ ) > Plantain one ( $2.1 \times 10^{-5}$ ). The concentration of BHC in the various samples of plantain were generally lower than its acceptable maximum residue level (0.01ppm) set by [10] and 30.0 ( $\mu\text{g}/\text{kg}$ ) (USEPA MRL).

Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of BHC ranged from  $1.3 \times 10^{-5}$  to  $1.9 \times 10^{-5}$ . The LOD and LOQ were  $1.0 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $3.3 \times 10^{-6}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in Banana one sample and lowest in banana two sample collected, as observed in the following pattern:

Banana one ( $1.9 \times 10^{-5}$ ) > Banana three ( $1.6 \times 10^{-5}$ ) > Banana two ( $1.3 \times 10^{-5}$ ). The concentration of BHC in the various samples of plantain were generally lower than its acceptable maximum residue level (0.01ppm) set by [10].

The presence of BHC was reported with highest mean concentration (14.85  $\mu\text{g}/\text{kg}$ ), followed by  $\delta$ -HCH (6.51  $\mu\text{g}/\text{kg}$ ) and then  $\beta$ -HCH (3.15  $\mu\text{g}/\text{kg}$ ) which are higher than the study

samples [13].

The occurrence of the BHC in the samples could be due to high persistent nature of the pesticides. The presence of BHC samples may suggest the historical use or illegal use of technical HCH mixtures in the study area, since technical BHC have been officially discontinued as restricted chemical for use.

**Diazinone:** Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of diazinone ranged from  $1.2 \times 10^{-5}$  to  $2.0 \times 10^{-5}$ . The LOD and LOQ were  $1.0 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $3.0 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain one sample and lowest in plantain two sample collected, as observed in the following pattern:

Plantain Two ( $2.0 \times 10^{-5}$ ) > Plantain three ( $1.6 \times 10^{-5}$ ) > Plantain one ( $1.2 \times 10^{-5}$ ). The concentration of diazinon in the various samples of plantain were generally lower than its acceptable maximum residue level (0.03  $\mu\text{g}/\text{kg}$ ) set by USEPA.

Diazinon was reported with mean concentration of (20.50  $\pm$  8.9  $\mu\text{g}/\text{kg}$ ) which is higher than the study samples [13].

**Aldrin:** Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of aldrin ranged from  $1.4 \times 10^{-5}$  to  $1.9 \times 10^{-5}$ . The LOD and LOQ were  $0.1 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $10 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest

concentration of was recorded in plantain 1 sample and lowest in plantain 2 sample collected, as observed in the following pattern:

Plantain one ( $1.9 \times 10^{-5}$ ) > Plantain three ( $1.7 \times 10^{-5}$ ) > Plantain two ( $1.4 \times 10^{-5}$ ). The concentration of aldrin in the various samples of plantain were generally lower than its acceptable maximum residue level (0.1 mg/kg) set by set by Codex Alimentarius MRL, (0.01ppm) EU pesticides database and 40.0 ( $\mu\text{g}/\text{kg}$ ) USEPA MRL.

Aldrin was reported with mean concentration of (1.72  $\mu\text{g}/\text{kg}$ ), the parent compound, was higher than that of dieldrin (1.39  $\mu\text{g}/\text{kg}$ ) but lower than the endrin concentration (4.68  $\mu\text{g}/\text{kg}$ ) which are all higher than the study samples [13].

*Mirex*: Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of mirex ranged from 0.000018 to 0.000022. The LOD and LOQ were  $0.33 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $1.0 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain one sample and lowest in plantain two sample collected, as observed in the following pattern:

Plantain one ( $2.2 \times 10^{-5}$ ) > Plantain two ( $2.0 \times 10^{-5}$ ) > Plantain three ( $1.8 \times 10^{-5}$ ). The concentration of mirex in the various samples of plantain were generally lower than its acceptable maximum residue level (0.01 mg/kg) set by FAO/WHO codex alimentarius standards for MRLs.

Very few studies have examined mirex. More recently, mirex was reported at lower and upper bound levels of 1.0 and 3.5 ng/g lipid in China [29]. These levels are comparable the level found in this study 0.000020 mg/kg mirex..

*Endosulfan 1*: Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of endosulfan 1 ranged from  $1.9 \times 10^{-6}$  to  $2.1 \times 10^{-5}$ . The LOD and LOQ were  $0.3 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $0.7 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain one sample and lowest in plantain two sample collected, as observed in the following pattern:

Plantain two ( $2.1 \times 10^{-6}$ ) > Plantain three ( $2 \times 10^{-6}$ ) > Plantain one ( $1.9 \times 10^{-6}$ ). The concentration of endosulfan in the various samples of plantain were generally lower than its acceptable maximum residue level (0.5ppm) set by FAO/WHO codex alimentarius standards for MRLs.

Endosulfan 1 was reported with mean concentration of ( $2.48 \pm 0.4$   $\mu\text{g}/\text{kg}$ ), which is higher than the study samples [13].

*DDT*: Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of DDT ranged from  $1.8 \times 10^{-6}$  to  $2.2 \times 10^{-6}$ . The LOD and LOQ were  $0.2 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $3.0 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain one sample and lowest in plantain two sample collected, as observed in the following pattern:

Plantain two ( $2.2 \times 10^{-5}$ ) > Plantain three ( $2.0 \times 10^{-5}$ ) > Plantain one ( $1.8 \times 10^{-5}$ ). The concentration of DDT in the various samples of plantain were generally lower than its acceptable maximum residue level 50.0 ( $\mu\text{g}/\text{kg}$ ) USEPA MRL.

Mean residue concentration (ppb) of DDT ranged from  $0.8 \times 10^{-5}$  to  $1.2 \times 10^{-5}$ . The LOD and LOQ were  $3.3 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $1.0 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  respectively. The highest concentration of was recorded in banana 1 sample and lowest in banana 2

sample collected, as observed in the following pattern:

Banana one ( $1.2 \times 10^{-5}$ ) > Banana three ( $1.0 \times 10^{-5}$ ) > Banana 2 ( $0.8 \times 10^{-5}$ ). The concentration of DDT in the various samples of plantain were generally lower than its acceptable maximum residue level (0.2 mg/kg) set by FAO/WHO codex alimentarius standards for MRLs (50ppm) set by Codex Alimentarius MRL in food [25]. EU MRL is 0.05 $\mu\text{g}/\text{kg}$ . The mean value of DDT recorded in the study was lower than the mean value of  $0.04 \pm 0.01$  ( $\mu\text{g}/\text{g}$ ) reported in watermelon [5]. The DDT reported in Kolanut samples ( $0.480 \text{ mg kg}^{-1}$ ) is higher than the study sample [22].

*Dimethoate*: Mean residue concentration ( $\mu\text{g}/\text{kg}$ ) of dimethoate ranged from  $0.9 \times 10^{-5}$  to  $1.1 \times 10^{-5}$ . The LOD and LOQ were  $0.01 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $1.0 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain one sample and lowest in plantain two sample collected, as observed in the following pattern:

Plantain two ( $1.1 \times 10^{-5}$ ) > Plantain 3 ( $1.0 \times 10^{-5}$ ) > Plantain 1 ( $0.9 \times 10^{-5}$ ). The concentration of dimethoate in the various samples of plantain were generally lower than its acceptable maximum residue level (0.01ppm) set by EU pesticides database and (0.03  $\mu\text{g}/\text{kg}$ ) for USEPA.

Dimethoate was reported with mean concentration of ( $33.42 \pm 24.0$   $\mu\text{g}/\text{kg}$ ), which is higher than the study samples [13].

*Cypermethrin*: Mean residue concentration (ppb or  $\mu\text{g}/\text{kg}$ ) of cypermethrin ranged from  $1.8 \times 10^{-5}$  to  $2.2 \times 10^{-5}$ . The LOD and LOQ were  $0.03 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $1.0 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain 1 sample and lowest in plantain 2 sample collected, as observed in the following pattern:

Plantain two ( $2.2 \times 10^{-5}$ ) > Plantain three ( $2.0 \times 10^{-5}$ ) > Plantain one ( $1.8 \times 10^{-5}$ ). The concentration of cypermethrin in the various samples of plantain were generally lower than its acceptable maximum residue level (50ppm) set by Codex Alimentarius and (0.01ppm) EU pesticides database.

Pesticide residues like chlorpyrifos, cypermethrin, deltamethrin and dichlorvos were monitored by an improved extraction method from apple, peach and potato using reverse phase high performance liquid chromatography. Maximum residue levels (MRL), 0.924, 1.630 and 0.454  $\mu\text{g}/\text{g}$  for chlorpyrifos, cypermethrin and deltamethrin were found in peach respectively [4]. The MRL 0.283  $\mu\text{g}/\text{g}$  for dichlorvos was found in apple samples. All these observed results were compared with Codex maximum residue levels. It was found that cypermethrin and chlorpyrifos residual amounts were greater than MRLs, so their spray on the target crops should be limited because these are resistant to the degradation. In guava (0.01 mg/kg), mango (0.04 mg/kg), litchi (0.10 mg/kg), ginger garlic (0.42 mg/kg), Cypermethrin was detected and 0.05 ppm of Cypermethrin in ginger paste as reported by [3] this is also higher than the study samples.

*Deltamethrin*: Mean residue concentration (ppb or  $\mu\text{g}/\text{kg}$ ) of deltamethrin ranged from  $1.4 \times 10^{-5}$  to  $1.9 \times 10^{-5}$ . The LOD and LOQ were  $0.1 \times 10^{-6}$   $\mu\text{g}/\text{kg}$  and  $3.0 \times 10^{-5}$   $\mu\text{g}/\text{kg}$ , respectively. The highest concentration of was recorded in plantain 1 sample and lowest in plantain 2 sample collected,



as observed in the following pattern:

Plantain two ( $1.9 \times 10^{-5}$ ) > Plantain three ( $1.5 \times 10^{-5}$ ) > Plantain one ( $1.9 \times 10^{-5}$ ). The concentration of deltamethrin in the various samples of plantain were generally lower than its acceptable maximum residue level (0.2 mg/kg) set by set by Codex Alimentarius and (0.01ppm) EU pesticides database.

Analysis of pesticide residues in fruit juices and vegetable paste, shows deltamethrin was not detected but in vegetable paste it was detected in ginger garlic paste (1.28 mg/kg) [7]. About 0.05 ppm of Deltamethrin was detected in ginger paste [3].

**Methoxychlor:** Mean residue concentration ( $\mu\text{g/kg}$ ) of methoxychlor ranged from  $0.4 \times 10^{-5}$  to  $0.8 \times 10^{-5}$ . The LOD and LOQ were  $6.6 \times 10^{-6} \mu\text{g/kg}$  and  $2.0 \times 10^{-5} \mu\text{g/kg}$ , respectively. The highest concentration of was recorded in banana one sample and lowest in banana two sample collected, as observed in the following pattern:

Banana one ( $8 \times 10^{-5}$ ) > Banana three ( $6 \times 10^{-5}$ ) > Banana two ( $4.0 \times 10^{-5}$ ). The concentration of methoxychlor in the various samples of plantain were generally lower than its acceptable maximum residue level (0.01ppm) set by Japan pesticides MRLs in banana [24] which is same with EU MRL pesticides database. The mean value of methoxychlor recorded in the study was lower than the mean value of 0.002 mg/kg reported by [12] in cocoa beans from Ghana. However, the finding of methoxychlor in this study was contrary to the findings of [11] which reported no methoxychlor residue in cocoa beans ready for export in Ghana. The presence of methoxychlor in the cocoa bean samples analysed suggests that methoxychlor had either been used on cocoa in the past (presence in the environment had not degraded) and/or currently illegally used, since they are banned for agricultural purposes in Ghana [18]. In addition, the detection of methoxychlor may be either as a result of historical use of DDT of which technically methoxychlor contains about 88 % of the p,p'- isomer together with more than 50 structurally related contaminants, which might have been added to the actual amount of methoxychlor present [6, 1].

## 4. Conclusion

Pesticide audit has been able to proof that some commonly used pesticides like Aldrin, DDT, BHC, Endosulphan, Mirex, Heptchlor and Dieldrin) which have been banned are currently in used in Benue State.

The result generally revealed that all samples of plantain and banana analysed showed some degree of contamination. Pesticide residue levels were generally below their acceptable maximum levels and acceptable daily intake, indicating that plantain and banana in Ogbadibo L. G. A. of Benue State is less contaminated and relatively less harmful.

However, the toxicological importance of pesticide residue data depends not only on the residue content of food but also on the quantity of contaminated food consumed and the length of time over which it is consumed. Thus, continuous consumption of less contaminated food over a long period of

time may lead to a dangerous high concentration of chemical residues in the body.

Since the improvement in crop yield by pesticide application is always concomitant with the occurrence of residues in food stuffs, there is need for regular control on the application of pesticides as this may go a long way towards preventing various environmental and public health hazards.

## Acknowledgements

I wish to acknowledge the contribution of Dr Peter Onuwa (PhD), Felix Sughnen Atsar and Golden Years Limited for analyzing the samples.

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