

Determination of pesticide residue in maize treated with mixed pesticides against stem borer disease in selected farms within Enugu state, Nigeria

Victor Chinedu Onyia^{1*}, Charles C. Chime¹, Prisca I. Udeozo¹, Okwesili C. Lotanna¹, Uka Job Ude¹

¹ Department of Industrial Chemistry, Enugu State University of Science and Technology, Agbani, Enugu State, Nigeria

Correspondence Author: Victor Chinedu Onyia

Received 26 Aug 2022; Accepted 12 Oct 2022; Published 5 Nov 2022

Abstract

The study was conducted to ascertain the level of pesticide residues in four selected maize farms (Agbo, Glory, Igboanugo, and Ogbodo) in Enugu State, Nigeria. The maize samples were identified at the Department of Applied Biology and Biotechnology, Enugu State University of Science and Technology, ESUT, and subsequently ground into powdery form, and then properly labelled and stored in clean hermetic polyethylene bags until required for analysis. At each sampling site, samples were collected and mixed thoroughly before a representative 1 kg sample was bagged for that farm and labelled. Sample analysis for pesticide residue was conducted using gas chromatography coupled with an electron capture detector (GC-ECD). The data analysis was done using IBM SPSS version 20.0 and Ms-Excel 2007. The results obtained revealed the sum of the respective concentrations of the pesticides in the roasted, cooked, and unprocessed fresh maize samples in Agbo (0.9715 ppm, 0.5133 ppm, and 1.1628 ppm respectively), Glory (2.2381 ppm, 0.9147 ppm, and 1.5495 ppm respectively), Igboanugo (0.7869 ppm, 0.3998 ppm, and 1.9247 ppm respectively), and Ogbodo (2.1421 ppm, 1.234 ppm, and 1.0234 ppm respectively). Generally, the pesticide residue concentrations were high, and there was no trend in the roasted, cooked, and unprocessed fresh maize from the studied maize farms. Furthermore, statistical correlation analysis revealed the origin of the pesticide residues while analysis of variance revealed the effects of the pesticides on the roasted, cooked, and unprocessed fresh maize samples at a 0.05% level. Generally, the pesticide levels in the maize samples were significantly higher than the Food and Agriculture Organization and the World Health Organization acceptable limits for pesticides in food crops, which indicated that the consumption of maize from the studied maize farms poses health hazards.

Keywords: maize, pesticides, estimated daily intake (EDI), enugu state

Introduction

The use of pesticides in modern farming practices has significantly contributed to the increased food production in the agricultural industry (Pano-Farias *et al.*, 2017; Jardim *et al.*, 2014) ^[1, 2]. Pesticides are synthetic organic chemicals used for preventing, destroying, or controlling pests. Over the last few decades, the pesticides used were mainly organochlorine pesticides (Salem *et al.*, 2009; Tao *et al.*, 2009) ^[3, 4]. Organochlorine pesticides (OCPs) are persistent in nature, toxic, highly stable, and have a high affinity for lipids (Agboyi *et al.*, 2015; USEPA., 2012) ^[5, 6]. Also, they bio-accumulate in human tissue, and this eventually poses a serious health risk to humans and animals (Gan *et al.*, 2005; Mahugija *et al.*, 2017) ^[7, 8]. Due to their adverse health and environmental effects, their use was banned in the early 1980s, even though they were readily available in local markets and are still used in developing countries.

In Nigeria, lack of law enforcement and the use of pesticides without proper knowledge or training of farmers are major concerns in pesticide monitoring due to the associated health implications ^[8]. This has, in effect, led to the use of any pesticide, even if they are banned or not recommended, provided that they are cheap or very effective in the control of pests (Agboyi *et al.*, 2015; Mahugija *et al.*, 2017; Eze *et al.*, 2020) ^[5, 8, 9]. Unfortunately, their continued use may lead to accumulation in the environment and could be transferred from

soil to plants via root intake.

Organochlorine pesticides cause acute and chronic health effects, such as irritation of the skin and eyes, affecting the nervous system, mimicking hormones, causing reproductive problems, cancer, neurological effects, birth defects, fetal death, and neurodevelopment disorders (Agboyi *et al.*, 2015; Rwetabula J., 2007) ^[5, 10]. Recently, OCPs have been replaced with organophosphorous pesticides, pyrethroids and carbamates, which are biodegradable and less persistent in the environment (Eze *et al.*, 2020; Ngowi *et al.*, 2002; Sarwar, 2015) ^[9, 12, 13].

Maize (*Zea mays*) is an important cereal and a popular staple food that is of great importance in most diets consumed in Nigeria (Morris *et al.*, 1999) ^[14]. It is widely cultivated in the rainforest and savannah zones (Kellogg *et al.*, 2002) ^[15]. Also, it is an economic crop with a huge boon on the Gross Domestic Product (GDP) of Nigeria. However, large-scale maize production has been greeted with numerous hampering challenges such as diseases and pests' interference. Most of its varieties are highly susceptible to maize rust, leaf blight, maize streak, maize mottle, curvularia leaf spot, downy mildew disease, stalk and ear rots (Iken *et al.*, 2004) ^[16]. In recent times, there has been an exponential increase in the demand for maize, which has led to the need for continuous availability of its varieties. Hence, the need for pesticide uses in order to manage pests and ensure food security. Furthermore, organochlorine

pesticides (OCPs) have been extensively used in maize production, although there is no routine monitoring of this food crop to check their residue level.

A literature review suggests that many studies on the adverse effects of pesticide residues have been carried out by Nigerian researchers (Salem *et al.*, 2009; Tao *et al.*, 2009; Agboyi., 2015; Mahugija *et al.*, 2017; Hussain *et al.*, 2010; Eze *et al.*, 2021; Khan *et al.*, 2020; Fang *et al.*, 2015) [3, 4, 5, 8, 17, 18, 19, 20]. There are only a few documented studies on organochlorine pesticides in food crops (Sarwar M., 2015; Jallow *et al.*, 2017) [13, 21]. However, due to the paucity of data on the quantification of organochlorine pesticide residue in maize crops, this study was conducted to fill the knowledge gap.

Furthermore, statistical methods such as correlation analysis (CA) and analysis of variance (ANOVA) have been widely used to investigate pesticide residue accumulation and origin in agricultural soils (Akhtar *et al.*, 2018; Onwukeme, 2021; Qishlaqi., 2007) [22, 23, 24]. In this study, correlation analysis was used to confirm the degree of relationship between the pesticides residue in maize crops cultivated on the studied

farms, whereas analysis of variance (ANOVA) was used to determine the significant difference in pesticides residue concentrations between the studied farms. This research would provide relevant data for establishing a monitoring programme for pesticides such as lindane, DDT, endrine, and atrazine in maize grown in Enugu State, Nigeria. Additionally, it would enable authorities to check for compliance with Nigeria's legislation on these chemicals and also provide relevant data to quantify the amount of these chemicals consumed in our diet.

Materials and Methods

Description of the study area

Experimental farms were located in some selected local government areas in Enugu State, which is located in the southeastern region of Nigeria (Figure 1). Maize samples were obtained from four popular maize farms located in LGA, Enugu State: Agbo maize farm, Opi Nsukka; Glory maize farm, Nkwubor road; Igboanugo maize farm, Ugwuoba; and Ogbodo maize farm, Akpuoga Nike.

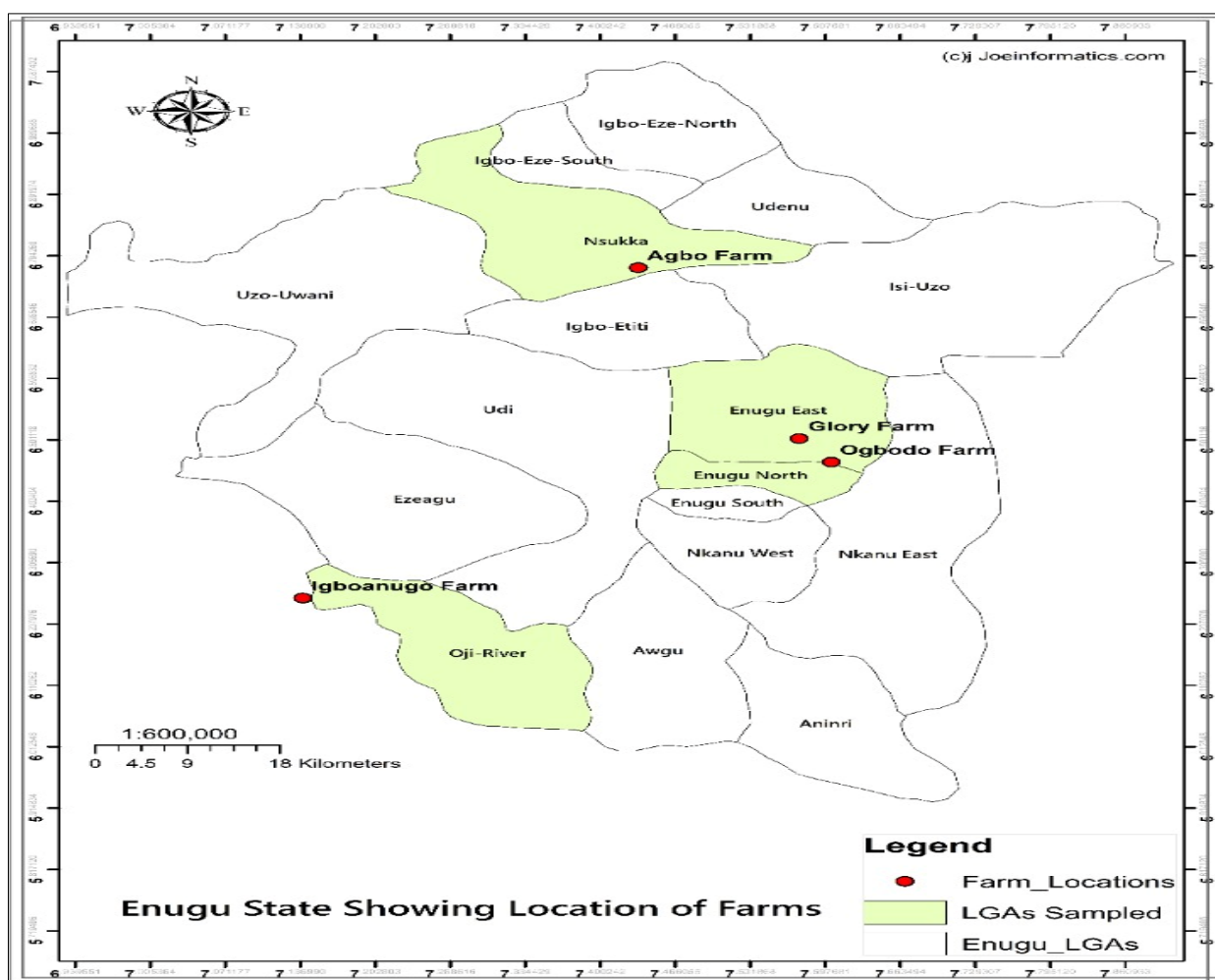


Fig 1: Map of Enugu State showing the maize farm locations

Sample Collection and Preparation

Maize samples were taken from all selected locations where pesticides were applied in the farms. The selected farms were: Agbo maize farm, Opi Nsukka; Glory maize farm, Nkwubor; Igboanugo maize farm, Ugwuoba; and Ogbodo maize farm, Akpuoga Nike. Samples were bagged in hermetic polyethylene bags, labeled appropriately, and subsequently identified at the

Department of Applied Biology and Biotechnology, Enugu State University of Science and Technology, ESUT. Samples were ground into powdery form, and then properly labelled and stored in clean, separate hermetic polyethylene bags until required for analysis. At each sampling site, samples were collected and mixed thoroughly before a representative 1 kg sample was bagged for that farm and labeled.

Sample Extraction and Clean-up

In this study, the extraction of the pesticide residues from the maize samples was done by the modified version of the quick, easy, cheap, effective, rugged, and safe (QuEChERS) method. Composite samples were collected from each of the maize farms. 20 g was homogenized and pulverized to powdered form and weighed using a Mettler Toledo PG 1003-S mass balance into a 50 mL centrifuge tube. After that, 10 ml of cold, deionized water and acetonitrile were added. The mixture was then vortexed for one minute using a Thermolyne max mix plus. A mixture of QuEChERS salts was then added [4 g of anhydrous MgSO₄ plus 1 g NaCl, 1 g Trisodium Citrate Dihydrate (TSCD), and 0.5 g of disodium hydrogen citrate sesquihydrate (DHS)]. The mixture was vortexed for a further one minute and then centrifuged for five minutes at 3000 rpm [25]. Furthermore, the sample extracts were then purified using Dispersive Solid Phase Extraction (SPE) method. An aliquot (6 ml) of the extracts was transferred into a polypropylene (PP) centrifuge tube containing 150 mg of primary and secondary amine (PSA) and 900 mg of MgSO₄. The mixture was vortexed for 1 minute and centrifuged for 5 minutes at 3000 rpm. Then, 4 ml of the supernatant (clean extract) was transferred into a volumetric flask. To adjust the pH, 40 µL of 5% formic acid in acetonitrile (v/v) was added. The filtrate was then concentrated to dryness at about 35 °C in a rotary evaporator. The dry concentrate was then redissolved in ethyl acetate (1 ml) plus 20 L of 1% polyethylene glycol, then transferred into a 2 ml standard opening auto sampler vials for quantification by GC-ECD for organochlorines (Pesticide Residue Committee Annual Report, 2006).

Sample analysis using GC-ECD

The main analytical tools used for the determination of pesticide residues in food or environmental samples are gas chromatography with electron capture (GC-ECD), mass spectrometer (GC-MS), or pulsed flame. In this work, maize samples were analyzed for pesticides on a gas chromatograph (varian CP-3800 GC-ECD with Combi PAL autosampler). It was fitted with a Varian analytical column 30 m long 10 µm + 10 m EZ guard. The column has a 0.25 mm id fused silica capillary coated with VF-5ms (0.25 micrometre film). Extracts of samples were interspersed with analytical standards of pesticides placed on the autosampler with standards at the start, between every 10 samples and the last of the GC sample run. Pesticides residue components were identified by comparing their retention times with those of the standards.

Quality Control/Quality Assurance

Quality control and quality assurance as prescribed by the CODEX Alimentarius Committee were incorporated into the analytical scheme. Quality assurance measures applied in the laboratory include rigorous contamination control procedures (strict washing and cleaning procedures), monitoring of blank levels of solvents, equipment, and other materials, and including blanks and duplicates in the analysis and re-calibration standards run frequently to check the integrity of the calibration curve. An aliquot (50 ml) of each solvent was concentrated to 1 ml and analyzed to check the contamination from the reagents (FAO/WHO., 2002) [27]. The quality of analytical methods was assessed by recovery experiments with maize. Maize matrices that had been assayed and were known

to have no detectable levels of pesticides were used for recovery tests. The validation and chemical recoveries were estimated by spiking pesticides-free maize blanks with pesticides standards overnight before extraction. Samples were extracted and cleaned-up as described above and subsequently analysed by GC-ECD. The percentage recovery was calculated using the formula:

$$\text{Recovery (\%)} = \frac{\text{Conc. of pesticide residue recovered from spiked sample}}{\text{Conc. of pesticide added to sample}} \times 100$$

Data analysis

The data analysis in this study was done using IBM SPSS (statistical software for sciences) version 20.0 and Ms-Excel 2007. At a 5% level of significance, Pearson's correlation matrix and analysis of variance (ANOVA) were used to establish a relationship between the pesticides concentrations in the studied maize farms.

Assessment of health risk

In risk estimation, various international organizations have successively established a series of standards and protocols for evaluating the potential health risks from environmental pollutants in maize crops (USEPA., 2012) [6]. A straightforward risk assessment involves comparing results with the levels set by laws and guidelines. However, this comparison is made without considering different eating habits and consumption rates. Thus, in this study, estimated daily intakes (EDI) of the pesticides were used for a health risk assessment and were calculated using Equation 2.

$$\text{EDI} = \frac{\text{mean concentration} \times \text{consumption rate} \times \text{exposure frequency} \times \text{exposure duration}}{\text{BW} \times \text{AT}}$$

In the above equations, C is the mean concentration of the pesticides in maize samples (ppm), CR is the consumption rate of the maize (0.15 kg day⁻¹), EF is the frequency of exposure (365 days/year), ED is the duration of exposure (365 × 30 years), BW is the average adult bodyweight (75 kg), average time (AT = 365 × 70) derived from the Exposure Factors Handbook (Fjeld *et al.*, 2007) [28].

Results and Discussions

Concentrations of the pesticides

The concentration of the pesticides in maize from Glory maize farm, Ogbodo maize farm, Igboanugo maize farm, and Agbo maize farm are presented in Tables 1, 2, 3, and 4. In Glory maize farm, fourteen compounds (2,4-dichloro, DichloroBiphenyl, HCB, Endosulfan, PP'-DDD, Carbofuran, Dichlorvos, Isopropylamin, Aldrin, Profenofos, DDVP, Heptachlor, Glyphosphate, t-nonachlor) were detected in the roasted, cooked, and unprocessed fresh maize samples. Also, the mean concentration was 1.567433ppm and sum of the concentrations of the pesticides in the roasted, cooked, and unprocessed fresh maize samples are 2.2381 ppm, 0.9147 ppm, and 1.5495 ppm respectively. It can be seen that the concentration of pesticides was highest in the roasted maize samples (2.2381 ppm), while the cooked maize samples

(0.9147 ppm) recorded the least concentration. In Ogbodo maize farm, eighteen compounds (2-4-dichloro, HCB, Endosulfan, Aldrin, P'P'-DDD, g-chlordane, Profenofos, Carbofuran, DDVP, Dichlovos, Heptachlor, t-nonachlor, Isopropylamin, DichloroBiphnyl, Lindane, Profenofos, Dichlorvos, Lindane) were detected in the roasted, cooked and unprocessed fresh maize samples. In addition, the mean concentration was 1.466833 and the sum of the concentrations of the pesticides in the roasted, cooked, and unprocessed fresh maize samples was 2.1421 ppm, 1.234 ppm, and 1.0234 ppm respectively. Again, the concentration of pesticide was highest in the roasted maize samples (2.1421 ppm) while the least concentration was recorded in the unprocessed fresh maize samples (1.0234 ppm). In the Igboanugo maize farm, twenty-three compounds (Isopropylamin, 4-4-bipyridinium dichloride, HCB, Endosulfan, Aldrin, P'P'-DDE, P'P'-DDD, g-chlordane, Profenofos, Carbofuran, DDVP, Dichlovos, Heptachlor, t-nonachlor, Isopropylamin, DichloroBiphnyl, Lindane, Profenofos, 2-4-dichloro, Lindane, Glyphosphate, Emamectine, DDT) were detected in the roasted, cooked and unprocessed fresh maize samples. The mean concentration was 1.037133ppm and sum of the concentrations of the pesticides in the roasted, cooked, and unprocessed fresh maize samples are 0.7869 ppm, 0.3998 ppm, and 1.9247 ppm respectively. It can be seen that the concentration of pesticides was highest in the unprocessed fresh maize sample (1.9247 ppm), while the cooked maize sample (0.3998 ppm) recorded the least concentration. In Agbo maize farm, twenty-three compounds (Isopropylamin, 4-4-bipyridinium dichloride, HCB, Endosulfan, Aldrin, P'P'-DDE, P'P'-DDD, g-chlordane, Profenofos, Carbofuran, DDVP, Dichlovos, Heptachlor, t-nonachlor, Isopropylamin, DichloroBiphnyl, Lindane, Profenofos, 2-4-dichloro, Lindane, Glyphosphate, Aldrin,

Emamectine). Furthermore, the mean concentration was 1.551067ppm and sum of the concentrations of the pesticides in the roasted, cooked and unprocessed fresh maize samples was 0.9715 ppm, 0.5133 ppm, and 1.1628 ppm. Concentration of pesticide was highest in the unprocessed maize sample (1.1628 ppm), while the least concentration was recorded in the cooked maize sample (0.5133 ppm). According to Kaushik *et al.*, the concentrations of pesticides, e.g., DDT, cypermethrin, and dichlorvos, were reduced by 25 % to 69.7 % during cooking. Muthukumar *et al.* found that cooking of endosulfan spiked meat resulted in a 55.93 to 64.59 % reduction in α -endosulfan and β -endosulfan. These studies also revealed that processes that occur during cooking can affect the pesticide residues due to volatilisation, hydrolysis, thermal breakdown, and other chemical degradations. Although it has been reported in the literature that cooking tends to remove about 20 – 25% of organochlorine, and 35 – 60% of the organophosphate pesticide residues (Onwuka *et al.*, 2017) [31], pesticide residues often migrates into the cooking water. The extent of this migration is driven by the solubility of the substance, the liquid/food ratio, the duration of the heating step, the strength of adhesion of residues to plant structure (conjugates), the change in food structure during cooking, and the temperature applied during the process (Mwanja *et al.*, 2017) [32]. The detection of high concentrations and increased numbers of pesticides may be due to the differences in the pesticide type, concentration, and accumulation, which subsequently result in contamination of the soil and environment around the maize farms (WHO., 2010). The impact of pesticides can be minimized by taking certain measures such as promoting organic farming, exploiting natural and bio-pesticides and proper implementation and amendment of pesticide-related laws.

Table 1: Glory maize farm

Pesticides	Roasted	Cooked	Unprocessed fresh maize	Mean	Minimum	Maximum
2-4-dichloro	0.119	0.1115	0.0304	0.086967	0.0304	0.119
DichloroBiphnyl	0.0007	0.0017	ND	0.0008	ND	0.0017
HCB	0.1998	0.1742	ND	0.124667	ND	0.1998
Endosulfan	0.1718	0.0771	0.1791	0.142667	0.0771	0.1791
P'P'-DDD	0.4521	0.1303	0.4519	0.344767	0.1303	0.4521
Carbofuran	0.0932	0.1504	ND	0.0812	ND	0.1504
Dichlovos	0.0484	0.1362	ND	0.061533	ND	0.1362
Isopropylamin	ND	0.005	ND	0.001667	ND	0.005
Aldrin	0.1343	0.1283	ND	0.087533	ND	0.1343
Profenofos	0.3017	ND	0.2067	0.169467	ND	0.3017
DDVP	0.2008	ND	0.2151	0.138633	ND	0.2151
Heptachlor	0.3277	ND	0.4663	0.264667	ND	0.4663
Glyphosphate	0.0492	ND	ND	0.0164	ND	0.0492
t-nonachlor	0.1394	ND	ND	0.046467	ND	0.1394
Sum of Pesticides	2.2381	0.9147	1.5495	1.567433	0.9147	2.2381

ND- Not Detected (Below Detection Limit)

Table 2: Ogbodo maize farm

Pesticide	Roasted	Cooked	Unprocessed fresh maize	Mean	Minimum	Maximum
2-4-dichloro	0.0353	0.0006	0.0021	0.012667	0.0006	0.0353
HCB	0.4681	0.1519	0.1294	0.2498	0.1294	0.4681
Endosulfan	0.1127	0.1694	0.1723	0.151467	0.1127	0.1723
Aldrin	0.2012	ND	ND	0.067067	ND	0.2012
P'P'-DDD	0.0002	ND	ND	6.67E-05	ND	0.0002
g-chlordane	0.0427	0.0766	0.0404	0.053233	0.0404	0.0766

Profenofos	0.2686	ND	ND	0.089533	ND	0.2686
Carbofuran	0.0746	0.2109	0.0378	0.107767	0.0378	0.2109
DDVP	0.4639	ND	ND	0.154633	ND	0.4639
Dichlovos	0.1261	ND	ND	0.042033	ND	0.1261
Heptachlor	0.1764	ND	ND	0.0588	ND	0.1764
t-nonachlor	0.1723	0.117	0.09	0.126433	0.09	0.1723
Isopropylamin	ND	0.0005	0.0008	0.000433	ND	0.0005
DichloroBiphnyl	ND	0.0973	0.0979	0.065067	ND	0.0979
Lindane	ND	0.26	ND	0.086667	ND	0.26
Profenofos	ND	0.1019	0.1457	0.082533	ND	0.1457
Dichlorvos	ND	0.0479	0.0477	0.031867	ND	0.0479
Lindane	ND	ND	0.2603	0.086767	ND	0.2603
Sum of Pesticides	2.1421	1.234	1.0234	1.466833	1.0234	2.1421

ND- Not Detected (Below Detection Limit)

Table 3: Igboanugo maize farm

Pesticide	Roasted	Cooked	Unprocessed fresh maize	Mean	Minimum	Maximum
Isopropylamin	ND	ND	0.7859	0.261967	ND	0.7859
4-4-bipyridinium dichloride	0.1862	ND	ND	0.062067	ND	0.1862
HCB	0.0013	0.0009	ND	0.000733	ND	0.0013
Endosulfan	0.0015	0.0013	0.3886	0.130467	0.0013	0.3886
Aldrin	ND	0.0001	ND	3.33E-05	ND	0.0001
P'P'-DDE	0.1362	0.1236	ND	0.0866	ND	0.1362
P'P'-DDD	0.2313	0.1545	ND	0.1286	ND	0.2313
g-chlordane	ND	ND	0.2721	0.0907	ND	0.2721
Profenofos	ND	ND	ND	ND	ND	ND
Carbofuran	ND	ND	0.0651	0.0217	ND	0.0651
DDVP	ND	ND	ND	ND	ND	ND
Dichlovos	0.1822	0.0952	0.0483	0.108567	0.0483	0.1822
Heptachlor	ND	ND	0.2234	0.074467	ND	0.2234
t-nonachlor	ND	ND	ND	ND	ND	ND
Isopropylamin	0.001	ND	ND	0.000333	ND	0.001
DichloroBiphnyl	ND	ND	ND	ND	ND	ND
Lindane	ND	ND	ND	ND	ND	ND
Profenofos	ND	ND	0.0511	0.017033	ND	0.0511
2-4 dichloro	ND	ND	ND	ND	ND	ND
Lindane	0.0061	ND	0.0832	0.029767	ND	0.0832
Glyphosphate	0.0078	0.0077	ND	0.005167	ND	0.0078
Emamectine	0.0333	0.0165	ND	0.0166	ND	0.0333
DDT	ND	ND	0.007	0.002333	ND	0.007
Sum of Pesticides	0.7869	0.3998	1.9247	1.037133	0.3998	1.9247

ND- Not Detected (Below Detection Limit)

Table 4: Agbo maize farm

Pesticide	Roasted	Cooked	Unprocessed fresh maize	Mean	Minimum	Maximum
Isopropylamin	ND	ND	ND	ND	ND	ND
4-4-bipyridinium dichloride	0.0644	0.0645	ND	0.042967	ND	0.0645
HCB	0.5074	0.0451	0.0012	0.184567	0.0012	0.5074
Endosulfan	0.1047	0.3015	ND	0.1354	ND	0.3015
Aldrin	0.2185	ND	0.015	0.077833	ND	0.2185
P'P'-DDE	ND	ND	0.178	0.059333	ND	0.178
P'P'-DDD	ND	ND	ND	ND	ND	ND
g-chlordane	ND	ND	0.0003	0.0001	ND	0.0003
Profenofos	ND	ND	ND	ND	ND	ND
Carbofuran	ND	ND	ND	ND	ND	ND
DDVP	ND	ND	ND	ND	ND	ND
Dichlovos	0.0465	0.0466	0.0263	0.0398	0.0263	0.0466
Heptachlor	ND	ND	0.5608	0.186933	ND	0.5608
t-nonachlor	0.0003	0.0003	0.0309	0.0105	0.0003	0.0309
Isopropylamin	ND	0.0162	0.0026	0.006267	ND	0.0162
DichloroBiphnyl	ND	ND	ND	ND	ND	ND
Lindane	ND	ND	ND	ND	ND	ND
Profenofos	ND	ND	ND	ND	ND	ND

2-4 dichloro	ND	ND	ND	ND	ND	ND
Lindane	0.0297	0.0263	0.3082	0.1214	0.0263	0.3082
Glyphosphate	ND	0.0128	ND	0.004267	ND	0.0128
Aldrin	ND	ND	ND	ND	ND	ND
Emamectine	ND	ND	0.0395	0.013167	ND	0.0395
Sum of Pesticides	0.9715	2.5189	1.1628	1.551067	0.9715	2.5189

ND- Not Detected (Below Detection Limit)

Correlation analysis

In this study, the degree of linear association between the pesticides in the studied maize farms was measured by the simple correlation coefficient (r), and the values are presented in Tables 5, 6, 7 and 8. The results of the correlation analysis revealed that a strong and positive correlation (r = 0.871717) was observed between roasted and unprocessed maize (Table 5); a weak and positive correlation (r = 0.244153) was observed between cooked and unprocessed maize (Table 6); a strong and positive correlation (r = 0.823347) was observed between roasted and cooked maize samples (Table 7); a strong and positive correlation (r = 0.782367) was observed between roasted and cooked maize samples (Table 8). It is of importance to note that the strong and positive correlation between roasted and unprocessed maize samples (Table 5), roasted and cooked maize samples (Table 7), and roasted and cooked maize samples (Table 8) indicates that a similar pesticide was applied on the maize farms. Hence, the pesticides are of the same origin (Sarwar, 2015; Moris *et al.*, 1999; Hussain, Siddique., 2010; Khan *et al.*, 2020) ^[13, 17, 19]. A weak and positive correlation between cooked and unprocessed maize (Table 6) indicates that their relationship is not strong even though they are of the same origin.

Table 5: Correlation matrix for Glory maize farm

	Roasted	Cooked	Unprocessed
Roasted	1		
Cooked	0.100144	1	
Unprocessed	0.871717	-0.16352	1

Table 6: Correlation matrix for Ogbodo maize farm

	Roasted	Cooked	Unprocessed
Roasted	1		
Cooked	-0.08044	1	
Unprocessed	-0.11026	0.244153	1

Table 7: Correlation matrix for Igboanugo maize farm

	Roasted	Cooked	Unprocessed
Roasted	1		
Cooked	0.823347	1	
Unprocessed	-0.20383	-0.16078	1

Table 8: Correlation matrix for Agbo maize farm

	Roasted	Cooked	Unprocessed
Roasted	1		
Cooked	0.782367	1	
Unprocessed	-0.10558	-0.00322	1

Analysis of variance (ANOVA)

The results of the two-way analysis of variance revealed that the observed p-values are greater than 0.05, which implies that the effects of the pesticides on the roasted, cooked, and

unprocessed fresh maize samples are independent of the pesticides.

Health risk estimation

The EDI was calculated and compared with the acceptable daily intake (ADI) recommended by the Food and Agriculture Organization and the World Health Organization (FAO/WHO) Joint Meeting on Pesticide Residues 2(FAO/WHO., 2002; WHO., 2010) ^[27, 33]. It was noted that the levels of the pesticides in the maize samples were significantly higher than the Food and Agriculture Organization and the World Health Organization acceptable limits for pesticides in food crops (maize), thus indicating that the consumption of maize from the studied maize farms poses health hazards. Similarly, recent studies have reported the use of pesticides in agriculture to control destructive pests and hence increase food supply. Consequently, this has inadvertently led to the accumulation of their residues in food crops (maize) and the environment. Worthy of note is that pesticides by nature are poisonous and exposure of humans to their residues through inhalation, and ingestion of food crops (maize) cultivated on farms where pesticides are used for pesticide control can cause adverse health effects which include neurotoxicity, carcinogenicity, and acute effects such as nausea, diarrhea, rashes, stinging eyes, blindness, dizziness and blisters (Khan *et al.*, 2020; Ogah *et al.*, 2011) ^[19, 34]. Therefore, the evaluation of pesticide residues in food is of public health importance and would help to ensure that their levels are kept within safe limits. Based on the findings of this study, there is a need for more stringent monitoring of the use of pesticides in agriculture and food storage in developing countries such as Nigeria.

Conclusion

Pesticide residues were found in high concentrations in all the maize samples from the studied maize farms in Enugu State, Nigeria. The highest concentration of pesticides was observed in Glory maize farm, while the least concentration was observed in Igboanugo maize farm. In addition, the origin and effects of the pesticide residues on the roasted, cooked, and unprocessed fresh maize samples have been ascertained. It is worth noting that the pesticide concentrations exceeded the Food and Agriculture Organization and the World Health Organization’s acceptable limit for pesticides in food crops. This implies that the consumption of maize from the studied maize farms poses health hazards. Based on the findings of this study, there is a need for more stringent monitoring on the use of pesticides in agriculture by Enugu State Government and food storage in developing countries such as Nigeria.

References

1. Pano-Farias NS, Ceballos-Magaña SG, Muñiz-Valencia R, Gonzalez J. “Validation and assessment of matrix effect and uncertainty of a gas chromatography coupled to mass

- spectrometry method for pesticides in papaya and avocado samples," *Journal of food and drug analysis*, 2017; 25(3):501-509.
2. Jardim ANO, Mello DC, Goes FCS, Frota Junior EF, Caldas ED. "Pesticide residues in cashew apple, guava, kaki and peach: GC- μ ECD, GC-FPD and LC-MS/MS multiresidue method validation, analysis and cumulative acute risk assessment," *Food Chemistry*, 2014; 164(1):195-204.
 3. Salem N, Ahmad R, Estaitieh H. Organochlorine pesticide residues in dairy products in Jordan. *Chemosphere*, 2009; 77(5):673-678.
 4. Tao S, Liu WX, Li XQ, Zhou DX, Li X, Yang YF, *et al.* Organochlorine pesticide residuals in chickens and eggs at a poultry farm in Beijing, China. *Environ Pollut*, 2009; 157(2):497-502.
 5. Agboyi LK, Djade KM, Ahadji-Dabla KM, Ketoh GK, Nuto Y, Glitho IA. Vegetable production in Togo and potential impact of pesticide use practices on the environment. *Int. J. Biol. Chem. Sci.*, 2015; 9(2):723-736. DOI: <http://dx.doi.org/10.4314/ijbcs.v9i2.13>.
 6. USEPA. Human health risk assessment; risk based screening table, 2012. http://www.epa.gov/reg3hwmd/risk/human/pdf/NOV_2012_FISH.pdf. Accessed Sept 2013
 7. Gan J, Lee SJ, Liu WP, Haver DL, Kabashima JN. "Distribution and persistence of pyrethroids in runoff sediments," *Journal of Environmental Quality*, 2005; 34(3):836-841.
 8. Mahugija JAM, Lutamyo N, Mmochi AJ. Levels and distribution of pesticide residues in soil and sediments in eastern, lake tanganyika environs (2017). *Int. J. Biol. Chem. Sci.*, 2017; 11(5):2537-2547.
 9. Eze VC, Onwukeme V, Enyoh CE. Pollution status, ecological and human health risks of heavy metals in soil from some selected active dumpsites in Southeastern, Nigeria using energy dispersive X-ray spectrometer. *Int J Environ Anal Chem*, 2020, 1-22.
 10. Rwetabula J. Modelling the Fate and Transport of Organic Micro-Pollutants and Phosphate in the Simiyu River and Speke Gulf (Lake Victoria), Tanzania. VUB-Hydrologie Vrije University: Brussel, 2007.
 11. Aktar W, Sengupta D, Chowdhury A. Impact of pesticides use in agriculture: their benefits and hazards. *Interdisciplinary Toxicology*, 2009; 2(1):1-12. doi: 10.2478/v10102-009-0001-7.
 12. Ngowi AVF. Health Impact of Exposure to Pesticides in Agriculture in Tanzania. Academic dissertation, School of Health, University of Tampere: Finland, 2002.
 13. Sarwar M. "The dangers of pesticides associated with public health and preventing of the risks," *International Journal of Bioinformatics and Biomedical Engineering*, 2015; 1(2):130-136.
 14. Morris ML, Tripp R, Dankyi AA. Adoption and Impacts of Improved Maize Production Technology: A Case Study of the Ghana Grains Development Project. Economics Program Paper 99-01. Mexico, D.F.: CIMMYT, 1999.
 15. Kellogg RL, Nehring RF, Grube A, Goss DW, Plotkin S. "Environmental Indicators of Pesticide Leaching and Runoff from Farm Fields". In Ball VE, Norton GW (eds). *Agricultural Productivity. Studies in Productivity and Efficiency*. 2. Boston: Springer. pp. 213–56. ISBN 97814613-52709. Archived from the original on, 2002.
 16. Iken JE, Amusa NA. "Maize research and production in Nigeria" *African Journal of Biotechnology*, 2004; 3:301-307.
 17. Hussain Z, Siddique S. Determination of Pesticides in Fruits and Vegetables using Acetonitrile Extraction and GC/MS Technique. *Journal of Scientific Research*, 2010; 2:19-29.
 18. Eze VC, Nwabudike AR, Duru CE, Isiuku BO, Ibe FC, Ogbuagu JO, *et al.* Human health risk assessment of the levels of dioxinlike polychlorinated biphenyls (PCBs) in soils from mechanic workshops within Nekede mechanic village, Imo State, Nigeria, *International Journal of Environmental Analytical Chemistry*, 2021. DOI: 10.1080/03067319.2021.1974424
 19. Khan N, Ghazala Y, Tahreem H, Tariq M. Assessment of Health Risk due to Pesticide Residues in Fruits, Vegetables, Soil, and Water, *Journal of Chemistry*, 2020; 2020:5497952. <https://doi.org/10.1155/2020/5497952>
 20. Fang Y, Zhiqiang N, Yanmei Y, Qingqi D, Feng L, Jie H. Human health risk assessment of pesticide residues in market-sold vegetables and fish in a northern metropolis of China, *Environ Sci Pollut Res*, 2015; 22:6135-6143. DOI 10.1007/s11356-014-3822-7
 21. Jallow MF, Awadh DG, Albaho MS, Devi VY, Ahmad N. Monitoring of Pesticide Residues in Commonly Used Fruits and Vegetables in Kuwait. *International Journal of Environmental Research and Public Health*, 2017; 14(8):833.
 22. Akhtar S, Ghazala Y, Almas H, Zainab AS. Determination of Pesticide Residues in Selected Vegetables and Fruits from a Local Market of Lahore, Pakistan, *Current World Environment*, ISSN: 0973-4929, 2018; 13(2):242-250. Doi: <http://dx.doi.org/10.12944/CWE.13.2.09>
 23. Onwukeme VI, Eze VC. Identification of Heavy Metals Source within Selected Active Dumpsites in Southeastern Nigeria, *Environmental Analysis Health and Toxicology*, 2021. <https://doi.org/10.5620/eaht.2021008>
 24. Qishlaqi A, Moore F. Statistical Analysis of Accumulation and Sources of Heavy Metals Occurrence in Agricultural Soils of Khoshk River Banks, Shiraz, Iran. *Am Eurasian J Agric Environ Sci*, 2007; 2(5):565-573.
 25. Sadiq MS, Yakassai MT, Ahmad MM, Lakpene TY, Abubakar M. Profitability and Production Efficiency of Small-Scale Maize Production in Niger State, Nigeria. *IOSR Journal of Applied Physics (IOSR-JAP)*, 2013; 3(4):19-23.
 26. Pesticide Residue Committee (PRC) Annual Report, 2006.
 27. FAO/WHO. Pesticide residues in food 2002. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group. FAO Plant Production and Protection Paper, 2002, 156-172.
 28. Fjeld RA, Eisenberg NA, Compton KL. Dose-response and risk characterization. In: *Quantitative Environmental Risk Analysis for Human Health*. John Wiley & Sons, Inc., Hoboken, New Jersey, 2007, 245-282. Available at. <https://is.muni.cz/auth/el/1431/jaro2015/C2003/um/55931844/Fjeld2007.pdf>

29. Kaushik G, Satya S, Naik SN. Food processing a tool to pesticide residue dissipation-A review. *Food Research International*, 2009; 42:26-40.
30. Muthukumar M, Sudhakar Reddy K, Narendra Reddy C, Kondal Reddy K, Gopala Reddy A, Jagdishwar Reddy D, *et al.* Detection of cyclodiene pesticide residues in buffalo meat and effect of cooking on residual level of endosulfan. *Journal of Food Science and Technology*, 2009; 47:325-329.
31. Onuwa PO, Ishaq SE, Adams UI, Sha'Ato R. Determination of Pesticide Residues in Edible Crops and Soil from University of Agriculture Makurdi Farm Nigeria (2017). *Asian Journal of Physical and Chemical Sciences*, 2017; 3(3):1-17. Article no. AJOPACS.35001, ISSN: 2456-7779.
32. Mwanja M, Choolwe J, Allan RM, Nosiku SM. Assessment of pesticide residue levels among locally produced fruits and vegetables in Monze district, Zambia, *International Journal of Food Contamination*, 2017; 4:11. DOI 10.1186/s40550-017-0056-8
33. WHO (World Health Organization). Inventory of IPCS and other WHO Pesticide Evaluations and Summary of Toxicological Evaluations Performed by the Joint Meeting on Pesticide Residues (JMPRs) through, 2010.
34. Ogah C, Coker HAB, Adepoju-Bello A. Pesticide residue levels in maize samples from markets in Lagos State, Nigeria. *Nigerian Quarterly Journal of Hospital Medicine*, 2011; 21(2):169-74.