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Assessment Of Organochlorine Pesticide Residues On Staple Grains Sold In Selected Markets In Imo State

Nwogu Stanley [1], Okorondu Justin N. [1,2], Diagi Bridget[1,2]

1. Department of Environmental Studies, Centre of Excellence in Sustainable Procurement, Environmental, and Social Standards, Federal University of Technology, Owerri, Imo State, Nigeria.

2. Department of Environmental Management, School of Environmental Sciences, Federal University of Technology, Owerri, Imo State, Nigeria.

Corresponding Author: edeoli@yahoo.com

ABSTRACT

This study focused on Human Health Risks Assessment of Organochlorine Pesticide Residues on Staple Grains Sold in Selected Markets in Imo State. The main objectives were to identify and quantify the presence of organochlorine pesticide residues in beans and maize sold in Imo State markets, assess the potential health risks associated with the consumption of these contaminated agricultural products and provide recommendations for reducing human exposure to OCPs through safer agricultural practices and regulatory measures. The study employed a cross-sectional research design to assess the presence and levels of organochlorine pesticide (OCP) residues in beans and maize sold in Imo State markets and to evaluate the associated human health risks. The study involved both fieldwork (sample collection) and laboratory analysis (pesticide residue detection and quantification). The GC-MS results indicated some significant levels of various OCP residues, including DDT, aldrin, dieldrin, endosulfan, and heptachlor, in beans and maize samples. This indicates OCP contamination in agricultural produce sold in Imo State markets. Prolonged exposure to these pesticides, even at low levels, can lead to serious health issues, including cancer, endocrine disruption, and neurological disorders. The presence of OCP residues in commonly consumed food items such as beans and maize pose a significant threat to food safety and public health. The findings underscore the need for urgent action to address this issue through improved regulatory enforcement, monitoring, and public awareness campaigns.

Keywords: Assessment, Organochlorine, pesticides, staple grains, selected markets

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1. INTRODUCTION

Organochlorine pesticides (OCPs) are a class of chemicals used to eradicate and prevent pests and diseases that affect agricultural production in the field or at retail outlets (Jayaraj *et al*., 2016; Taiwo, 2019; Tagne- Fotso *et al*., 2023). Organochlorines represent the first important class of insecticides developed by the burgeoning chemical industry in the first half of the 20th century (Rincón-Rubio *et al*., 2024, Van den, 2023). Some organochlorine compounds have been banned in the past several years but still define a substantial share of the insecticides market (Van den, 2023). Chemical insecticides, including OCs, are neurotoxicants, as they act by poisoning the nervous system of the target organisms, including insects (Wahab *et al*., 2016; Shah, 2020; Namiki *et al*., 2020; Ruan *et al*., 2023). Organochlorine insecticides are known to be toxic to several insect pests, mammals, including humans (Jayaraj *et al*., 2015; Lotti *et al*., 2015; Martín-Durán *et al*., 2021; Thakur *et al*., 2023).The National Agency for Food and Drug Administration and Control (NAFDAC), a Nigerian organization that regulates the importation, exportation, manufacture, sale, and use of food, drugs, cosmetics, and related products, conducted an investigation. Through laboratory analyses, NAFDAC determined that the food poisoning incident was caused by consuming beans exposed to pesticides, which directly led to fatalities (Oyeyiola *et al*., 2017). There have been several reports on the danger and health hazards of insecticides usage in Nigeria (Amusat *et al*., 2019). Most of which were a result of the indiscriminate and inappropriate use of these chemicals. These have led to food poisoning, several health cases and even some have led to death (Mohammad *et al*., 2018, Ogwo, 2021). Organochlorine pesticide residues have been reported in different food commodities in Nigeria (Sosan *et al*., 2020, Mohammad *et al*., 2018, Oshatunberu, 2023, Idowu *et al*., 2022). Also, a study carried out in Nigeria showed contamination levels of DDT, endrin, dieldrin, and lindane in beans (P. vulgaris) collected from both field and storage facilities (Fadina *et al*., 2021). Adefemi *et al*. (2018) reported multi-residue OCs pesticide and health risk assessment in edible vegetables, while Akande *et al*., (2020) also evaluated organophosphate insecticide in post–harvest cowpea in Gwagwalada, Abuja. While Idowu *et al*. (2022) examined OCs residue in cocoa pods and beans. Similarly, Fagbohun *et al*. (2023b) and Fagbohun *et al*. (2024) assessed glyphosate residue in retailed cowpea and maize grains in FCT, Abuja markets, respectively. The persistence of OCPs in the environment and their ability to bioaccumulate further exacerbate the problem, as they can remain in the food chain for extended periods, leading to chronic exposure even at low levels (Gonzalez *et al*., 2012). Despite existing regulations and monitoring efforts by agencies such as the National Agency for Food and Drug Administration and Control (NAFDAC), the effectiveness of these measures in controlling OCP residues in local markets remains questionable (NAFDAC, 2012). This study addresses the critical need to understand the extent of OCP contamination in beans and maize in Imo State markets and the potential health risks to consumers. By doing so, it aims to contribute to the development of strategies to mitigate these risks and ensure food safety.

2. MATERIALS AND METHODS

2.1. Study Area

The study was conducted in Imo State, Nigeria. It is located in the Southeast geopolitical zone of Nigeria. It lies between latitude 4º 45'N and 7º 15'N and longitude 6º 50'E and 7º 25'E with land area of 5,530Km^2 and an estimated population of about 4.8 million people (National Population Commission, NPC, 2016). It is bordered by Abia State in the west, in the south and east by Rivers State and Anambra State on the north. The state is divided into three agricultural zones, namely Okigwe (6 LGAs), Orlu (10 LGAs) and Owerri (11 LGAs).

Agriculture is assumed to be one of the major sources of income of most of the 3.9 million inhabitants of the area though most inhabitants are government employees. The staple food crops grown in the area include cassava, cocoyam, yam, maize, okra, garden egg, pepper, melon, and vegetables etc (National Bureau Statistics [NBS], 2008). Many agribusiness activities such as livestock farming, crop production, agro processing and sale of agricultural inputs are undertaken in the state.

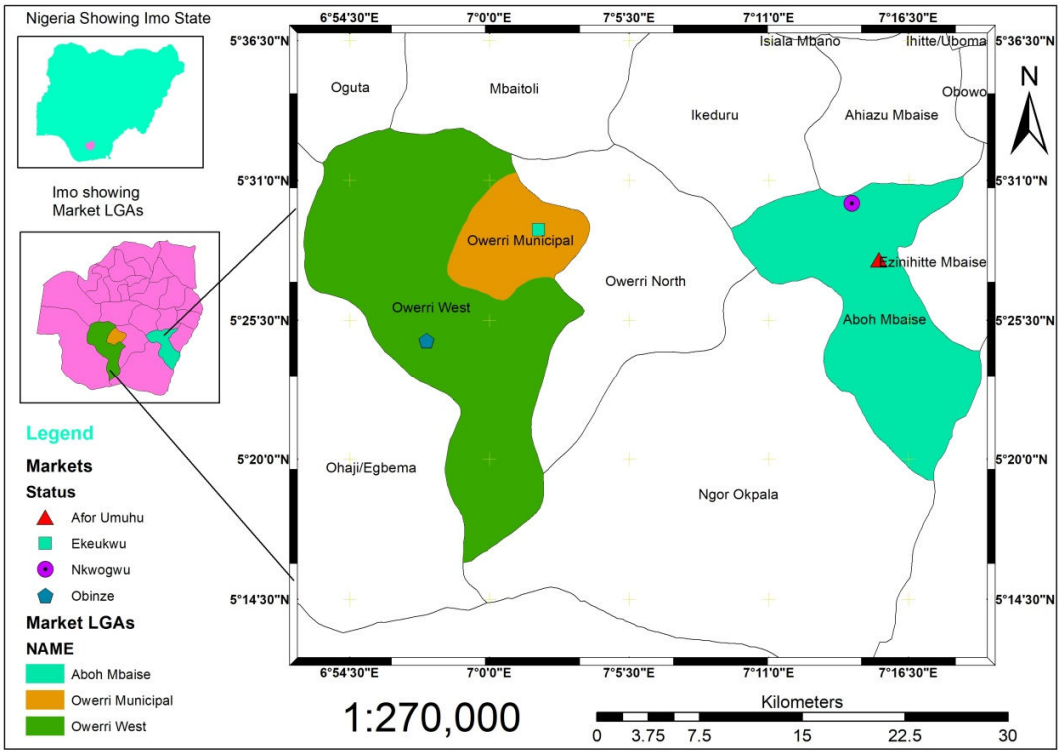

Figure 1. Study area showing all the Local Government Areas.

2.2. Sample Size Determination

The sample size was determined based on the population of traders in the selected markets and the volume of beans and maize sold. Using a stratified random sampling technique, samples were collected from different sections of each market to ensure representativeness.

2.3. Sampling Procedure

Sampling Units: The primary sampling units were individual vendors selling beans and maize.

Sample Collection: Approximately 500 grams of beans and maize were collected from each vendor. 2 samples were collected from each market to give a total of 8 samples.

Sample Handling and Transportation: Collected samples were placed in clean, labelled polyethylene bags and transported to the laboratory under refrigerated conditions to prevent degradation of pesticide residues.

2.4. Laboratory Analysis

Sample Preparation

Homogenization: Samples were homogenized using a blender to ensure uniform distribution of any pesticide residues.

Extraction: The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method were used for extracting pesticide residues from the samples. This involved the use of acetonitrile for extraction followed by a cleanup step using dispersive solid-phase extraction.

2.5. Equipment And Apparatus

- Sample (Beans and Maize)
- n-Hexane
- Glassware
- Whatman No 1 Filter Paper
- Teflon-line Screw cap vial (GC grade)
- Mortar and pestle
- Sieve (2mm)
- Gas Chromatography
- Mass Spectrometer

2.6. Sample Preparation

- The dried samples were crushed to powder with a mortar and pestle.
- The crushed samples were sieved with a 2mm sieve to fine powder attaining a homogenous sample.

2.7. Extraction Procedure

- 10g of the prepared samples were weighed representatively into a clean beaker.
- 10ml of n-Hexane was added appropriately.
- The mixture was Sonicated for 45-60 minutes under 70 degrees to extract the pesticides.
- The solvent phase was separated from the sample through a Whatman No. 1 filter paper.

2.8. Clean-Up Procedure

- The pesticides were eluted from the column with n-Hexane.
- The eluate was concentrated to 1ml.

2.9. GC Condition

The extracted samples were analyzed for Organochlorine Pesticide analysis by Gas Chromatography-Mass Spectrometry with an Agilent 6890A Gas chromatograph (GC) coupled with an Agilent 5973 mass selective detector (Agilent Technologies, Santa Clara, USA). A DB-5 capillary column (30m length × 0.15 µm film thickness × 0.25 mm I.D.) was used for separation, and pure helium gas at a flow velocity of 2.0mL/min was used as the carrier gas. The gas chromatographic column had an initial temperature of 90°C, which was held for 0min, and was then increased by 24°C min-1 to 300°C at a final run time of 9mins. The temperature of the injection port, ion source, quadrupole, and transfer line were 250, 230, 150, and 280 °C respectively. The sample was injected into the GC via a split mode in a ratio of 20:1 with an injection volume of 3µL.

3. RESULTS AND DISCUSSION

Organochlorine pesticides (OCPs) are a class of synthetic chemicals used extensively for pest control in agriculture and public health. They are known for their effectiveness in eliminating insects and pests. Still, they also have significant environmental and health impacts due to their persistence and bioaccumulation properties, leading to widespread bans and restrictions. Modern pest control strategies focus on safer alternatives to mitigate these negative effects. In this study, 16 organochlorine pesticides were studied in Beans and Maize from four markets in Nigeria. The table below shows the analyzed OCP results from these markets in Beans and maize.

Table 1. Organochlorine Pesticides on Selected Agricultural Products Sold in Nigeria Market.

CONGENERS, (ppb)	AFOR UMUHU MARKET BEANS	AFOR UMUHU MARKET MAIZE	EKE UKWU MARKET BEANS	EKE UKWU MARKET MAIZE	NKWO OGWU MARKET BEANS	NKWO OGWU MARKET MAIZE	OBINZE MARKET BEANS	OBINZE MARKET MAIZE
Alpha-BHC	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Hexachlorocyclohexane	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Heptachlor	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.47
Beta-BHC	1.52	0.40	0.65	0.96	2.46	0.43	2.05	1.79
Aldrin	<0.01	<0.01	<0.01	0.29	<0.01	<0.01	<0.01	0.64
Delta-BHC	0.43	<0.01	<0.01	<0.01	<0.01	<0.01	0.40	7.06
Heptachlor Epoxide	1.88	1.90	3.34	11.53	3.85	1.79	3.65	116.51
Endosulfan I	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Dieldrin	9.43	1.12	2.64	2.55	1.49	1.15	1.95	1.4
Endrin	46.44	41.27	62.84	21.69	56.13	37.79	45.37	12.48
m, p'-DDD	8.60	<0.01	5.78	<0.01	<0.01	<0.01	8.31	6.15
Endosulfan II	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
p, p'-DDT	0.64	<0.01	0.43	0.18	0.20	<0.01	0.21	0.20
Endrin aldehyde	6.43	6.22	4.02	5.70	6.06	6.18	2.98	7.27
Endosulfan Sulfate	<0.01	11.24	6.36	14.92	<0.01	26.00	<0.01	<0.01
Endrin ketone	15.93	15.11	17.7	41.93	14.70	23.55	8.44	7.35
TOTAL, (ppb)	91.30	77.26	103.76	99.75	84.89	96.89	73.36	161.32

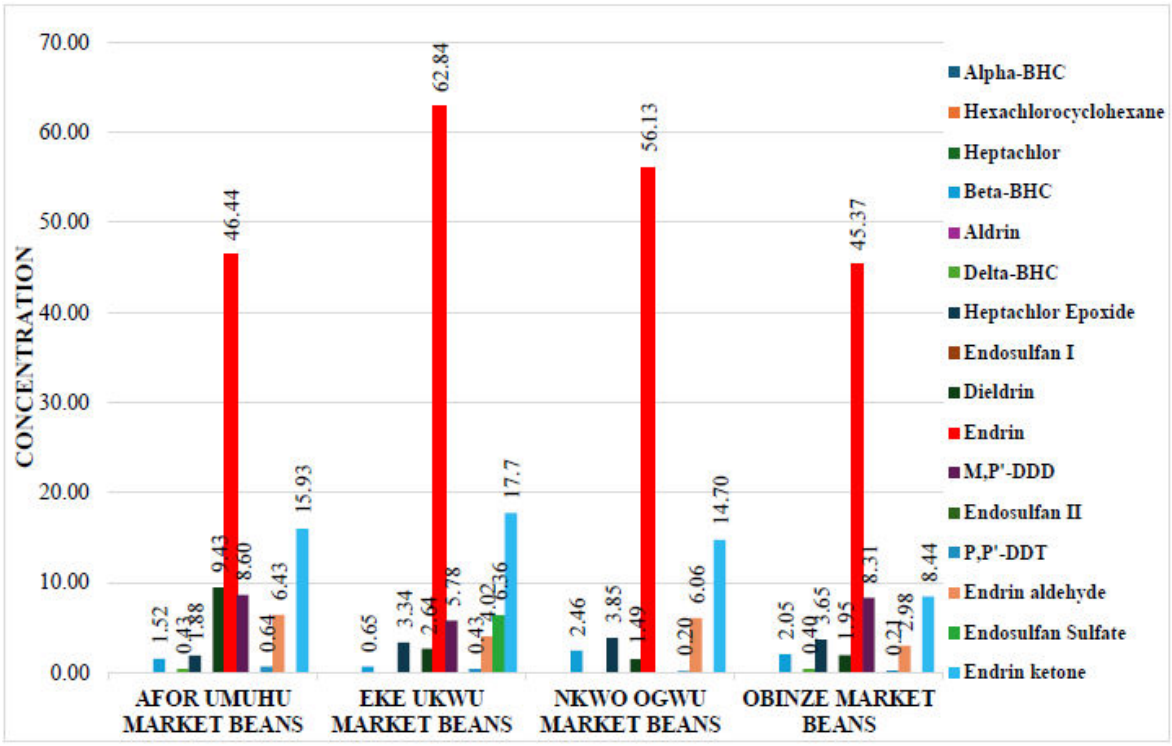

Figure 2. A chart showing the concentration levels of the 16 OCP congeners in Beans from four studied Markets in Nigeria.

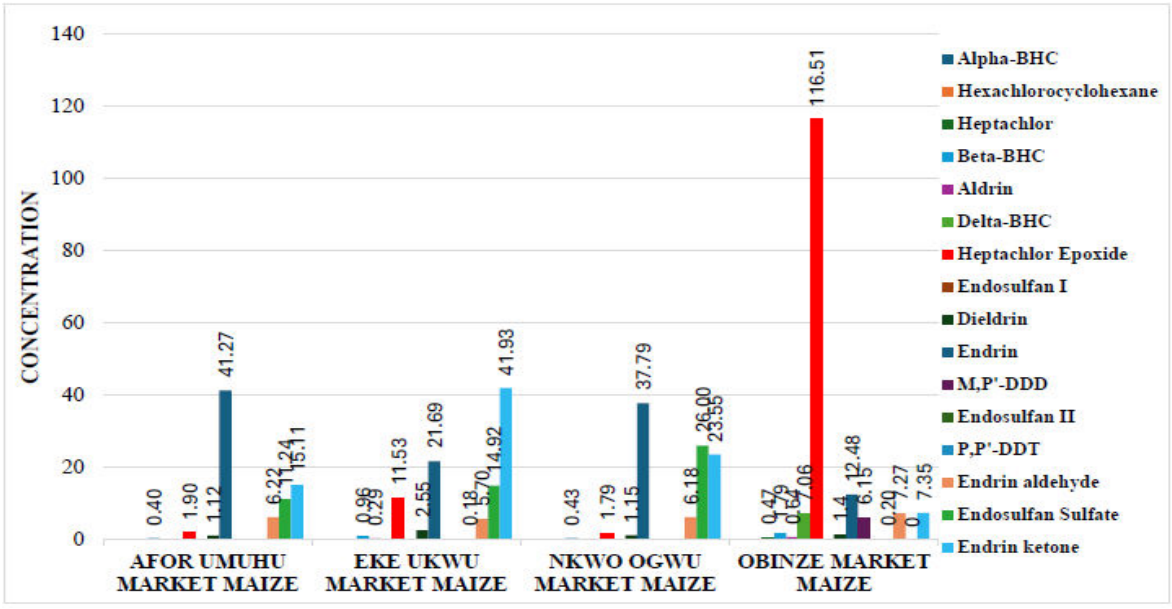

Figure 3. A chart showing the concentration levels of the 16 OCP congeners in Maize from four studied Markets in Nigeria.

From Table 1 above, the cumulative concentration of organochlorine pesticides (OCP) in the Bean samples ranges between 0.43 – 62.84 ppb for Eke Ukwu Market > 0.43 – 46.44 ppb for Afor Umuhu Market > 0.20 – 56.13 ppb for Nkwo Ogwu Market > 0.21 – 45.37 ppb for Obinze Market respectively. Similarly, the concentrations of the Maize samples range between 0.47 – 116.51 ppb for Obinze Market > 0.18 – 41.93 ppb for Eke Ukwu Market > 0.43 – 37.79 ppb for Nkwo Ogwu Market > 0.40 – 41.27 ppb for Afor Umuhu Market. Key observations from the analysis indicate a significant presence of toxic primary pesticides (which are used as Soil insecticides, Rodenticides, and Fungicides) such as Endrin, Aldrin, Endosulfan I, Heptachlor, and Hexachlorohexane. Other Pesticides present are derivatives such as Endrin Aldehyde and Endrin Ketone, degradative forms of Endrin, Endosulfate, and Endosulfan II, and degradative forms of Endosulfan I—likewise, Heptachlor Epoxide and Dieldrin, which are degradative form of Heptachlor and Aldrin, respectively.

Endrin is found at high levels in all samples, with the highest in Beans from Eke Ukwu Market > Nkwo Ogwu Market > Afor Umuhu Market > Obinze Market. A high level of Endrin was also analyzed in Maize, with the highest in Afor Umuhu Market > Nkwo Ogwu Market > Eke Ukwu Market > Obinze Market.

Endrin Ketone and Endrin Aldehyde, which are notable derivatives of Endrin, have a remarkable concentration, with Endrin Ketone having its highest in Beans from Eke Ukwu Market > Afor Umuhu Market > Nkwo Ogwu Market > Obinze Market. In the Maize, the highest concentration was seen in Eke Ukwu Market > Nkwo Ogwu Market > Afor Umuhu Market > Obinze Market. Endrin Aldehyde with a less significant but notable concentration is highest in Beans from Afor Umuhu Market > Nkwo Ogwu Market > Eke Ukwu Market > Obinze Market. While in Maize, the highest was considered in Obinze Market > Afor Umuhu Market > Nkwo Ogwu Market > Eke Ukwu Market.

Heptachlor Epoxide is found at a significantly high level in Maize from Obinze Market and Eke Ukwu Market, others are considered notable. The concentrations of Heptachlor Epoxide in Beans were at significant notable values respectively.

Aldrin was non-detected in Beans from all four Markets, with a notable concentration in Maize from Obinze Market > Eke Ukwu Market and was non-detected in Afor Umuhu and Nkwo Ogwu.

Dieldrin, another organochlorine pesticide, forms as a metabolite when Aldrin is broken down in the environment from living organisms, has a notable concentration across all samples, with the highest concentration in Beans from Afor Umuhu Market > Eke Ukwu Market > Obinze Market > Nkwo Ogwu Market. While in Maize, the highest concentration was noted in Eke Ukwu Market > Obinze Market > Nkwo Ogwu Market > Afor Umuhu Market.

Beta-BCH, also known as beta-hexachlorocyclohexane or β-BHC, is a chemical compound belonging to the group of organochlorine pesticides. It is one of several isomers of hexachlorocyclohexane. It has a notable concentration across all samples from the four markets, with the highest concentration in Beans from Nkwo Ogwu Market > Obinze Market > Afor Umuhu Market > Eke Ukwu Market, and in Maize, the highest concentration was seen in Obinze Market > Eke Ukwu Market > Nkwo Ogwu Market > Afor Umuhu Market.

Delta-BHC (delta-hexachlorocyclohexane or δ-BHC) is one of the isomers of hexachlorocyclohexane (HCH) and was detected with a notable concentration of less than 1.00 ppb in Beans from two Markets with the highest concentration in Afor Umuhu Market > Obinze Market, and non-detected in Eke Ukwu Market and Nkwo Ogwu Market. While in Maize samples, it was non-detected in Eke Ukwu Market, Nkwo Ogwu Market, and Afor Umuhu, with a significant concentration in Obinze Market, respectively.

Endosulfan Sulfate was discovered to be non-detected in Beans from three markets, with only Eke Ukwu Market having a notable concentration. While on Maize, three Markets were seen with a significant concentration, with the highest in Nkwo Ogwu Market > Eke Ukwu Market > Afor Umuhu Market, and non-detected in Obinze Market.

m,p'-DDD (also known as 2,2-bis(p-chlorophenyl)-1,1-dichloroethane) is one of the isomers and breakdown products of DDT (Dichlorodiphenyltrichloroethane), a well-known organochlorine pesticide, was detected in Beans from three markets, with the highest concentration in Afor Umuhu Market > Obinze Market > Eke Ukwu Market, and non-detected in Nkwo Ogwu Market. While in Maize, it was non-detected in three markets, with only Obinze Market having a notable concentration.

p,p'-DDT (para,para'-DDT or 1,1,1-Trichloro-2,2-bis(4-chlorophenyl)ethane) is the most common and well-known isomer of DDT (Dichlorodiphenyltrichloroethane) having a notable concentration less than 1.00ppb in Beans samples from all four markets, with the highest detected in Afor Umuhu Market > Eke Ukwu Market > Obinze Market > Nkwo Ogwu Market. In the Maize samples, a concentration less than 1.00 ppb was detected, with the highest noted in Obinze Market > Eke Ukwu Market, and non-detected in Afor Umuhu and Nkwo Ogwu Market.

Other congeners like Alpha-BCH, Hexachlorocyclohexane, Endosulfan I, Endosulfan II, and Heptachlor were non-detected across all samples with the last having only a notable concentration in Maize from Obinze Market.

Table 2. Analysis of Variance on Beans Sold in Imo State Market, Nigeria.

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
AFOR UMUHU MARKET BEANS	16	91.37	5.710625	139.9552329
EKE UKWU MARKET BEANS	16	103.83	6.489375	246.4743663
NKWO OGWU MARKET BEANS	16	84.98	5.31125	198.3450383
OBINZE MARKET BEANS	16	73.43	4.589375	126.1427663

ANOVA

Source of Variation	*SS*	*Df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	30.16894219	3	10.05631406	0.05658218	0.982143	2.758078
Within Groups	10663.76106	60	177.7293509			
Total	10693.93	63				

Table 2 shows One-ANOVA conducted to compare residue levels across four different markets on beans. Comparisons were made among Afor Umuhu Market Beans, Eke Ukwu Market Beans, Nkwo Ogwu Market Beans and Obinze Market Beans. The result showed that there was significant difference in beans residue levels across the four different markets at the $p < 0.05$ level for the four markets $F(3,60) = 2.758$, $p = 0.982$.

Table 3. Analysis of Variance on Maize Sold in Imo State Market, Nigeria.

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
AFOR UMUHU MARKET MAIZE	16	77.35	4.834375	115.1758263
EKE UKWU MARKET MAIZE	16	99.82	6.23875	133.4271717
NKWO OGWU MARKET MAIZE	16	96.98	6.06125	140.9180917
OBINZE MARKET MAIZE	16	161.37	10.085625	820.2126529

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	248.3146625	3	82.77155417	0.273685196	0.844142904	2.758078296
Within Groups	18146.00614	60	302.4334356			
Total	18394.3208	63				

Table 3 shows One-ANOVA conducted to compare residue levels across four different markets on maize. Comparisons were made among Afor Umuhu Market Maize, Eke Ukwu Market Maize, Nkwo Ogwu Market Maize, and Obinze Market Maize. The result showed that there was significant difference in maize residue levels across the four different markets at the $p < 0.05$ level for the four markets $F(3,60) = 2.758$, $p = 0.844$.

To further elucidate and understand the significant difference amongst the beans and maize from the four different markets, Pearson Correlation analysis is conducted.

Table 4. Pearson Correlation on Beans Sold in Imo State Market, Nigeria.

Descriptive Statistics

	Mean	Std. Deviation	N
AforUmuhuMarketBeans	5.7106	11.83027	16
EkeUkwuMarketBeans	6.4894	15.69950	16
NkwoOgwuMarketBeans	5.3113	14.08350	16
ObinzeMarketBeans	4.5894	11.23133	16

Correlations

		Afor Umuhu Market Beans	Eke Ukwu Market Beans	Nkwo Ogwu Market Beans	Obinze Market Beans
Afor Umuhu	Pearson Correlation	1	.975**	.968**	.974**
	Sig. (2-tailed)		.000	.000	.000
Market Beans	N	16	16	16	16
Eke Ukwu	Pearson Correlation	.975**	1	.989**	.984**
	Sig. (2-tailed)	.000		.000	.000
Market Beans	N	16	16	16	16
Nkwo Ogwu	Pearson Correlation	.968**	.989**	1	.978**
	Sig. (2-tailed)	.000	.000		.000
Market Beans	N	16	16	16	16
Obinze Market	Pearson Correlation	.974**	.984**	.978**	1
	Sig. (2-tailed)	.000	.000	.000	
Beans	N	16	16	16	16

**. Correlation is significant at the 0.01 level (2-tailed).

Table 4 shows Pearson Correlation analysis conducted for residue levels of beans in the four different markets shows that there is a significant difference at 1% level of probability tested at 2-tailed test. This implies that the beans from the four different markets when tested independently with each other in pairwise order all showed significant difference at 1% level of probability.

Table 5. Pearson Correlation on maize Sold in Imo State Market, Nigeria.

Descriptive Statistics

	Mean	Std. Deviation	N
Afor Umuhu Market Maize	4.8344	10.73200	16
Eke Ukwu Market Maize	6.2387	11.55107	16
Nkwo Ogwu Market Maize	6.0612	11.87089	16
Obinze Market Maize	10.0856	28.63935	16

Correlations analysis

		Afor Umuhu Market Maize	Eke Ukwu Market Maize	Nkwo Ogwu Market Maize	Obinze Market Maize
Afor Umuhu	Pearson Correlation	1	.685**	.933**	.025
	Sig. (2-tailed)		.003	.000	.927
Market Maize	N	16	16	16	16
Eke Ukwu	Pearson Correlation	.685**	1	.802**	.194
	Sig. (2-tailed)	.003		.000	.472
Market Maize	N	16	16	16	16
Nkwo Ogwu	Pearson Correlation	.933**	.802**	1	-.015
	Sig. (2-tailed)	.000	.000		.956
Market Maize	N	16	16	16	16
Obinze Market	Pearson Correlation	.025	.194	-.015	1
	Sig. (2-tailed)	.927	.472	.956	
Maize	N	16	16	16	16

**. Correlation is significant at the 0.01 level (2-tailed).

Table 5 shows Pearson Correlation analysis conducted for residue levels of maize in the four different markets shows that there is a positive but no significant difference at 1% level of probability tested at 2-tailed test for "Afor Umuhu Market Maize" with "Eke Ukwu Market Maize", "Afor Umuhu Market Maize" with "Nkwo Ogwu Market Maize", "Eke Ukwu Market Maize" with "Nkwo Ogwu Market Maize". The comparison between "Obinze Market Maize" and maize from the other Markets shows significant difference at the $p < 0.05$ level. This implies that the maize from Obinze Market shows significant difference in their residue level when compared with maize from the other markets while maize form Afor Umuhu, Eke Ukwu and Nkwo Ogwu markets shows no significant difference in their residue level when compared with each other at $p < 0.05$ level.

4. CONCLUSION

This study has provided a comprehensive assessment of the levels of organochlorine pesticide (OCP) residues in beans and maize sold in Imo State markets and evaluated the associated human health risks. The key findings and their implications are summarized as follows:

The analysis detected significant levels of various OCP residues, including DDT, aldrin, dieldrin, endosulfan, and heptachlor, in beans and maize samples. This indicates a persistent problem with OCP contamination in agricultural produce sold in Imo State markets.

The presence of OCP residues in commonly consumed food items such as beans and maize pose a significant threat to food safety and public health. The findings underscore the need for urgent action to address this issue through improved regulatory enforcement, monitoring, and public awareness campaigns.

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