

## Assessment of Insecticide Residues in Maize Grains from Major Markets in Ogbomoso, Nigeria

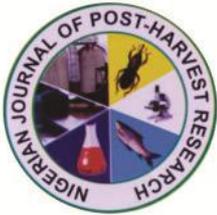
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### Abstract

*Maize (Zea mays L.) is a staple cereal crop of significant economic and nutritional importance worldwide. However, its production and postharvest storage are frequently compromised by infestations of insect pests, leading to yield and quality losses. The predominant method of pest control in maize storage involves the application of synthetic insecticides. This study evaluated the concentration of insecticide residues in maize grains sourced from major markets in Ogbomoso, Nigeria. Snowballing sampling technique was used to randomly select maize marketers from three markets: Odo-Oba, Iluju, and New Waso. Ten maize stores were sampled in each market. Composite samples representative of each market were prepared by combining 100 g subsamples from each store, with three replicates per market. Samples were transported to the laboratory, milled, and analyzed for pesticide residues using the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) extraction method, followed by gas chromatography – mass spectrometry (GC-MS) analysis on a Varian 3800/4000 instrument. Descriptive statistics and one-way analysis of variance (ANOVA) were performed using SAS version 9.2, with mean comparisons conducted using Duncan's Multiple Range Test at  $p < 0.05$ . Dichlorvos (2,2-dichlorovinyl dimethyl phosphate) was detected in all maize samples from three markets, ranging from 144.1 to 269.7 mg/kg, vastly exceeding the EU-MRL of 0.01 mg/kg. Such high residues pose severe health risks, including genotoxic, neurotoxic, reproductive, carcinogenic, respiratory, and dermal effects, potentially causing systemic toxicity or death. Urgent measures are needed to mitigate contamination and protect public health.*

### Keywords:

Dichlorvos, Gas Chromatograph, Maize, Pesticide, QuEChERS.

### Introduction

Food is essential for life, providing the necessary nutrients to sustain it, even at the cellular level. Food security is a crucial scientific and socioeconomic factor in human society, affecting all individuals regardless of age, background, gender, or social status (Ilesanmi et al., 2021). The United Nations Committee on World Food Security defines food security as a state where everyone has reliable access to adequate, safe, nutritious, and culturally acceptable food, allowing them to meet dietary needs for an active and healthy life (Verneau et al., 2021). However, this is often not the case in practice, as food is primarily considered in quantity, with less focus on its safety and nutritional value (Ojogiwa et al., 2023).

Maize (*Zea mays* L.) is one of the world's most widely cultivated cereal crops (FAOSTAT, 2022). It is a staple food for millions of people in developing West African countries and is key to human nutrition (Fasanya & Odudu, 2021). In Nigeria, millet and maize are widely consumed in diverse states. Maize is used to prepare traditional dishes such as *tuwo* and *akamu* or transformed into various products, including complementary infant foods (Oshatunberu et al., 2023). Maize cultivation is essential in ensuring food and livelihood security for resource-limited smallholder farmers in regions where maize

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is a primary food source. These farmers typically function as both producers and consumers (joint production-consumption), with the possibility of selling any surplus yield (Poole et al., 2021). Maize is a fundamental ingredient in poultry and livestock feed and is also a vital cereal in supplemental foods for infants older than six months (Hirvonen et al., 2021). While particular attention was given to increasing maize production, comparatively little has been done to limit postharvest losses, which have increased between 30 and 40% in the last decade (Hotegni et al., 2024). Losses in stored maize are induced by insect pests, among which *the major ones are Sitophilus zeamais and Prostephanus truncatus*.

Thus, maize producers still rely on synthetic pesticides to control insect pests in stored maize (Sissinto-Gbenou et al., 2021). A survey study revealed the application by producers of insecticides containing the following active ingredients: Dichlorvos and lambda-cyhalothrin are commonly applied by farmers and marketers for the control of fall armyworm (*Spodoptera frugiperda*) in field, as well as for the control of stored-product pests, specifically *Sitophilus zeamais* and *Prostephanus truncatus*, in storage facilities (Gbadegesin et al., 2024).

Pesticides are broadly used to guarantee trim efficiency and to preserve the tall dietary and clean quality of grains by securing crops from bugs, pathogens, and weeds (Mert et al., 2020). Since they are connected amid the edit cycle, residues of active ingredients (A.I.) associated with the field can be identified in agrarian commodities after gathering (Santarelli et al., 2018). Their drawn-out ingestion may represent a danger to human well-being since there's expanding evidence of the frequency of long-term pesticide exposure on several chronic human infections, including cancer. For these reasons, each nation has set up Maximum Residue Levels (MRLs), the most noteworthy amounts allowed in foods and feeds (Das et al., 2020). Cultivated crops, natural products, and vegetables are the primary dietary sources contributing to human exposure to pesticide residues due to their widespread consumption. Pesticides are routinely applied in agricultural practices to mitigate pre- and postharvest losses, with residues often persisting from the point of field application through to market distribution (Szpyrka et al., 2015). In 2018, approximately 147,446 tonnes of pesticides were imported into the country, primarily for agricultural use, of which 584 were classified as highly hazardous (FAOSTAT, 2020). Inadequate regulatory oversight and the frequent detection of pesticide residues in food commodities

have emerged as significant global public health and environmental concerns (Wahab et al., 2022).

In Nigeria, the staple diet is predominantly based on two major food groups: root crops and tubers, which contribute approximately 20% of dietary calories and 8% of protein, and cereal grains, which provide 46% of calories and 52% of protein. An estimated 200 million hectares (M ha) are dedicated to cultivating the three primary staple cereals. However, food security across Africa is significantly undermined by high levels of rural poverty and substantial postharvest losses, often exacerbated by pest infestation. Conventional methods to reduce such losses have frequently affected food quality and environmental safety, raising further concerns about sustainability and human health (Thakur et al., 2025).

The present study evaluated the levels of organophosphate and organochlorine pesticide residues in maize grain samples collected from Ogbomoso, Nigeria. Pesticide residues were extracted using the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) method and subsequently analyzed via gas chromatography–mass spectrometry (GC-MS) using a Varian 3800/4000 instrument.

## Materials and Methods

### Sampling area

The research was conducted at the Crop and Environmental Protection Department of Ladoke Akintola University of Technology (LAUTECH), Ogbomoso, where LAUTECH is situated. Ogbomoso is a city in Oyo State, southwestern Nigeria, with an estimated population of about 551,474 in 2020. A larger percentage of the farmers are located in the city's rural areas. In terms of the climatic condition of the town, Ogbomoso is found in the tropical rain forest zone and lies 334 m above sea level with an assurance of a bimodal rainfall pattern. The rainfall is about 1217 mm annually, with temperatures between 16 °C and 29 °C.

### Sampling

Snowballing sampling technique was used to select three major markets: New Wazo, Odo-Oba, and Iluju, located along the Oba River in Ogbomoso. Marketers in Iluju sourced their grains from the Iluju Irrigation Farms along the Oba River. In contrast, those in Odo-Oba and New Wazo obtained their maize from the farmers cultivating dry and wet seasons in the riverbank of the Oba River. In each market, 10 maize stores were randomly selected, and 100 g of maize was collected from each store. The samples from each

market were composited to form a representative sample, which was subsequently replicated three times. All samples were bagged separately and transported to the Crop and Environmental Protection Laboratory, Ladoke Akintola University of Technology (LAUTECH), Ogbomosho, for further laboratory procedures.

### **Extraction and clean-up procedures**

AOAC Pouch Qsep (QuEChERS Sample Extraction Pouches) sachets containing magnesium sulfate and sodium acetate were obtained from Agilent Technologies (Santa Clara, USA). HPLC-grade acetonitrile was sourced from Romil (Cambridge, UK). At the same time, glacial acetic acid, primary and secondary amine (PSA), deionised water, acetone, and HPLC-grade pesticide standard solutions were procured from Sigma-Aldrich (Germany). Pesticide residues were analysed using a multi-residues extraction and clean-up procedure based on the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method, following the AOAC official method 2007.01 with slight modifications to accommodate the spiking of multiple analytes.

### **Gas chromatography analysis**

The samples were analyzed using Gas Chromatography-Mass Spectrometry (GC-MS) using a Varian 3800/4000 system to identify and quantify pesticide residues. Chromatographic separation was achieved on a capillary column AT-1, length was 30 m, ID was 0.25 mm, and film thickness was 0.25  $\mu\text{m}$ . The carrier gas was nitrogen (purity 99.00%) supplied at a constant flow rate of 1.5 mL/min. The GC oven was programmed with an initial temperature of 70  $^{\circ}\text{C}$  to 300  $^{\circ}\text{C}$  and held for 2 min, then rose to 300  $^{\circ}\text{C}$  and held again for 7 min at 10  $^{\circ}\text{C} \cdot \text{min}^{-1}$ . The total run time was 32.0 minutes. The GC-MS interface temperature was at 280  $^{\circ}\text{C}$ . Injector and detector temperatures were set at 250  $^{\circ}\text{C}$ , flow control mode: linear velocity; 1  $\mu\text{l}$  of sample was injected in a split ratio of 30:0. The Mass Spectrometer was operated with a scan range of 40-800 Da. Suspected pesticide compounds were identified by comparing their retention times in sample chromatograms to those of the corresponding pure analytical standards, along with data from an established compound library. The quantities of detected compounds were expressed as relative area percentages, as calculated by the system integrator.

Mafura (2024) pointed out that the detection limit (LOD) of GC-MS equipment used for each pesticide was determined by running an air blank sample under

the experimental conditions to obtain the detector baseline noise. A detectable ion should produce a signal at least three times the baseline noise. Individual pesticide LOD was estimated by serially running diluted solutions of the pesticide standard and recording the baseline noise ratio (signal-to-noise = 3).

The analysis was conducted at Afe Babalola University's Chemical Engineering Laboratory in Ado Ekiti, Nigeria.

### **Statistical analysis**

Descriptive statistics such as mean, range, and standard error were employed to analyze the data using SAS software version 9.2 (SAS Institute, 2005). Additionally, pesticide residue levels from the three selected markets were statistically evaluated using Analysis of Variance (ANOVA), and mean differences were determined through Duncan's Multiple Range Test at a 5% significance level.

### **Quality assurance procedures**

Quality assurance procedures were implemented to ascertain that the analytical methods rigorously adhered to standards and specifications. Additionally, these measures helped guarantee that the results evaluated were valuable, valid, and of high standard, including assessing recovery rate and detection limits.

## **Results and Discussion**

### **Concentration of insecticide residue in maize samples sourced from selected markets**

The quantification of pesticide residues in food samples through nutritional testing indicates human exposure levels to these chemical agents, thereby enabling an assessment of potential risks to health and well-being. As shown in Table 1, the measured concentrations of dichlorvos residues indicate detectable levels of this compound in the tested samples. The crucial concentration identified extended from 144.01 to 269.7  $\text{mg kg}^{-1}$ . The lowest residue concentration of dichlorvos (144.01  $\text{mg kg}^{-1}$ ) was detected in samples from Odo Oba market, while the highest mean concentration was observed in samples from New Wazo market. Compared to the European Union Maximum Residue Limits (EU MRLs) of 0.01  $\text{mg kg}^{-1}$ , all detected dichlorvos levels in the sampled grains significantly exceeded the regulatory threshold. These findings indicate dichlorvos' unregulated and excessive use in the selected markets. This observation aligns with Yusuf et al. (2021), who reported persistent dichlorvos residues in cowpea grains up to six months after application. The concentrations observed in this

study surpass established safety limits, suggesting that the treated grains are unfit for human consumption and may pose significant health risks due to the inherent toxicity of dichlorvos.

The results confirm that marketers routinely apply dichlorvos to preserve stored cereal grains. Although various commercial formulations were used, the active ingredient in all cases was identified as dichlorvos. While primarily intended for field control of insect pests, dichlorvos is widely repurposed for postharvest pest management in storage systems. This practice is consistent with findings by Pil et al. (2019), who documented the prevalent use of dichlorvos in agricultural communities in Makurdi, Benue State, highlighting its integration into local pest control strategies. The typical method of application involves diluting the pesticide in water and spraying it directly onto grains before storage in polyethylene (commonly referred to as "pillow" or "polybag") packaging. This practice, especially when applied at non-recommended rates, leads to substantial residual contamination of the grains.

According to the World Health Organization (WHO), dichlorvos (DDVP) is classified as a Class 1B "highly hazardous" organophosphorus insecticide. It is frequently involved in cases of intentional self-poisoning in low- and middle-income countries (Jain et al., 2024). The widespread and unregulated handling of such toxic substances by stakeholders in postharvest storage systems poses a serious public health threat by distributing contaminated food products (Nwosu et al., 2018). Market demand, economic incentives, profit maximization strategies, and differences in individual vendor practices may influence variability in residue levels across samples. Yusuf et al. (2021) define the "waiting period" as the interval between pesticide application and chemical degradation into non-toxic metabolites. This implies that longer storage durations may reduce residue toxicity, improving food safety. Conversely, grains stored for only one to two months may retain hazardous levels of dichlorvos, rendering them unsafe for consumption.

No organochlorine pesticides were detected in any of the analyzed samples, which is consistent with their official ban on agricultural use. Dichlorvos was the sole organophosphate insecticide identified in the tested grain samples, and all detected concentrations exceeded EU-MRLs. This is concerning, as the European Food Safety Authority (EFSA, 2023) has previously reported a higher likelihood of fruits and vegetables exceeding MRLs than other food commodities. However, in this context, the violation

pertains to staple grains. The misuse appears to stem from limited awareness among vendors in rural and semi-urban markets and the unregulated application of agrochemicals.

Chronic exposure to pesticide residues can lead to bioaccumulation in humans and animals, potentially resulting in long-term toxicological effects (Ogah & Coker, 2012). The present findings corroborate those of Waras et al. (2024), who also detected dichlorvos residues in beetroot samples from retail outlets in Kano State, Nigeria, further confirming its use in postharvest grain protection.

In Nigeria, improper and often excessive use of insecticides for grain storage is widespread. Documented cases include acute poisoning, sudden deaths, blindness, and dermatological reactions linked to dichlorvos exposure (Adeleke, 2009; Anaduaka et al., 2023). Luiz et al. (2002) reported a case of delayed organophosphate-induced neuropathy in a 39-year-old woman following ingestion of a dichlorvos-based insecticide two weeks prior.

Moreover, several incidents of mass food poisoning have been attributed to pesticide-contaminated stored foods. For instance, a study reported an outbreak of food poisoning in a rural community after consuming wheat contaminated with malathion. The grain had been stored in gunny bags previously used to transport insecticides. Symptoms included vomiting, diarrhea, and neurological signs. The investigation confirmed malathion residues in the wheat samples (Sharma et al., 2008). Similarly, a report documented the death of four individuals in a household within the Enugu State University community following the consumption of beans contaminated with unauthorized storage chemicals (PUNCH, August 2024; Whistler Newspaper, September 2024). The World Health Organization (WHO) has highlighted that improper use of insecticides in grain storage is a significant public health concern, particularly in developing countries. Chronic low-level exposure can lead to long-term health issues, including neurodevelopmental problems in children (WHO, 2021).

### **Variation in residue levels of insecticide concentration in maize grain samples among the three selected locations**

Figure 1 illustrates the variation in residue levels of insecticide concentration in maize grain samples from the selected markets. The highest pesticide residues were detected in maize grains sampled from Wazo market, with a mean concentration of 270 mg kg<sup>-1</sup>, significantly higher ( $p < 0.05$ ) than those recorded in

other selected markets within the study area. In contrast, the lowest residue level ( $144 \text{ mg kg}^{-1}$ ) was observed at Odo Oba market. These findings suggest an excessive application of dichlorvos at the Wazo market, rendering the maize grains sold therein unsafe for human consumption compared to samples from Odo Oba and Iluju markets. The variation in residue levels among the three selected markets may be due to inadequate knowledge regarding pesticide use for

postharvest grain protection among marketers and farmers. This is consistent with anecdotal evidence indicating reliance on experiential practices rather than standardized protocols in pesticide handling. While accumulated practical experience may contribute to certain aspects of pest management, it cannot substitute for formal training and scientific understanding required to safely and effectively use chemical agents.

**Table 1: Concentration of Insecticide Residue in Maize Samples Sourced from Selected Markets**

Pesticides	Locations and Concentration				P- value	MRL ( $\text{mg kg}^{-1}$ )
	Iluju ( $\text{mg kg}^{-1}$ )	Wazo ( $\text{mg kg}^{-1}$ )	Odo Oba ( $\text{mg kg}^{-1}$ )	R/Time		
Dichlorvos	241 <sup>b</sup> ±0.47	269 <sup>a</sup> .7±0.48	144.1 <sup>c</sup> ±0.44	7.65	0.05	0.01
Carbofuran	<0.001	<0.001	<0.001	-	<0.001	0.01
Mevinfos	<0.001	<0.001	<0.001	-	<0.001	0.01
Dimothoate	<0.001	<0.001	<0.001	-	<0.001	0.01
Diazinon	<0.001	<0.001	<0.001	-	<0.001	0.01
Etrimfos	<0.001	<0.001	<0.001	-	<0.001	0.01
Pirimicarb	<0.001	<0.001	<0.001	-	<0.001	0.01
Chlorpyrifos	<0.001	<0.001	<0.001	-	<0.001	0.01
Metribuzin	<0.001	<0.001	<0.001	-	<0.001	0.01
Fenitrothion	<0.001	<0.001	<0.001	-	<0.001	0.01
Malathion	<0.001	<0.001	<0.001	-	<0.001	0.01
Chlorfenvifos	<0.001	<0.001	<0.001	-	<0.001	0.01
Bromophos	<0.001	<0.001	<0.001	-	<0.001	0.01
Ethion	<0.001	<0.001	<0.001	-	<0.001	0.01
Azinphos-methyl	<0.001	<0.001	<0.001	-	<0.001	0.01
PCB – 153	<0.001	<0.001	<0.001	-	<0.001	0.01

Means with the same superscript(s) are not significantly different along the column at 5% probability ( $p>0.05$ ). LOD:  $<0.001 \text{ mg kg}^{-1}$ . Results represent mean  $\pm$  standard deviation of group pesticide residues obtained ( $n = 3$ ). MRL: Maximum Residue Limit.

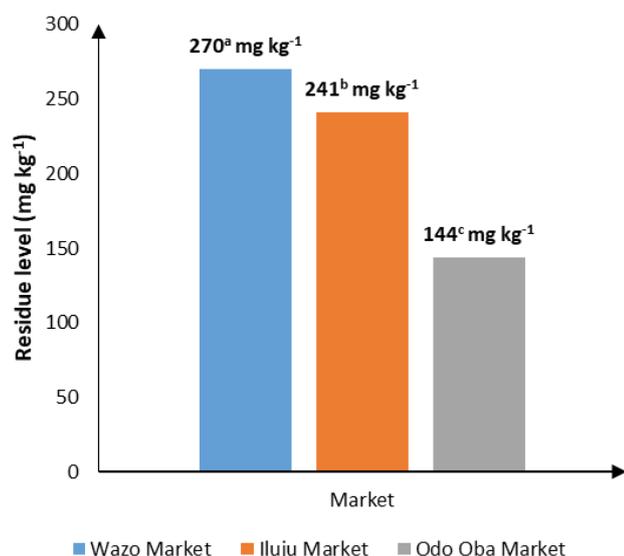


Figure 1: Insecticide Residue Levels in Maize Grain Samples among the Three Selected Locations.

Means with the same superscript(s) are not significantly different along the column at 5% probability ( $p>0.05$ ).

These results align with findings by Bhandari et al. (2018), who reported widespread non-compliance with

label instructions during pesticide application, including overuse, increased frequency of treatments, and shortened intervals between applications. Similarly, Nwadike et al. (2021) identified the misuse of agrochemicals in grain storage as a major contributor to Nigeria's rising incidence of foodborne illnesses. The significant variation in dichlorvos residue concentrations across the three markets may also be linked to premature release of treated grains, excessive dosage, and deviation from manufacturer guidelines, and insufficient awareness of safe application procedures (Oshatunberu, 2023).

These findings imply lack of studies assessing health risks associated with chronic exposure to dichlorvos through maize consumption in local populations, limited data on the specific sources and timing of contamination and insufficient understanding of socio-behavioral factors influencing pesticide misuse among smallholder farmers and traders also a lack of adherence to recommended application guidelines, likely driven by inadequate knowledge and reliance on informal, experiential practices among farmers and

marketers. This aligns with existing literature (Bhandari et al., 2018; Nwadike et al., 2021; Oshatunberu, 2023), which reported agrochemical misuse and its public health impacts.

### Conclusion and recommendation

This study revealed that maize grains sold in major markets in Ogbomoso contain high levels of Dichlorvos residues, ranging from 144.1 to 269.7 mg/kg, significantly exceeding the EU Maximum Residue Limit of 0.01 mg/kg. The widespread detection of this highly hazardous organophosphate pesticide, prohibited for postharvest use, indicates improper handling practices among farmers and marketers who apply field pesticides directly to stored grains. These findings underscore a critical public health risk, as chronic exposure to such residues is linked to neurotoxicity, organ damage, and increased risk of chronic diseases. The absence of organochlorine pesticides reflects compliance with existing bans, yet the unchecked use of dichlorvos highlights gaps in regulation, monitoring, and awareness within the food supply chain.

### Recommendation

To safeguard food safety and public health, there is an urgent need for strengthened regulatory enforcement, improved extension services, and nationwide surveillance of pesticide residues in staple crops. Promoting safer alternatives such as hermetic storage and biological control can reduce reliance on toxic chemicals while preserving grain quality. These findings raise urgent public health concerns, particularly regarding chronic dietary exposure. Regulatory agencies must intensify surveillance and farmer/marketer education, while promoting safer storage alternatives. Future studies should evaluate consumer exposure levels and explore integrated pest management options for stored grain protection.

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