



Research Article

Assessment of Pesticide Residues on Stored Cereals and Legumes in Major Markets of North-Eastern Nigeria

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ARTICLE INFO:

Keywords:

Pesticides,
Residue,
Cereals,
Legume.

ABSTRACT

The study was conducted to evaluate pesticide residue levels in cereals and legumes from major markets in Adamawa, Bauchi, Borno, Gombe, Taraba, and Yobe States in North-eastern Nigeria. Grain/legumes samples were randomly collected and analysed using Gas Chromatography equipped with an Electron Capture Detector (GC-ECD). The concentrations of commonly used pesticides, including cypermethrin, deltamethrin, carbaryl, dichlorvos, and propoxur, were determined and compared with established European Union Maximum Residue Limits (MRLs). The results revealed that several samples, particularly cowpea, maize, and groundnut, contained pesticide residues exceeding permissible limits, with cypermethrin reaching up to 5.3 mg/kg and dichlorvos up to 9.7 mg/kg in Taraba state when compared with other states. Significant variation in residue levels were observed in the study areas. The findings indicate widespread misuse and over-application of pesticides during storage, posing potential risks to food safety and public health. Strengthening regulatory enforcement, promoting safe pesticide handling practices, and routine monitoring of food commodities are necessary to mitigate exposure risks.

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INTRODUCTION

Cereals and legumes storage prior to sale by merchants has faced challenges to maintain its safety qualities during post-harvest handling (Nicholson and William, 2021). They play a crucial role in feeding families across Sub-Saharan Africa. Poor practices from storage methods, through handling and pesticide usage have been of great concern, leading to the suspension of its exportation into some countries (Nipper *et al.*, 2024). However, Pesticides play a crucial role in modern agriculture, aiding in pest control (Kaur *et al.*, 2019; Tesi *et al.*, 2022). The use of pesticides in preserving agricultural produce plays a significant role in mitigating post-harvest losses, extending shelf life, and ensuring food security (Amare *et al.*, 2022). By controlling pests and diseases that can degrade quality and quantity, pesticides enhance the longevity during storage and transport (Obafemi *et al.*, 2024). However, this practice must be carefully managed to prevent the accumulation of harmful pesticide residues, which can pose health risks to consumers (Hajam and Kumar, 2022). Balancing the benefits of pesticide use with the need for food safety requires adherence to recommended application rates, timing, and methods, along with rigorous monitoring and regulation to ensure that residue levels remain within safe limits (Ogbeide *et al.*, 2018). However, the indiscriminate use and improper application of pesticides can lead to harmful residues in food products, posing risks to human health and the environment (Oshatunberun *et al.*, 2023; Rabiun and Abubakar (2025). These include potential health hazards to humans-ranging from acute poisoning to long-term effects such as cancer and endocrine disruption-as well as environmental issues like biodiversity loss, water contamination, and the decline of beneficial species such as pollinators (Osipitan *et al.*, 2021; Iniaghe *et al.*, 2025). Concerns over pesticide contamination led the European Union (EU) in 2015 to impose a ban on Nigerian dried grains, after repeated detection of dichlorvos and other residues above Maximum Residue Limits (MRLs) (European Commission, 2015). This embargo has had serious implications for Nigeria's agricultural economy, excluding the country from a lucrative export market and undermining the reputation of its value chain (Oyebamiji *et al.*, 2018). This limitation points to a critical need for expanded, nationwide surveillance to fully capture residue patterns, compliance rates, and risk hotspots across Nigeria and beyond (Popoola *et al.*, 2024). Furthermore, Nigeria lacks a centralized

Database, a structured digital platform for aggregating sample-level data on production, pest management practices, residue testing outcomes, and trade readiness indicators (Rahman and Chima, 2018). Such a database would enable policymakers, researchers, and regulators to monitor trends, benchmark compliance, and design interventions more effectively (Silva *et al.*, 2019; Ekanem *et al.*, 2025). This study addresses this gap by providing a comparative assessment of pesticide residues across key cereals and legumes in major markets in north eastern Nigeria.

METHODOLOGY

Collection and Preparation of samples for quantification of pesticide residues

A grain/legume samples of maize, guinea corn, groundnut, cowpea, Bambara nut, rice, and wheat were collected from each market across north-eastern states. One kilogram (1kg) sample of grains/legume was randomly purchased from grain merchant in each of the markets. Preparation of collected sample for analysis was done as described by Asogwa *et al.* (2023). The samples were sorted to remove stones and debris. A fraction of each sample (30.0g) was evenly pulverized into 20 mesh particle size and were individually stored in glass bottles with appropriate labels for further analysis.

Analysis and quantification of samples for pesticide residues on stored cereals and legumes grains

The analysis was conducted according to the standard methods of AOAC (1990).

Extraction of Polychlorinated Biphenyls (PCBs) from samples

A ten gram grounded sample was weighed and quantitatively transferred into a 500ml beaker. 6g sodium sulphate was added and extracted using 300ml n hexane to get a concentrated filtrate. 1ml of filtered residue was dissolved in 50ml of chloroform and transferred to a 100ml volumetric flask which afterwards was diluted to the mark. Most chloroform were allowed to evaporate at room temperature. 1ml of the reagent (20 vol% benzene and 55 vol% methanol) was added. This was sealed and heated at 40°C water bath for 10 minutes. Thereafter, extraction of the organic sample was carried out using hexane and water in the ratio of 1:1:1 (i.e. the final mixture of the reagent, hexane and water respectively). The mixture was manually shaken vigorously for 2min. lastly, half of the top hexane phase was carefully transferred to a small test tube for injection.

Gas Chromatographic conditions for PCB Determination

Preparation of Standard

An aliquot (10 μ l) of AccuStandard was injected in the chromatography and the retention time compared with retention time of standard.

Statistical Analysis

The results obtained were statistically analysed through statistix version 8.0. The concentration of pesticide residues in the samples was compared with the MRLs recommended by the European Union (2011). MRL of a pesticide is the maximum concentration of its residue that is legally permitted to remain in food after it has been treated with the pesticide (FAO, 2002).

Level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Yobe State.

The results show that all measured pesticide residue levels (RLs) in the samples were significantly different from each other. And some are lower than the corresponding European Union Maximum Residue Limits (MRLs), indicating full compliance with European food safety standards (European Commission, 2005). The result indicates that, Cypermethrin, deltamethrin, carbaryl and propoxur in maize sample in the Yobe sample was detected at 1.2, 1.5, 1.2 and 2.0 mg/kg respectively, which falls above the EU's maximum allowed limit of 0.05 mg/kg, reflecting a high and unacceptable concentration (EU Pesticides Database, 2023). In

Guinea corn samples it reveals that all the pesticides analysed had shown lower concentration except cypermethrin, diclorvous and propoxur. Furthermore, in ground nut sample the maximum concentration level was recorded also against cypermethrin 2.3 mg/kg, deltamethrin 2.6 mg/kg, diclorvous 2.3 mg/kg all above the maximum limit level. In cowpea sample all the pesticide analysed that is organochlorine, organophosphate, pyrethriod and carbamate had shown significantly higher concentration which exceed the maximum limit level as set by the European union, in Bambara groundnut all the pesticide analysed shows higher than the maximum limit set, except the chlorvous and metalaxyl shows lower concentration. Moreover, in rice sample the highest concentration of pesticide was recorded on cypermethrin 4.3 mg/kg, deltamethrin 1.5 mg/kg and 3.6 mg/kg on propoxur, while other pesticides that were analysed shows zero concentration which are significantly below the maximum limit. In wheat all the pesticides analysed shows higher maximum limit level except deltamethrin, carbaryl, chlorvous, malathion and metalaxyl which shows lower limit concentration which were not up to maximum limit set by the European union (Table 1).

Table 1: Showing the level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Yobe State

Insecticides	Maize	Guinea corn	Ground nut	Cowpea	Bambara nut	Rice	Wheat
Bifenthrin	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^b	0.0 \pm 0.0 ^c	7.4 \pm 5.7 ^a	1.2 \pm 3.3 ^c	0.0 \pm 0.0 ^d	1.5 \pm 1.2 ^{ab}
Cypermethrin	1.2 \pm 3.3 ^c	1.3 \pm 8.8 ^a	2.3 \pm 8.8 ^b	5.3 \pm 8.8 ^c	1.4 \pm 1.2 ^{de}	4.3 \pm 8.8 ^a	1.7 \pm 8.8 ^a
Deltamethrine	1.5 \pm 5.7 ^b	0.0 \pm 0.0 ^b	2.6 \pm 5.7 ^a	6.3 \pm 3.3 ^b	1.5 \pm 8.8 ^d	1.5 \pm 8.8 ^c	0.0 \pm 0.0 ^c
Carbaryl	1.5 \pm 1.2 ^b	0.0 \pm 0.0 ^b	0.0 \pm 0.0 ^c	6.4 \pm 1.2 ^b	2.3 \pm 3.3 ^c	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^c
Chlorvos	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^b	0.0 \pm 0.0 ^c	6.3 \pm 8.8 ^b	0.0 \pm 0.0 ^f	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^c
Chlorpyrifos	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^b	0.0 \pm 0.0 ^c	3.3 \pm 1.2 ^c	2.2 \pm 5.7 ^c	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^c
Malathion	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^b	0.0 \pm 0.0 ^c	4.2 \pm 8.8 ^d	2.0 \pm 5.8 ^c	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^c
Diclorvous	0.0 \pm 0.0 ^d	1.3 \pm 5.7 ^a	2.3 \pm 5.7 ^b	6.4 \pm 1.2 ^b	3.2 \pm 5.7 ^b	0.0 \pm 0.0 ^d	1.3 \pm 5.7 ^b
Propoxur	2.0 \pm 5.8 ^a	1.3 \pm 5.8 ^a	2.6 \pm 5.8 ^a	4.5 \pm 5.7 ^d	4.5 \pm 3.3 ^a	3.6 \pm 8.8 ^a	1.5 \pm 8.8 ^{ab}
Metalaxyl	0.0 \pm 0.0 ^d	0.0 \pm 0.0 ^b	0.0 \pm 0.0 ^b	0.0 \pm 0.0 ^f	0.0 \pm 0.0 ^f	0.0 \pm 0.0 ^f	0.0 \pm 0.0 ^c
F	354	283	901	647	549	1186	171
P	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

Level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Taraba State.

The result indicates that, bifenthrin, deltamethrin, carbaryl and metalaxyl in maize sample in the Taraba sample shows zero concentrations, which falls below the EU's maximum allowed limit of 0.05 mg/kg, reflecting an acceptable concentration for human

and animal consumption, but the remaining chemical/ pesticide analysed had shown a greater or higher maximum concentration set by the European commission. In Guinea corn samples it reveals that all the pesticides analysed had shown maximum higher concentration limit except bifenthrine, carbaryl and metalaxyl shows lower limit concentration. In Ground nut sample all the tested pesticides recorded maximum

concentration level which was above the maximum limit level, except bifenthrin and metalaxyl recorded the lower limit levels. In cowpea sample all the pesticide analysed had shown significantly higher concentration which exceed the maximum limit level except carbaryl, chlorvos, chlorpyrifos, Malathion and metalaxyl had shown the lower concentration limit. In Bambara groundnut all the pesticide analysed shows higher than the maximum limit set, except the metalaxyl shows lower concentration. Moreover, in rice sample the highest concentration of pesticide which exceed the maximum limit concentration was recorded on cypermethrine 4.3 mg/kg, chlorvos 9.7

mg/kg, chlorpyrifos 2.0mg/kg, malathion 2.3mg/kg and 7.3 mg/kg on diclorvos, while remaining other pesticides shows zero concentration which are significantly lower or significantly below the maximum limit. In wheat all the pesticides analysed shows higher maximum limit level except deltamethrine, carbaryl, chlorvos, propoxur and metalaxyl which shows lower limit concentration which were not up to maximum limit level set. However, bifenthrin records 2.3mg/kg, cypermethrine 1.3 mg/kg, chlorpyrifos 8.0mg/kg, malathion 1.5 mg/kg and diclorvos 5.3 mg/kg respectively which were greater than the maximum limit level (Table 2).

Table 2: Showing the level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Taraba State

Insecticides	Maize	Guinea corn	Ground nut	Cowpea	Bambara nut	Rice	Wheat
Bifenthrin	0.0±0.0 ^d	0.0±0.0 ^d	0.0±0.0 ^c	8.2±5.7 ^a	1.7±7.3 ^c	0.0±0.0 ^c	2.3±8.8 ^b
Cypermethrin	2.3±8.8 ^c	3.2±5.7 ^{ab}	4.6±3.4 ^a	0.1±5.8 ^c	3.2±8.8 ^b	4.3±1.2 ^b	1.3±3.3 ^c
Deltamethrine	0.0±0.0 ^d	3.4±8.8 ^{ab}	3.3±5.7 ^b	8.3±8.8 ^a	3.3±8.8 ^b	0.0±0.0 ^c	0.0±0.0 ^d
Carbaryl	0.0±0.0 ^d	0.0±0.0 ^d	2.0±2.0 ^c	0.0±0.0 ^c	1.1±1.1 ^c	0.0±0.0 ^c	0.0±0.0 ^d
Chlorvos	9.3±4.7 ^a	1.6±8.2 ^c	3.1±1.6 ^b	0.0±0.0 ^c	1.5±7.5 ^c	9.7±4.9 ^a	0.0±0.0 ^d
Chlorpyrifos	4.7±4.6 ^b	8.0±8.0 ^a	2.5±1.1 ^c	0.0±0.0 ^c	2.2±6.7 ^{bc}	2.0±3.3 ^{bc}	8.0±4.0 ^a
Malathion	8.3±4.3 ^a	8.6±4.3 ^a	1.3±1.0 ^d	0.0±0.0 ^c	2.2±5.7 ^{bc}	2.3±8.8 ^{bc}	1.5±1.5 ^c
Diclorvos	1.3±6.7 ^{cd}	2.7±6.3 ^b	1.3±1.2 ^d	9.1±1.9 ^a	2.2±5.7 ^{bc}	7.3±7.3 ^{ab}	5.3±5.3 ^{ab}
Propoxur	4.3±4.3 ^b	1.1±1.1 ^c	4.3±4.3 ^{ab}	2.4±2.4 ^b	7.3±7.3 ^a	0.0±0.0 ^c	0.0±0.0 ^d
Metalaxyl	0.0±0.0 ^d	0.0±0.0 ^d	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^d
F	6.90	5.50	2.90	10.9	3.86	22.8	13.5
P	0.0002	0.0007	0.0227	<0.0001	0.0057	<0.0001	<0.0001

Level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Gombe State.

The results show that all measured pesticide residue levels (RLs) in the samples were significantly different from each other. And some are higher than the corresponding European Union Maximum Residue Limits (MRLs). The result indicates that, Bifenthrin, Cypermethrin and Propoxur in maize sample in Gombe state sample was 2.3, 2.2 and 4.9 mg/kg respectively, which falls above the EU's maximum allowed limit of 0.05 mg/kg, reflecting a high and unacceptable concentration that is unsafe for consumption (EU Pesticides Database, 2023), while Deltamethrin, Carbaryl, Chlorvos, Chlorpyrifos, Malathion and Metalaxyl recorded zero concentration. In Guinea corn samples analysed it reveals that all the pesticides had also shows higher concentration which exceed the maximum limit level with cypermethrine 2.3 mg/kg, deltamethrine 1.4 mg/kg, carbaryl 1.3 mg/kg, chlorvos 1.7 mg/kg, diclorvos 3.1 mg/kg and propoxur with 3.1 mg/kg while bifenthrin, chlorpyrifos, Malathion and metalaxyl recorded zero concentration.

Furthermore, in ground nut sample the maximum concentration level was recorded on bifenthrin 2.3 mg/kg, cypermethrine 1.4 mg/kg, deltamethrine 1.4 mg/kg, carbaryl 1.6 mg/kg and propoxur 1.7 mg/kg all pesticide concentration exceed the maximum limit level. In cowpea sample all the pesticide analysed shown significant higher concentration which exceed the maximum limit level as set by the European union, with bifenthrin 6.3 mg/kg, cypermethrine 0.1 mg/kg, deltamethrine 4.2 mg/kg, carbaryl 6.7 mg/kg, diclorvos 8.6 mg/kg and metalaxyl 1.7 mg/kg, while chlorvos, chlorpyrifos, Malathion and propoxur shows no concentration level. In Bambara nut all the pesticide analysed only cypermethrine 3.2 mg/kg, carbaryl 2.6 mg/kg and propoxur 2.4 mg/kg shows higher concentration than the maximum limit set, except bifenthrin, deltamethrine, chlorvos, Malathion, diclorvos and metalaxyl shows zero concentration level. Moreover, in rice sample the highest concentration was recorded on cypermethrine 2.5 mg/kg, diclorvos 2.2 mg/kg and 2.4 mg/kg on propoxur, while bifenthrin, deltamethrin, carbaryl, chlorvos, chlorpyrifos, Malathion and metalaxyl shows zero concentration

which are significantly below the maximum limit level. In wheat all the pesticides analysed shows higher maximum limit level with bifenthrin 3.2 mg/kg, cypermethrin 2.8 mg/kg, deltamethrin 2.4

mg/kg, carbaryl 2.7 mg/kg, diclorvovous 2.2 mg/kg, propoxur 2.5 mg/kg except chlorvovous, malathion and metalaxyl which shows zero concentration level (Table 3).

Table 3: Showing the level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Gombe State

Insecticides	Maize	Guinea corn	Ground nut	Cowpea	Bambara nut	Rice	Wheat
Bifenthrin	2.3±8.8 ^b	0.0±0.0 ^d	2.3±8.8 ^a	6.3±8.8 ^b	0.0±0.0 ^c	0.0±0.0 ^b	3.2±3.3 ^a
Cypermethrin	2.2±5.7 ^b	2.3±8.8 ^b	1.4±8.8 ^c	0.1±3.3 ^c	3.2±8.8 ^a	2.5±2.1 ^a	2.8±1.2 ^b
Deltamethrine	0.0±0.0 ^c	1.4±8.8 ^{cd}	1.6±3.3 ^{bc}	4.2±8.8 ^c	0.0±0.0 ^c	0.0±0.0 ^b	2.4±8.8 ^{cd}
Carbaryl	0.0±0.0 ^c	1.3±8.8 ^d	1.6±8.8 ^{bc}	6.7±8.8 ^b	2.6±8.8 ^b	0.0±0.0 ^b	2.7±5.7 ^{bc}
Chlorvos	0.0±0.0 ^c	1.7±5.7 ^c	0.0±0.0 ^d	0.0±0.0 ^f	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^e
Chlorpyrifos	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^d	0.0±0.0 ^f	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^e
Malathion	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^d	0.0±0.0 ^f	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^e
Dichlorvovous	0.0±0.0 ^c	3.1±8.8 ^a	0.0±0.0 ^d	8.6±5.7 ^a	0.0±0.0 ^c	2.2±5.7 ^a	2.2±5.7 ^d
Propoxur	4.9±8.8 ^a	2.2±8.8 ^b	1.7±5.7 ^b	0.0±0.0 ^f	2.4±8.8 ^b	2.4±8.8 ^a	2.5±5.8 ^c
Metalaxyl	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^e	1.2±3.3 ^d	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^e
F	1499	312	326	6482	782	243	601
P	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

Level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Borno State.

The result indicates that maximum limit level were recorded on Bifenthrin 1.3 mg/kg, Cypermethrin 1.7 mg/kg and Propoxur 1.4 mg/kg in maize sample in Borno state. While other pesticide recorded zero concentration. In Guinea corn samples analysed it reveals that all the pesticides had also shows higher concentration where diclorvovous 2.7 mg/kg and propoxur 2.6 mg/kg exceed the maximum limit level, while other recorded zero concentration. Furthermore, in ground nut sample the higher concentration that exceed the maximum limit level was recorded on bifenthrin 1.4 mg/kg, cypermethrin 2.3 mg/kg, deltamethrin 2.2 mg/kg, carbaryl 2.5 mg/kg,

diclorvovous 2.5 mg/kg and propoxur 1.4 mg/kg. In cowpea sample result shows significant higher concentration which exceed the maximum limit level, with cypermethrin 4.4 mg/kg, carbaryl 6.3 mg/kg, chlorvovous 5.3 mg/kg, chlorpyrifos 7.2 mg/kg, malathion 9.1 mg/kg, diclorvovous 6.5 mg/kg and metalaxyl 1.4 mg/kg, while other pesticides shows no concentration level. In Bambara nut, cypermethrin showed 2.5 mg/kg, deltamethrin 1.4 mg/kg, carbaryl 2.5 mg/kg, diclorvovous 2.5 mg/kg and propoxur 1.4 mg/kg. Moreover, in rice sample the highest concentration was recorded on cypermethrin 1.4 mg/kg, deltamethrin 1.4 mg/kg, diclorvovous 1.4 mg/kg and propoxur 1.3 mg/kg. In wheat, higher than maximum limit level were recorded on cypermethrin 2.3 mg/kg, diclorvovous 1.4 mg/kg and propoxur 1.2 mg/kg (Table 4).

Table 4: Showing the level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Borno State

Insecticides	Maize	Guinea corn	Ground nut	Cowpea	Bambara nut	Rice	Wheat
Bifenthrin	1.3±8.8 ^b	0.0±0.0 ^b	1.4±8.7 ^{bc}	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^d
Cypermethrin	1.7±5.7 ^a	0.0±0.0 ^b	2.3±5.7 ^{ab}	4.4±1.2 ^c	2.5±1.2 ^a	1.4±8.8 ^a	2.3±5.7 ^a
Deltamethrine	0.0±0.0 ^c	0.0±0.0 ^b	2.2±5.7 ^b	0.0±0.0 ^c	1.4±8.8 ^b	1.4±1.2 ^a	0.0±0.0 ^d
Carbaryl	0.0±0.0 ^c	0.0±0.0 ^b	2.5±1.2 ^a	6.3±0.0 ^{ab}	2.5±1.2 ^a	0.0±0.0 ^c	0.0±0.0 ^d
Chlorvos	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^c	5.3±1.2 ^c	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^d
Chlorpyrifos	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^c	7.2±5.7 ^{ab}	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^d
Malathion	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^c	9.1±3.3 ^a	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^d
Diclorvovous	0.0±0.0 ^c	2.7±3.3 ^a	2.5±1.2 ^a	6.5±5.7 ^b	2.5±1.1 ^a	1.4±3.3 ^a	1.4±3.3 ^b
Propoxur	1.4±0.0 ^b	2.6±8.8 ^a	1.4±8.8 ^c	0.0±0.0 ^c	1.4±8.8 ^b	1.3±5.7 ^b	1.2±0.0 ^c
Metalaxyl	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^c	1.4±8.8 ^d	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^{ds}
F	455	1441	286	7.41	242	203	1588
P	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

Level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Bauchi State.

The result indicates that Bifenthrin 1.3 mg/kg, Cypermethrin 1.7 mg/kg and Propoxur 1.4 mg/kg in maize sample in Bauchi state which falls above the EU's maximum allowed limit of 0.05 mg/kg. While Deltamethrin, Carbaryl, Chlorvos, Chlorpyrifos, Malathion, dichlorvos and Metalaxyl recorded zero traces of pesticide contamination. In Guinea corn samples analysed it reveals that diclorvos 2.8 mg/kg and propoxur 2.6 mg/kg shows higher concentration level which exceed the maximum limit level while bifenthrin, cypermethrine, deltamethrine, carbaryl, chlorpyrifos, Malathion and metalaxyl recorded no any concentration level. Furthermore, in ground nut sample the higher maximum concentration level which is above the maximum limit level were recorded on cypermethrine 2.5 mg/kg, deltamethrine 1.4 mg/kg, carbaryl 2.5 mg/kg, diclorvos 2.5 mg/kg and propoxur 1.4 mg/kg all pesticide

concentration exceed the maximum limit level. In cowpea sample all the pesticide analysed shown significant higher concentration which exceed the maximum limit level are bifenthrin 0.1 mg/kg, cypermethrine 4.4 mg/kg, carbaryl 6.3 mg/kg, chlorvos 5.3 mg/kg, chlorpyrifos 7.2 mg/kg, malathion 9.1 mg/kg and metalaxyl 1.5 mg/kg. In Bambara nut the following pesticide shows higher percentage of concentration level above the maximum limit levels cypermethrine 3.2 mg/kg, carbaryl 2.6 mg/kg and propoxur 2.4 mg/kg shows higher concentration than the maximum limit, except bifenthrin, deltamethrine, chlorvos, Malathion, diclorvos and metalaxyl had shown zero concentration level. Moreover, in rice sample the highest concentration was recorded on cypermethrine 1.4 mg/kg, deltamethrin 1.4 mg/kg, diclorvos 1.4 mg/kg and 1.3 mg/kg on propoxur. In wheat higher than maximum limit level were recorded on cypermethrin 2.3 mg/kg, diclorvos 1.2 mg/kg and propoxur 1.2 mg/kg (Table 5).

Table 5: Showing the level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Bauchi State

Insecticides	Maize	Guinea corn	Ground nut	Cowpea	Bambara nut	Rice	Wheat
Bifenthrin	1.3±8.8 ^b	0.0±0.0 ^c	0.0±0.0 ^c	0.1±3.3 ^c	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^d
Cypermethrin	1.7±5.8 ^a	0.0±0.0 ^c	2.5±1.1 ^a	4.4±1.2 ^c	3.2±8.8 ^a	1.4±8.8 ^a	2.3±5.8 ^a
Deltamethrine	0.0±0.0 ^c	0.0±0.0 ^c	1.4±8.8 ^b	0.0±0.0 ^f	0.0±0.0 ^c	1.4±1.1 ^a	0.0±0.0 ^d
Carbaryl	0.0±0.0 ^c	0.0±0.0 ^c	2.5±1.1 ^a	6.3±0.0 ^f	2.6±8.8 ^b	0.0±0.0 ^b	0.0±0.0 ^d
Chlorvos	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^c	5.3±0.0 ^b	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^d
Chlorpyrifos	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^c	7.2±5.7 ^{ab}	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^d
Malathion	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^c	9.1±3.3 ^a	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^d
Diclorvos	0.0±0.0 ^c	2.8±5.7 ^a	2.5±1.1 ^a	0.0±0.0 ^f	0.0±0.0 ^c	1.4±8.8 ^a	1.4±3.3 ^b
Propoxur	1.4±0.0 ^b	2.6±5.7 ^b	1.4±8.8 ^b	0.0±0.0 ^f	2.4±8.8 ^b	1.3±5.7 ^{ab}	1.2±0.0 ^c
Metalaxyl	0.0±0.0 ^c	0.0±0.0 ^c	0.0±0.0 ^c	1.5±8.8 ^d	0.0±0.0 ^c	0.0±0.0 ^b	0.0±0.0 ^d
F	455	1947	243	11.2	782	157	1588
P	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

Level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Adamawa State.

The results shows that all measured pesticide residue levels (RLs) in the samples were significantly different from each other. And some are higher than the corresponding European Union Maximum Residue Limits (MRLs). The result indicates that, Bifenthrin 2.2 mg/kg, Cypermethrin 2.7 mg/kg, deltamethrin 2.2 mg/kg, carbaryl 1.8 mg/kg, chlorvos 3.4 mg/kg, chlorpyrifos 7.3 mg/kg diclorvos 2.5 mg/kg and Propoxur 3.4 mg/kg in maize sample in Adamawa state, which

falls above the EU's maximum allowed limit of 0.05 mg/kg, reflecting a high concentration levels while Malathion and Metalaxyl recorded zero concentration. In Guinea corn samples analysed it reveals that only cypermethrin 2.2 mg/kg, diclorvos 1.7 mg/kg and propoxur 1.6 mg/kg had shown the higher concentration level which exceed the maximum limit level. In ground nut sample the maximum concentration level was recorded on bifenthrine 1.5 mg/kg, cypermethrine 1.4 mg/kg, carbaryl 1.7 mg/kg, diclorvos 2.0 mg/kg and propoxur 3.3 mg/kg all exceed the maximum limit level. In cowpea sample all the pesticide analysed

shown significant higher concentration which exceed the maximum limit level as set by the European union, with bifenthrin 0.1 mg/kg, cypermethrine 8.3 mg/kg, deltamethrine 1.6 mg/kg, carbaryl 3.5 mg/kg, chlorvos 7.3 mg/kg, diclorvos 6.6 mg/kg and propoxur 8.3 mg/kg, while chlorpyrifos, Malathion and metalaxyl have shown no concentration level. In Bambara nut only cypermethrine 3.4 mg/kg and carbaryl 4.3 mg/kg shows higher concentration than the maximum limit set, except bifenthrin, deltamethrine, chlorvos, chlorpyrifos, Malathion, diclorvos, propoxur and metalaxyl shows no level of

concentration. In rice the highest concentration was recorded on bifenthrin 3.2 mg/kg, cypermethrine 2.4 mg/kg, carbaryl 2.4 mg/kg, chlorvos 2.8 mg/kg, diclorvos 2.7 mg/kg and 1.5 mg/kg on propoxur, while deltamethrin, chlorpyrifos, Malathion and metalaxyl shows zero or no concentration level which are significantly less than the maximum limit level. In wheat the higher maximum limit level were recorded with cypermethrin 1.6 mg/kg, chlorvos 4.5 mg/kg, dichlorvos 1.5 mg/kg and propoxur 1.3 mg/kg (Table 6).

Table 6: Showing the level of pesticide residues in comparison with EU MRL from samples of cereals and leguminous grains collected in Adamawa State

Insecticides	Maize	Guinea corn	Ground nut	Cowpea	Bambara nut	Rice	Wheat
Bifenthrin	2.2±8.8 ^{cd}	0.0±0.0 ^d	1.5±8.8 ^{cd}	0.1±5.8 ^c	0.0±0.0 ^c	3.2±3.3 ^a	0.0±0.0 ^d
Cypermethrin	2.7±1.2 ^c	2.2±5.7 ^a	1.4±1.1 ^d	8.3±5.7 ^a	3.4±1.5 ^b	2.4±8.8 ^{bc}	1.6±5.7 ^b
Deltamethrine	2.2±5.7 ^{cd}	0.0±0.0 ^d	0.0±0.0 ^e	1.6±8.8 ^{cd}	0.0±0.0 ^c	0.0±0.0 ^e	0.0±0.0 ^d
Carbaryl	1.8±1.7 ^{de}	0.0±0.0 ^d	1.7±8.8 ^{bc}	3.5±1.2 ^d	4.3±5.7 ^a	2.4±1.2 ^c	0.0±0.0 ^d
Chlorvos	3.4±8.8 ^b	0.0±0.0 ^d	0.0±0.0 ^e	7.3±1.1 ^b	0.0±0.0 ^c	2.8±5.7 ^b	4.5±5.7 ^a
Chlorpyrifos	7.3±1.2 ^a	0.0±0.0 ^d	0.0±0.0 ^e	0.0±0.0 ^e	0.0±0.0 ^c	0.0±0.0 ^e	0.0±0.0 ^d
Malathion	0.0±0.0 ^f	0.0±0.0 ^d	0.0±0.0 ^e	0.0±0.0 ^e	0.0±0.0 ^c	0.0±0.0 ^e	0.0±0.0 ^d
Diclorvos	2.5±8.8 ^{cd}	1.9±5.7 ^b	2.0±5.7 ^b	6.6±3.3 ^c	0.0±0.0 ^c	2.7±8.8 ^c	1.5±0.0 ^b
Propoxur	3.4±8.8 ^b	1.6±3.3 ^c	3.3±8.8 ^a	8.3±8.8 ^a	0.0±0.0 ^c	1.5±1.2 ^d	1.3±3.3 ^c
Metalaxyl	0.0±0.0 ^c	0.0±0.0 ^d	0.0±0.0 ^e	0.0±0.0 ^e	0.0±0.0 ^c	0.0±0.0 ^e	0.0±0.0 ^d
F	455	1119	336	2837	1005	396	2694
P	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

DISCUSSION

The analysis revealed that all grain samples collected from markets across the northeastern states contained four main classes of pesticides - organochlorines, organophosphates, carbamates, and pyrethroids, consistent with recent evidence that multiple pesticide classes co-occur in Nigerian grains. This finding agrees with reports by Anaduaka *et al.*, (2023) and Omeje *et al.*, (2022), who documented the extensive use of pesticides in Nigerian agriculture. They noted that although pesticides are essential for crop protection, improper handling, excessive application, and poor adherence to safety guidelines contribute to residue accumulation in food products. Fadina *et al.*, (2021) similarly detected measurable concentrations of organochlorines and organophosphates in grains and other crops, emphasizing the need for continuous monitoring and stronger regulation. More recently, a review on pesticide residues in Nigerian food grains in 2025 underscored that residues in cereal grains remain a major public health challenge despite existing laws and regulatory agencies, largely due to weak enforcement, limited analytical capacity, and low awareness among farmers and traders. In southern Nigeria, Omokpariola *et al.*, (2023) reported

significant organochlorine and organophosphate residues in cereals and legumes, while Asogwa *et al.*, (2023) quantified high insecticide residues in maize from southeastern markets. Collectively, these studies show that pesticide contamination of grains is widespread across multiple regions of Nigeria and persists into the mid-2020s Sulaiman and Muazu, 2025; Oshatunberu *et al.*, 2023; . Abare-Jen *et al.*, 2024).

The presence of pesticide residues in grains raises important public health concerns. Chronic exposure to these chemicals has been linked to cancer, reproductive disorders, and neurological problems (Sabarwal *et al.*, 2018), and reviews of more recent epidemiological data confirm associations with endocrine disruption, immune dysfunction, and adverse developmental outcomes. Vulnerable populations such as children and pregnant women are particularly at risk (Omeje *et al.*, 2022). The high concentrations observed across all pesticide classes in the present study may reflect the high susceptibility of dry grains, especially legumes, to weevil infestation during storage. Due to inadequate training and limited awareness of safe pesticide use, grain dealers frequently resort to repeated and sometimes excessive applications in an attempt to preserve grain quality and prevent

postharvest losses. As a result, residues often exceed permitted limits, as reported by Mohammed (2019) and confirmed by more recent residue analyses in stored maize and beans from northern Taraba and other Nigerian states, where many measured levels were above European Union MRLs (Oliveira *et al.*, 2025; Sulaiman and Muazu, 2025).

Several factors may account for the elevated residue concentrations observed in leguminous grains in the present study. These include multiple treatments over time, overlapping or similar active ingredients in different formulations, cross-contamination among stored lots, and broader environmental contamination of production areas (Nibbering, 2023). Recent investigations in southwest and north-central Nigeria have also shown that banned or severely restricted compounds, such as some organochlorines and highly toxic organophosphates, still occur in grains and soils, indicating ongoing misuse or legacy contamination. In the present work, all detected residues exceeded their respective Maximum Residue Limits (MRLs). An MRL is the highest legally permitted concentration of a pesticide residue in food that is considered safe when the compound has been applied according to good agricultural practice; any exceedance renders the food unsafe for consumption (Ogwo, 2021). Recent health risk assessments of organochlorine and organophosphorus residues in grains have reported hazard quotients and indices greater than 1 for some commodities, confirming that dietary exposure can pose substantial non-carcinogenic and carcinogenic risks to consumers (Akinwola *et al.*, 2025; Ekwoba *et al.*, 2026).

From a regulatory standpoint, Nigeria has established MRLs and a regulatory framework for pesticide registration, distribution, and use, intended to protect public health by ensuring that marketed food is safe. However, 2025 evidence indicates that pesticide residues in grains remain a major challenge due to weak enforcement, fragmented institutional responsibilities, limited testing capacity, and inadequate monitoring of markets. Effective surveillance programs - based on regular sampling and laboratory analysis of grains and other staple foods - are therefore critical to ensure compliance with safety standards and to guide risk management. Recent health-risk and residue studies in grains from different Nigerian regions have repeatedly recommended intensified routine monitoring, stricter enforcement of bans on highly hazardous pesticides, and targeted education of farmers, traders, and grain merchants. Likewise, recent reviews on pesticide residues in food globally emphasize integrating mitigation strategies—such as improved storage practices, adoption of safer alternatives, and post-harvest processing techniques that reduce residues—into

food safety policies (Abare-Jen *et al.*, 2024; Oliveira *et al.*, 2025; Ekwoba *et al.*, 2026).

The urgency of investigating pesticide residues in grains sold in the northeastern states stems from several interrelated factors. First, cereals and legumes are staple foods in Nigeria, and in the northeast they constitute a major component of daily diets, making any contamination a direct threat to food and nutrition security. Earlier studies, including those by Fadina *et al.*, (2021) and Hajam and Kumar (2022), demonstrated the presence of pesticide residues in grain samples from Nigerian markets, indicating potential chronic exposure among consumers. However, there is still a notable gap in comprehensive, region-specific data on pesticide contamination of cereals and legumes in northeastern Nigeria, in contrast to the growing body of work from southwest, north-central, and other regions. Recent risk-assessment studies carried out between 2024 and 2026 show that organochlorine and organophosphorus residues in grains can contribute significantly to cumulative dietary risk, underscoring the need for continuous environmental and food monitoring. Furthermore, chronic exposure to pesticide residues has been implicated in cancer, reproductive and endocrine disorders, and neurological problems (Sabarwal *et al.*, 2018; Upadhyay *et al.*, 2020), with newer reviews reinforcing these links and calling for stronger preventive measures. Given the high and increasing consumption of grains in the region, generating robust data on residue levels in cereals and legumes is essential for evidence-based regulation, targeted risk communication, and the development of safer pest-management strategies that protect both public health and food security.

CONCLUSION AND RECOMMENDATIONS

The results from this study show that grains sample being sold at the market contains pesticide residue in high concentrations. Most of the residues tested were found to be above maximum residue limits which are dangerous to human health. Organophosphate and pyrethroids are recommended for crop protection in Nigeria however strict monitoring and control, enlightenment, training and enforcement is needed by the appropriate agencies. Therefore, the data we have, needs to be updated periodically as pesticide use improves. Organophosphate and pyrethroids are recommended for crop protection in Nigeria however strict monitoring and control, enlightenment, training and enforcement is needed by the appropriate agencies. The use of natural protectants such as the diatomaceous earth patented by Nigerian Stored Products Research Institute which is a non-toxic, biodegradable, inert dust was tested against insect pests in storage by should be encouraged; the trials results were very satisfactory.

ACKNOWLEDGEMENT

The authors gratefully acknowledge the support of the Tertiary Education Trust Fund (TETFund), Nigeria, for funding this research through the Institutional Based Research (IBR) grant No:TETF/DR&D/CE/UNI/BORNO/IBR/2024/VO L 1. The funding provided by TETFund was instrumental in facilitating the successful execution of this study.

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