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Assessment of Some Selected Pesticide Residues and Associated Soils from Six Farming Communities of Girei Local Government Area, Adamawa State, Nigeria

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Abstract

This study assessed the concentrations of pesticide residues in leguminous crops and associated soils from six farming communities in Girei Local Government Area, Adamawa State, Nigeria. Sampling was conducted in Tashan Turmi, Wuro Dole, Madugu, Sebere, Bakalci, and Jabi Lamba. Standard analytical procedures were used, with pesticide residue analysis performed using a Gas Chromatograph-Mass Spectrometer (GC-MS), Agilent Technologies GC 7890B, MSD 5977A. In legume samples, detected pesticide residues included atrazine (0.93 $\mu g \cdot kg^{-1}$), 2,4-D (0.275 $\mu g \cdot kg^{-1}$), cypermethrin (0.066 $\mu g \cdot kg^{-1}$), and fenitrothion (0.051 $\mu g \cdot kg^{-1}$), all of which were below Codex Maximum Residue Limits (MRLs). In soil samples, higher concentrations were observed, with 2,4-D at 86.361 $\mu g \cdot kg^{-1}$, dimethoate at 1.009 $\mu g \cdot kg^{-1}$, alachlor at 3.382 $\mu g \cdot kg^{-1}$, and atrazine at 0.857 $\mu g \cdot kg^{-1}$. These results indicate moderate environmental contamination due to agrochemical usage, reflecting anthropogenic influence in the agricultural practices of the region. The study underscores the need for regular pesticide monitoring, the adoption of safer agricultural practices, and increased public awareness to ensure food safety and safeguard environmental health.

Keywords: Pesticide Residues; Contamination; Leguminous Crops; Farm Soil; Food Safety

INTRODUCTION

The twenty-first century has witnessed an unparalleled increase in both agriculture and industrialization, which have significantly transformed the geochemical cycling of toxic elements and xenobiotics (Hryhorczuk *et al.,* 2018; Kalisińska, 2019). Pesticide residues refer to the small amounts of pesticides that remain on agricultural products after they have been applied, which includes both the active substances and their breakdown products. Extensive use of synthetic pesticides especially historically prevalent organochlorines (OCs), the highly toxic organophosphates (OPs), and the increasingly favored pyrethroids has introduced a second category of contaminants into agro-ecosystems (Arif *et al.,* 2018).

Pesticides can worsen these effects through additive or synergistic reactions; for instance, co-exposure to Pb and chlorpyrifos has been shown to amplify neurobehavioral issues in animal studies (Constantinescu *et al.,* 2025). Pyrethroids have been linked to endocrine disruption, immune toxicity, and a heightened risk of acute lymphoblastic leukemia (Sharma *et al.,* 2019). The Joint FAO/WHO Expert Committee on Food Additives (JECFA) has established provisional tolerable weekly intake levels for Pb (25 μg kg^{-1} bw), Cd (7 μg kg^{-1} bw), and Hg (5 μg kg^{-1} bw), but dietary surveys in Nigeria reveal that more than 60% of urban adults exceed these guidelines (WHO, 2019).

Organophosphates (like chlorpyrifos, diazinon, and malathion) and pyrethroids (such as cypermethrin, bifenthrin, and deltamethrin) have largely taken the place of organochlorines due to their comparatively short environmental half-lives (ranging from days to months). Nonetheless, organophosphates are acutely toxic because they inhibit cholinesterase, while pyrethroids have significant aquatic toxicity and possible endocrine-disrupting effects (Zhang *et al.,* 2024; Ehsanifar *et al.*, 2025). Legumes, especially cowpea (Vigna unguiculata), groundnut (Arachis hypogaea), soybean (Glycine max), and pigeon pea (Cajanus cajan), play a crucial role in the diets of Nigerians, providing over 40% of the population's plant-based protein and serving as a significant export product (FAO, 2020).

MATERIALS AND METHODS

Study Area

Girei Local Government Area (LGA) is one of the 21 LGAs located in Adamawa State, Nigeria. Positioned at approximately 9°22'N latitude and 12°33'E longitude, it lies in the northeastern part of the state and shares boundaries with Yola North, Yola South, and Fufore LGAs. Covering a landmass of about 4,800 square kilometers, Girei's terrain includes flat plains, undulating hills, and fertile valleys. It is intersected by several rivers, with the Benue River being the most prominent, contributing to the area's agricultural viability. The climate is tropical and marked by two seasons the wet season from May to October and the dry season from November to April offering conducive conditions for farming activities year-round.

The population of Girei LGA was estimated at 270,000 during the 2006 census, although current figures are likely higher due to growth in settlement patterns, driven in part by the presence of key institutions. Communities within the LGA are predominantly rural, with agriculture as the mainstay of the local economy. Ethnic groups such as the Fulani, Hausa, and Bachama coexist in the area, contributing to its cultural diversity.

Agriculture in Girei is anchored on crop cultivation and livestock rearing. Staples like maize, sorghum, millet, and particularly leguminous crops such as cowpeas (beans) and groundnuts are widely cultivated. Beans are consumed heavily in Girei, forming the base for traditional breakfast meals like akara and moi moi. Groundnuts are similarly important, with high oil and protein content, making them valuable for both local consumption and market trade. These leguminous crops are intercropped with cereals, enhancing food security and soil health.

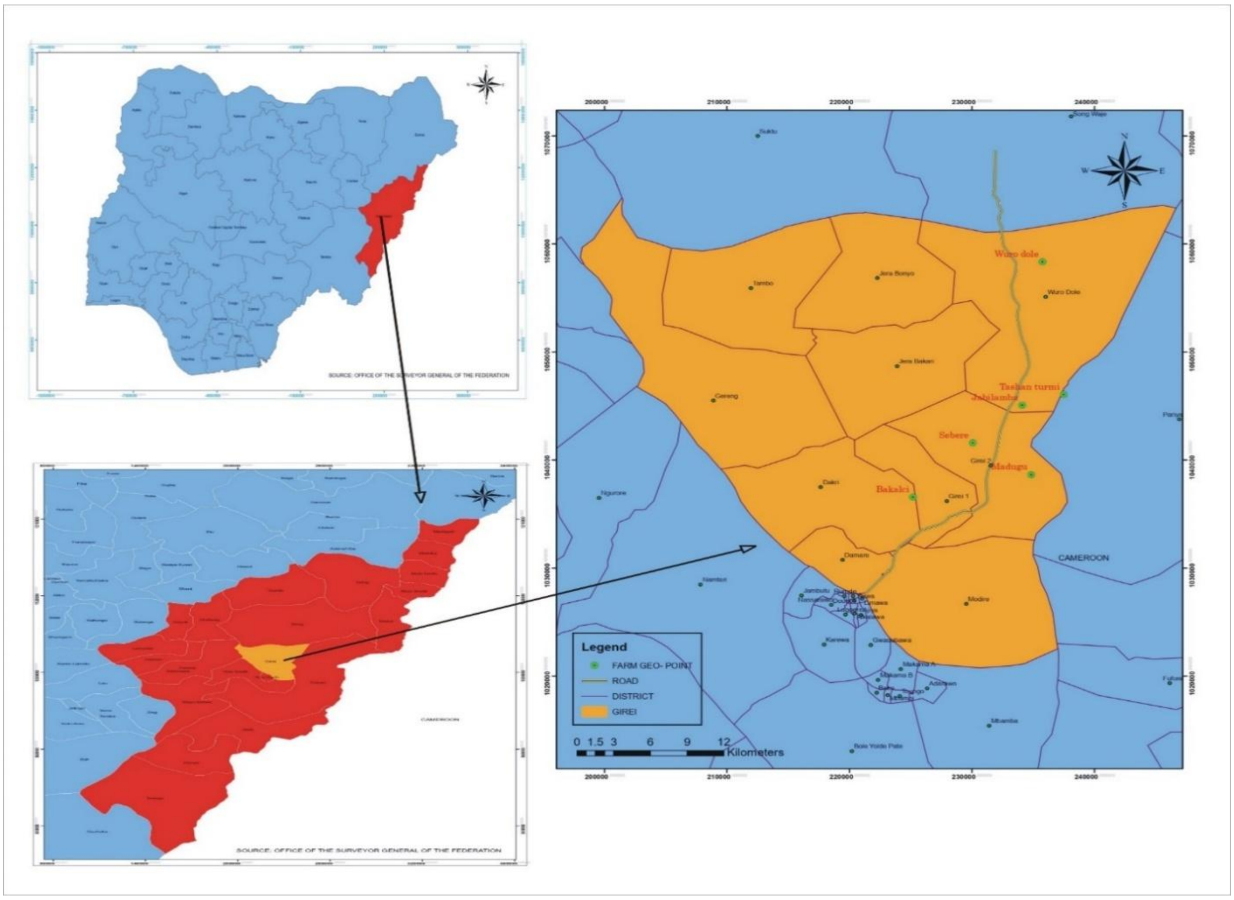

Fig. 1 Map of Girei L.G.A indicating Sample Collection Areas, (Source: OSGOF)

Table. 1 Selected farmlands and method of coding for collected samples

CODE	Beans	Groundnut	Soil	LOCATION	LAT	LONG
FARM 1	BF1	GF1	SF1	Tashan Turmi	9.454853	12.609186
FARM 2	BF2	GF2	SF2	Wuro Dole	9.387776	12.585641
FARM 3	BF3		SF3	Madugu	9.565719	12.59266
FARM 4	BF4		SF3	Sebere	9.413993	12.541895
FARM 5		GF5	SF5	Bakalci	9.368428	12.49783
FARM 6		GF6	SF6	Jabilamba	9.445682	12.578308

Survey method for soil collection

A survey was conducted to identify farmers who had cultivated leguminous crops, specifically groundnut and cowpea, during the previous growing season in Girei Local Government Area, Adamawa State. Farmers capable of providing these crops and guiding researchers to the exact cultivation sites were selected for participation. A total of six farms were chosen for the study. Among these, some farms had both cowpea and groundnut cultivated together, while others had either cowpea or groundnut cultivated separately.

Collection and Pretreatment of Samples

Crop samples were collected from the same farms associated with the soil samples. The soil samples from the various sampling sites were taken at a depth of 0.5 to 0.25 cm depth each with the help of plastic Parker wash with clean water and allow to dry. The crop samples were placed in airtight containers to preserve their integrity and were labeled accordingly to maintain alignment with the respective soil sample identifiers and geographical data points. Geo-location information for each farm was recorded to ensure accurate traceability.

Pesticides Determination (crop samples)

Extraction Process

Each 0.2 g sample was weighed and immersed in 20 ml of dichloromethane (DCM) within a 250 ml amber bottle, then left to stand for 72 hours. After this period, the resulting solution was filtered.

Clean Up/ Pre-Concentration

Following concentration, the dried residue was reconstituted in an appropriate solvent typically hexane or acetone depending on the target analytes and the requirements of subsequent instrumental analysis. This reconstituted solution was then transferred into clean, labeled vials and stored under controlled conditions to prevent degradation or contamination prior to analysis. The prepared samples were subsequently subjected to chromatographic evaluation, ensuring that all instrumental parameters were optimized for accurate detection and quantification of the analytes of interest (Oyekunle *et al.,* 2017).

GC-MS Analysis (crop samples)

Prior to analysis, the GC-MS system (Agilent Technologies GC 7890B, MSD 5977A) was thoroughly calibrated using stock solutions of known concentrations of organochlorine pesticides. These standard solutions were injected under predefined chromatographic conditions, and the resulting mean peak areas were plotted against their respective concentrations to generate calibration curves for each pesticide compound. Once calibration was confirmed, the concentrated extracts from the bean samples were introduced into the GC-MS for the determination of organochlorine pesticide residues. The instrument's sensitivity and selectivity enabled precise quantification of the target analytes based on retention times and mass spectral data.

Soil Sample Analysis

Extraction of soil

The QuEChERS extraction procedure followed the standards outlined by the European Committee for Standardization (2018). A 5-gram quantity of the homogenized sample was placed into a 50 mL polypropylene centrifuge tube. To this, 10 mL of acetonitrile (MeCN) was added, and the tube was vortexed for 30 seconds. Next, 1 g of sodium chloride (NaCl) and 4 g of anhydrous magnesium sulfate ($MgSO_4$) were introduced, and the mixture was immediately agitated using a vortex mixer for 60 seconds to avoid clumping of the $MgSO_4$. The sample was then centrifuged at 2,900 × g for 5 minutes. A 3 mL portion of the acetonitrile layer was transferred into a 15 mL microcentrifuge tube containing 600 mg of anhydrous $MgSO_4$ and 120 mg of Primary Secondary Amine (PSA). This mixture was vortexed again for 30 seconds and centrifuged at 4,000 rpm for 5 minutes. Finally, 1 mL of the supernatant was filtered through a 0.2 μm PTFE membrane and transferred into a clean vial for subsequent injection.

RESULTS AND DISCUSSION

Pesticide residues levels in Leguminous Crops and Soil samples

The pesticide residue concentrations obtained from leguminous crop (beans and groundnut) and soil samples across six communities in Girei LGA, Adamawa State were studied. The result of the levels of these pesticide residues in Leguminous crops and soil samples were compared with findings against international standards, review similar regional studies, and assess health and environmental implications. The pesticides residues vary in concentrations in all the locations as presented in Table 2 and 3 below.

Pesticide residues levels in Leguminous Crops

From Table 2 beans farms (BF-series) and groundnut farms (GF-series) yielded detectable residues of dichlorvos, atrazine, 2,4-D, clopyralid, triclopyr, alachlor, cypermethrin, and fenitrothion. The highest values were recorded for atrazine in BF3 (Sebere) at 0.93 μg/kg, 2,4-D in GF6 (Jabi Lamba) at 0.275 μg/kg, cypermethrin in BF3

(Madugu) at 0.066 µg/kg, and fenitrothion in BF2 (Wuro Dole) at 0.051 µg/kg. Other sites largely showed no residues or below 0.05 µg/kg, indicating heterogeneous application practices and variable degradation rates across communities. According to Codex Alimentarius, maximum residue limits (MRLs) for pulses are 1,000 µg/kg for dichlorvos and 50 µg/kg for atrazine, 2,4-D, and alachlor (FAO/WHO, 2019). All detected residues in Girei crops fall well below these thresholds. The atrazine residue at BF3 (0.93 µg/kg) in (Sebere) location was 50 times below its 50 µg/kg MRL, and 2,4-D at GF6 (0.275 µg/kg) in Jabi Lamba location was 180 times below its limit (Table 2). These results reflect low dietary risk from single compound exposure (FAO/WHO, 2019). Abubakar *et al.* (2024) detected obsolete OCPs in 30 % of cereals and legumes in northwest Nigeria, frequently exceeding Codex limits. Similarly, Olutona and Aderemi (2019) found hexachlorobenzene medians at 25 µg/kg in Ibadan cowpea 2.5 times the 10 µg/kg MRL and dieldrin up to 20,000 µg/kg. The study from Girei samples locations showed negligible OCP levels, suggesting a phase-out of banned compounds or rapid degradation under the region's high UV and microbial activity (Ezeani *et al.,* 2022).

Chronic exposure to mixtures even at sub-MRL levels can pose cumulative risks. Atrazine is linked to endocrine disruption and reproductive toxicity, while 2,4-D is a Group 2B possible human carcinogen (Hayes *et al.,* 2011; IARC, 2015). Although single compound hazard quotients remain < 0.01, combined exposures through daily legume consumption may increase endocrine and neurodevelopmental risks, especially in children (Beyuo *et al.,* 2024).

Table 2: Concentration of Pesticide residues in leguminous Crops samples from Gerei LGA (µg/kg)

				Sample I.D					
Pesticides (µg/kg)	**BF1** Tashan Turmi	**BF2** Wuro Dole	**BF3** Madugu	**BF4** Sebere	**GF1** Tashan Turmi	**GF2** Wuro Dole	**GF5** Bakalci	**GF6** Jabi Lamba	**RT (min)**
Dichlorvos	0.00	0.050	0.076	0.00	0.000	0.00	0.032	0.007	5.0-6.0
Clopyralid	0.00	0.000	0.000	0.00	0.027	0.00	0.000	0.000	6.0-7.0
Triclopyr	0.00	0.000	0.000	0.00	0.000	0.00	0.044	0.000	7.0-7.3
Phenol, 2,4-dichloro-	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	7.3-7.4
Dimethoate	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	7.4-7.7

				Sample I.D					
Methomyl	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	7.7-7.9
Atrazine	0.25	0.000	0.000	0.93	0.000	0.00	0.000	0.000	8.0-8.2
2,4-D	0.00	0.240	0.204	0.00	0.000	0.00	0.000	0.275	8.2-9.0
Alachlor	0.00	0.000	0.000	0.00	0.026	0.03	0.000	0.000	10.0-10.9
Butachlor	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	16.8-17.3
Lindane (γ-HCH)	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	17.3-17.5
Fenitrothion	0.00	0.051	0.000	0.00	0.000	0.00	0.000	0.000	17.5-17.7
Profenofos	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	17.7-17.9
Malathion	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	17.9-18.2
Terbufos	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	18.2-18.5
Heptachlor	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	18.5-18.7
Chlorpyrifos	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	18.7-18.9
Aldrin	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	18.9-19.0
Endosulfan (α)	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	19.0-19.3
Methoxychlor	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	19.4-19.7
Cypermethrin	0.00	0.000	0.066	0.00	0.000	0.00	0.000	0.000	19.7-20.5
Cyhalothrin (λ-)	0.00	0.000	0.000	0.00	0.000	0.00	0.000	0.000	21.5-21.9

Pesticide residues levels in Soil samples

Table 3 shows soils samples from SF1–SF6 contained detectable levels of 2,4-D, dimethoate, triclopyr, alachlor, atrazine, butachlor, dichlorvos, and lindane. The highest concentration was 2,4-D at SF2 (Wuro Dole) with 86.361 µg/kg, followed by dimethoate at SF3 (Madugu) at 1.009 µg/kg. Other sites showed mixed but generally lower residues (< 4 µg/kg). USEPA sets 100 µg/kg for atrazine and 7.5 µg/kg for alachlor while the values obtained from this study area had (0.857 µg/kg and 3.382 µg/kg, respectively) which fall within these bounds (USEPA, 2002, 2006). Dichlorvos and dimethoate residues (< 1.01 µg/kg) are well below thresholds linked to soil biota toxicity (> 5 mg/kg), indicating low acute ecological risk but possible chronic effects. Atrazine was only observed in SF3 Madugu location (low risk), suggesting potential transient impact on soil microbial nitrifiers and nitrogen fixing organisms (Silva *et al.,* 2024). Aldrin is an extremely persistent pollutant which does not easily degrade in the environment but tend to biomagnified when it enters the food chain. When ingested, it causes headache, dizziness, and vomiting. It was also found that this chemical can remain in the soil for decade and accumulate in agricultural produce and is unsuitable to humans (Momohshaibu et *al*., 2022).

Organophosphates detected in beans likely result from late season applications by farmers in Northern Nigeria often harvest within 1–5 days' post-spray, violating recommended 7-day pre-harvest intervals and elevating crop residues (Yami *et al.,* 2025). Despite individual residues falling below single-compound MRLs, chronic co-exposure to multiple chemicals may pose cumulative risks. Beyuo *et al.* (2024) emphasize that mixtures of phenoxy acids, triazines and organophosphates even at sub-MRL levels interact synergistically to disrupt endocrine and neurodevelopmental pathways over long-term intake. Onwudiegwu *et al.* (2025) further warn that soil-borne residues migrate through runoff and uptake, compounding human and ecological exposure in subsistence farming systems.

Table 3: Concentration of Pesticide residues in Soil samples from Gerei LGA (µg/kg)

Pesticides Residue (µg/kg)	SF1 Tashan Turmi	SF2 Wuro Dole	SF3 Sebere	SF3 Madugu	SF5 Bakalci	SF6 Jabi Lamba	RT (min)
Dichlorvos	0.064	0.000	0.068	0.000	0.203	0.029	5.0-6.0
Clopyralid	0.000	0.000	0.045	0.000	0.000	0.000	6.0-7.0
Triclopyr	0.000	0.000	0.000	0.000	0.250	0.000	7.0-7.3
Phenol, 2,4-dichloro-	0.000	0.000	0.000	0.000	0.000	0.000	7.3-7.4
Dimethoate	0.054	0.000	0.000	1.009	0.000	0.000	7.4-7.7
Methomyl	0.000	0.000	0.000	0.000	0.000	0.000	7.7-7.9
Atrazine	0.000	0.000	0.000	0.857	0.000	0.000	8.0-8.2
2,4-D	0.000	86.361	1.715	4.286	1.270	0.000	8.2-9.0
Alachlor	0.000	0.000	0.000	3.382	0.876	0.000	10.0-10.9
Butachlor	0.000	0.000	0.572	0.000	0.000	0.050	16.8-17.3
Lindane (γ-HCH)	0.038	0.000	0.000	0.000	0.000	0.000	17.3-17.5
Fenitrothion	0.000	0.000	0.000	0.000	0.000	0.000	17.5-17.7
Profenofos	0.000	0.000	0.000	0.000	0.000	0.000	17.7-17.9
Malathion	0.000	0.000	0.000	0.000	0.000	0.000	17.9-18.2
Terbufos	0.000	0.000	0.000	0.000	0.000	0.000	18.2-18.5
Heptachlor	0.000	0.000	0.000	0.000	0.000	0.000	18.5-

							18.7
Chlorpyrifos	0.000	0.000	0.000	0.000	0.000	0.000	18.7-18.9
Aldrin	0.000	0.000	0.000	0.000	0.000	0.000	18.9-19.0
Endosulfan (α)	0.000	0.000	0.000	0.000	0.000	0.000	19.0-19.3
Methoxychlor	0.000	0.000	0.000	0.000	0.000	0.000	19.4-19.7
Cypermethrin	0.000	0.000	0.000	0.000	0.000	0.000	19.7-20.5
Cyhalothrin (λ-)	0.000	0.000	0.000	0.000	0.000	0.000	21.5-21.9

CONCLUSION

Pesticide residues in both crops and soils are low and within international safety margins, indicating responsible application practices or efficient degradation. Nonetheless, the co-occurrence of multiple agrochemicals even at sub-MRL levels suggests potential additive or synergistic effects on human health and soil ecosystems over time. This study demonstrates that while legume micronutrient profiles remain nutritionally adequate, targeted interventions are required to curb and monitor chronic pesticide exposure in this subsistence farming region.

Declaration of Conflict of Interest: The authors declare that no known conflict of interest or personal relationships that could have influence the work reported in this paper

Data Availability: Data are available upon request from the corresponding author or any of the other authors

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