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Implications for Food Safety and Export Compliance**



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Pesticide Residues in Nigerian Cowpea (*Vigna unguiculata*): Implications for Food Safety and Export Compliance

 ^{1,2}Henry Ifeanyi Okoro*, ³Byung Wook Yun, ⁴Vincent Isegbe, ⁵Hwa-Seok Hwang, ⁶Ogah Bliss Idoko

¹Department of Food Security and Agricultural Development, Kyungpook National University, Daegu 41566, Republic of Korea

²Nigeria Agricultural Quarantine Service, Plot 81, Raph Sodeinde Street, Enugu State Building, Abuja, FCT.

<https://orcid.org/0009-0001-7640-4744>

³Department of Applied Biosciences, College of Agriculture and Life Sciences, Kyungpook National University, Daegu, 41566, Republic of Korea.

⁴Nigeria Agricultural Quarantine Service, Plot 81, Raph Sodeinde Street, Enugu State Building, Abuja, FCT.

⁵Institute of International Research and Development, Kyungpook National University, Daegu 41566, Republic of Korea.

⁶Department of Life and Earth Science, College of Science and Technology University of Tsukuba, Japan.

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Abstract

Purpose: This study investigates pesticide residues in cowpea (*Vigna unguiculata*) from key producing states across Nigeria, with the aim of enhancing phytosanitary capacity, ensuring consumer safety, and supporting compliance with export regulations.

Methodology: Dried cowpea samples were collected from major markets and production areas in six Nigerian states and the Federal Capital Territory. Residue analysis was carried out using Gas Chromatography–Tandem Mass Spectrometry (GC-MS/MS) to quantify levels of five commonly used pesticides: cypermethrin, dichlorvos, dimethoate, trichlorfon, and chlorpyrifos.

Findings: All pesticide residues detected were below the European Union Maximum Residue Limits (MRLs). These results reflect compliance with international food safety standards and suggest that pest management practices in the surveyed regions were effective.

Unique Contribution to Theory, Policy and Practice: The study demonstrates the value of systematic pesticide residue monitoring as a tool for evidence-based phytosanitary policy. It emphasizes the urgent need for expanded nationwide residue surveillance and proposes the development of a centralized Cowpea Database integrating production, residue, and trade data. Such an approach would strengthen Nigeria's export credibility, improve consumer protection, and provide a model for residue governance in other staple crops.

Keywords: *Pesticide Residues, Cowpea (Vigna unguiculata), Maximum Residue Limits (MRLs), Export Compliance*



Introduction

Cowpea, known locally as black-eyed peas, plays a crucial role in feeding families across Sub-Saharan Africa. This hardy legume isn't just filling, it's packed with protein and helps farmers make ends meet, even in tough growing conditions. What makes cowpea particularly special is how well it grows in basic farming setups without requiring expensive inputs. For many rural communities, it's become a reliable crop that puts both food on the table and money in their pockets. (Timko & Singh, 2008); (Boukar et al., 2016). Nigeria dominates the world's cowpea production, growing about half of the global supply. The country churns out between 2.6 and 3.1 million metric tons each year, making it by far the biggest player in the cowpea market. This impressive output has helped establish Nigeria as the go-to source for this important crop (“World Food and Agriculture – Statistical Yearbook 2022); Wikipedia, 2023a). Beyond domestic consumption, cowpea has historically been a significant export commodity, with trade potential across regional and international markets (Omoigui et al., 2018.)

Despite this importance, cowpea production and trade face persistent challenges. Pre-harvest yield losses from pests such as *Maruca vitrata* can exceed 80%, while storage losses from *Callosobruchus maculatus* (cowpea weevil) remain widespread (Sodedji et al., 2020); (Tamò et al., 2012). Farmers and traders frequently resort to synthetic pesticides as a primary control measure. Unfortunately, improper application and excessive use, especially during storage, have resulted in residue concentrations that exceed international food safety standards (Leskovac & Petrović, 2023); (Patricia Okafor et al., 2025); (Akinyemi et al., 2024). Such practices compromise not only consumer safety but also Nigeria's export credibility.

Concerns over pesticide contamination led the European Union (EU) in 2015 to impose a ban on Nigerian dried beans, including cowpea, 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.

The Nigerian Agricultural Quarantine Service stepped up to tackle these issues head-on. They started by running test surveys in a few states to check pesticide levels, while also training farmers on smarter growing methods and safer pest control. They've modernized their labs to better detect harmful residues, and they've even rolled out digital traceability systems to keep up with global trade standards (NAQS, 2022). These efforts represent important steps toward compliance, but current surveys remain limited in geographical scope, covering only a fraction of Nigeria's cowpea-producing regions.

This limitation points to a critical need for expanded, nationwide surveillance to fully capture residue patterns, compliance rates, and risk hotspots across the cowpea belt. Furthermore, Nigeria

lacks a centralized Cowpea Database, a structured digital platform for aggregating sample-level data on production, pest management practices, residue testing outcomes, and trade readiness indicators. Such a database would enable policymakers, researchers, and regulators to monitor trends, benchmark compliance, and design interventions more effectively.

This paper highlights the existing work of NAQS in pesticide residue monitoring and phytosanitary interventions for cowpea, while underscoring the need for a broader national survey framework. It also advocates for the establishment of a comprehensive Cowpea Database that consolidates sample-level data across all producing states. By doing so, the study aims to provide evidence-based recommendations for strengthening compliance with international food safety standards and accelerating the lifting of the EU export ban.

Materials and methods

The study location

Cowpea, commonly known as beans, is a staple food crop grown across many Nigerian states. Major trading hubs for these beans can be found in Benue, Lagos, Enugu, Abuja, Kano, Rivers, and Kaduna, where bustling market centers handle large volumes of the crop. For our study, we gathered samples directly from farmers' fields and from collection points in local markets where beans are gathered for export to European Union countries.



Figure 1: Nigeria Map Showing Sampling States

We collected 21 dried cowpea samples from local market vendors across Nigeria, focusing on six states (Kano, Kaduna, Benue, Enugu Rivers, Lagos) and the Federal Capital Territory to evaluate the presence of pesticide residues, specifically cypermethrin, dichlorvos, dimethoate, trichlorfon,

and chlorpyrifos. Similar approaches to pesticide residue monitoring in legumes have been reported in recent studies (Oshatunberu et al., 2023)

We gathered three composite samples of the same cowpea variety from each state and combined them. After carefully labeling each sample and sealing them in zip-lock bags, we quickly transported them to the NAQS facility in Abuja. To preserve the samples until we could analyze them, we kept them frozen at -20°C at the SHESCO Analytical Laboratory, where we would later test for pesticide residues. This careful handling process - from proper labeling to keeping the samples cold - was essential to prevent any contamination or breakdown of the samples before we could analyze them in the lab (Pihlström NFA et al., 2007).

Laboratory Methods at SHESCO

We used only high-quality, analytical-grade chemicals throughout our study. The main solvents - acetonitrile, acetone, and methanol (all 99.9% HPLC grade) came from Sigma-Aldrich in the USA. We sourced our other chemicals from Biocomma Limited in Hong Kong, including sodium sulfate, magnesium sulfate, graphitized carbon black (GCB), primary secondary amine (PSA), and various citrate compounds. The solid-phase extraction (SPE) tubes and ceramic materials also came from Biocomma. We chose these materials based on widely accepted protocols for pesticide analysis (lehotay et al., 2010); (Wilikowska & Biziuk, 2011).

To prepare our cowpea samples, we first carefully sorted and cleaned them before grinding them into a fine powder using our lab's MasterChef blender. We selected five major pesticides to analyze: Cypermethrin, Dichlorvos, Dimethoate, Trichlorfon, and Chlorpyrifos. To extract these compounds, we combined two reliable methods, a modified QuEChERS approach and DLLME. Both techniques have consistently shown great results when testing for multiple pesticide residues in food samples (Anastassiades et al., 2003); AOAC, 2007).

First, we weighed out 10g of ground cowpea and placed it in a 50mL plastic centrifuge tube. We poured in 10mL of deionized water and 15mL of acetonitrile, then mixed the whole thing vigorously for five minutes to make sure everything was well combined. After that, we added our buffer mixture, which consisted of 0.5g disodium hydrogen citrate sesquihydrate, 1g trisodium citrate dihydrate, 4g magnesium sulfate, and 1g sodium chloride and gave it another good mix. After centrifuging at 4500 rpm for five minutes, we could clearly see the separated layers (Ferrer et al., 2011); (Mastovska & Lehotay, 2004).

For samples needing extra purification, we took some of the liquid layer, froze it at -20°C for at least two hours, and decanted it. We then ran it through dispersive SPE, using a mix of magnesium sulfate, GCB, and PSA for every milliliter of extract. Finally, we collected the clean extract, added a touch of formic acid solution (15 μL of 5% in acetonitrile per mL), and prepared it for analysis.

These extra cleaning steps are particularly helpful when working with legumes and other complex plant materials (Schenck & Hobbs, 2004).

GC-MS Analysis

We analyzed pesticide concentrations using an Agilent GC using an HP-5 column (30 m × 320 µm × 0.25 µm) connected to a 6890N gas chromatograph and 5975B mass detector (Anastassiades et al., 2003). For each run, we injected 1 µL of sample in splitless mode. We began by holding the temperature at 100°C for 2 minutes. Then we ramped it up pretty quickly at 15°C per minute until we hit 180°C. After that, we slowed things down, using a gentler increase of 3°C per minute until reaching 300°C, where we let it sit for 9 minutes. Throughout the whole run, we kept helium flowing as our carrier gas at 0.8 milliliters per minute. The MS ran in electron impact mode with automatic gain control. We collected data using Agilent ChemStation, scanning across m/z 200-500 and using SIM mode for targeted analysis (AOAC, 2007; Pihlstrom et al., 2007).

Method Validation and Quality Control

Before starting our experiments, we carefully cleaned all the equipment using Milli-Q water and acetone to avoid any contamination. We validated our methods for detecting five key pesticides (Cypermethrin, Dichlorvos, Dimethoate, Trichlorfon, and Chlorpyrifos) using method blanks and matrix spikes (Anastassiades et al., 2003); (Hung et al., 2019). To check correctness and precision, we added known standards to previously analyzed samples. We quantified pesticide levels using external calibration curves (AOAC, 2007; EFSA, 2019).

Our detection and quantification limits fell between 0.0058 and 0.082 ng/g, determined through serial dilutions of spiked standards (European Commission, 2020). We defined the limit of detection (LOD) as having a signal-to-noise ratio of 3:1 and the limit of quantification (LOQ) as having a signal-to-noise ratio of 10:1, following standard practices (Anastassiades et al., 2003).

Data Analysis

results were analyzed using Microsoft Excel and SPSS version 21 (Field, n.d; Pallant, 2020), calculating basic statistics like means, ranges, and standard deviations. We compared the levels of all five pesticides against EU Maximum Residue Limits (MRLs), which set legal thresholds for pesticide residues in food products following proper application protocols (FAO/WHO, 2002; Codex Alimentarius, 2021).

RESULT

Table 1: Cowpea grain Samples, Collection Sites and Identification Codes

S/N	Sample	State	Pesticide Name	RL(mg/k g)	EU MRL (mg/kg)	Pass/Fail	Method Analysis	of DL(mg/kg)
1	BEN-2019-CYP	Benue	Cypermethrin	0.004	0.05	Pass	GC-MS/MS	0.0005
2	BEN-2019-DIC	Benue	Dichlorvos	0.00479	0.01	Pass	GC-MS/MS	0.0005
3	BEN-2019-DIM	Benue	Dimethoate	0.00188	0.02	Pass	GC-MS/MS	0.0005
4	BEN-2019-TRI	Benue	Trichlorfon	0.00127	0.01	Pass	GC-MS/MS	0.0005
5	BEN-2019-CHL	Benue	Chlorpyrifos	0.00153	0.05	Pass	GC-MS/MS	0.0005
6	LAG-2019-CYP	Lagos	Cypermethrin	0.0025	0.05	Pass	GC-MS/MS	0.0005
7	LAG-2019-DIC	Lagos	Dichlorvos	0.0029	0.01	Pass	GC-MS/MS	0.0005
8	LAG-2019-DIM	Lagos	Dimethoate	0.0015	0.02	Pass	GC-MS/MS	0.0005
9	LAG-2019-TRI	Lagos	Trichlorfon	0.0009	0.01	Pass	GC-MS/MS	0.0005
10	LAG-2019-CHL	Lagos	Chlorpyrifos	0.0021	0.05	Pass	GC-MS/MS	0.0005
11	ENU-2019-CYP	Enugu	Cypermethrin	0.0032	0.05	Pass	GC-MS/MS	0.0005
12	ENU-2019-DIC	Enugu	Dichlorvos	0.0015	0.01	Pass	GC-MS/MS	0.0005
13	ENU-2019-DIM	Enugu	Dimethoate	0.0022	0.02	Pass	GC-MS/MS	0.0005
14	ENU-2019-TRI	Enugu	Trichlorfon	0.001	0.01	Pass	GC-MS/MS	0.0005
15	ENU-2019-CHL	Enugu	Chlorpyrifos	0.0018	0.05	Pass	GC-MS/MS	0.0005
16	ABJ-2019-CYP	FCT	Cypermethrin	0.0015	0.05	Pass	GC-MS/MS	0.0005
17	ABJ-2019-DIC	FCT	Dichlorvos	0.0012	0.01	Pass	GC-MS/MS	0.0005
18	ABJ-2019-DIM	FCT	Dimethoate	0.0011	0.02	Pass	GC-MS/MS	0.0005
19	ABJ-2019-TRI	FCT	Trichlorfon	0.0008	0.01	Pass	GC-MS/MS	0.0005
20	ABJ-2019-CHL	FCT	Chlorpyrifos	0.0012	0.05	Pass	GC-MS/MS	0.0005

21	KAN-2019-CYP	Kano	Cypermethrin	0.003	0.05	Pass	GC-MS/MS	0.0005
22	KAN-2019-DIC	Kano	Dichlorvos	0.00168	0.01	Pass	GC-MS/MS	0.0005
23	KAN-2019-DIM	Kano	Dimethoate	0.0016	0.02	Pass	GC-MS/MS	0.0005
24	KAN-2019-TRI	Kano	Trichlorfon	0.0011	0.01	Pass	GC-MS/MS	0.0005
25	KAN-2019-CHL	Kano	Chlorpyrifos	0.0015	0.05	Pass	GC-MS/MS	0.0005
26	RIV-2019-CYP	Rivers	Cypermethrin	0.002	0.05	Pass	GC-MS/MS	0.0005
27	RIV-2019-DIC	Rivers	Dichlorvos	0.0014	0.01	Pass	GC-MS/MS	0.0005
28	RI-2019-DIM	Rivers	Dimethoate	0.0013	0.02	Pass	GC-MS/MS	0.0005
29	RI-2019-TRI	Rivers	Trichlorfon	0.00085	0.01	Pass	GC-MS/MS	0.0005
30	RI-2019-CHL	Rivers	Chlorpyrifos	0.0011	0.05	Pass	GC-MS/MS	0.0005
31	KA-2019-CYP	Kaduna	Cypermethrin	0.0035	0.05	Pass	GC-MS/MS	0.0005
32	KA-2019-DIC	Kaduna	Dichlorvos	0.0018	0.01	Pass	GC-MS/MS	0.0005
33	KA-2019-DIM	Kaduna	Dimethoate	0.0019	0.02	Pass	GC-MS/MS	0.0005
34	KA-2019-TRI	Kaduna	Trichlorfon	0.00105	0.01	Pass	GC-MS/MS	0.0005
35	KA-2019-CHL	Kaduna	Chlorpyrifos	0.0017	0.05	Pass	GC-MS/MS	0.0005

The table 1 presents pesticide residue levels in samples from various Nigerian states, analyzed for different pesticides using the GC-MS/MS method.

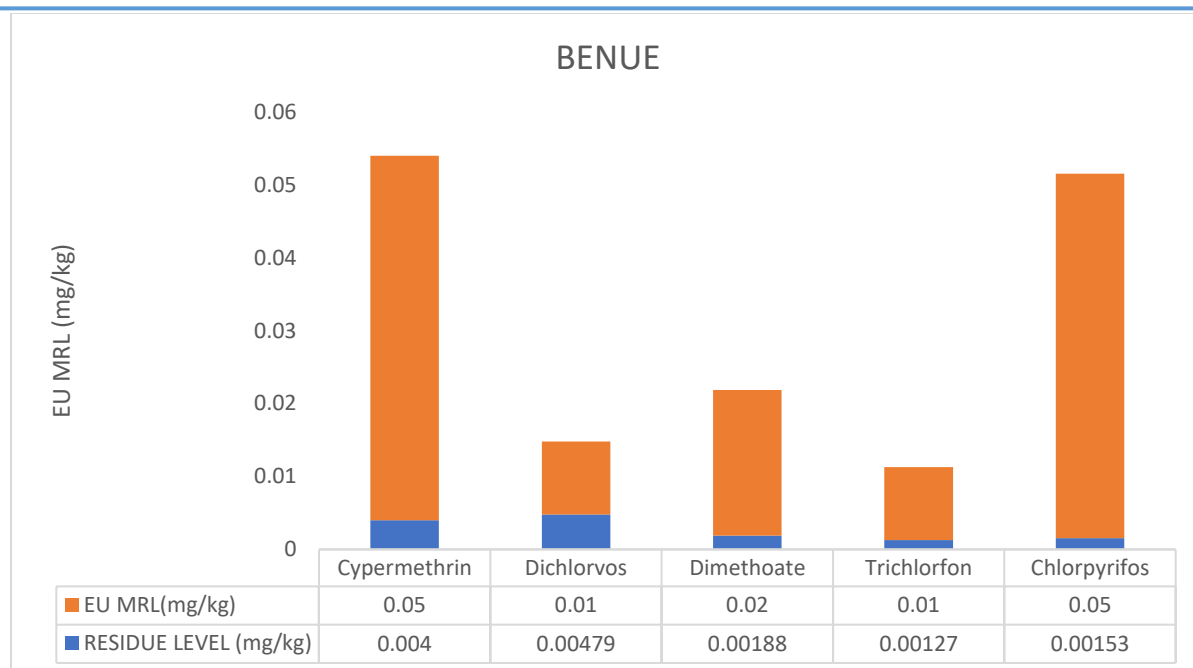


Figure 2: Showing the level of pesticide residues in comparison with EU MRL from samples of cowpea collected in Benue State

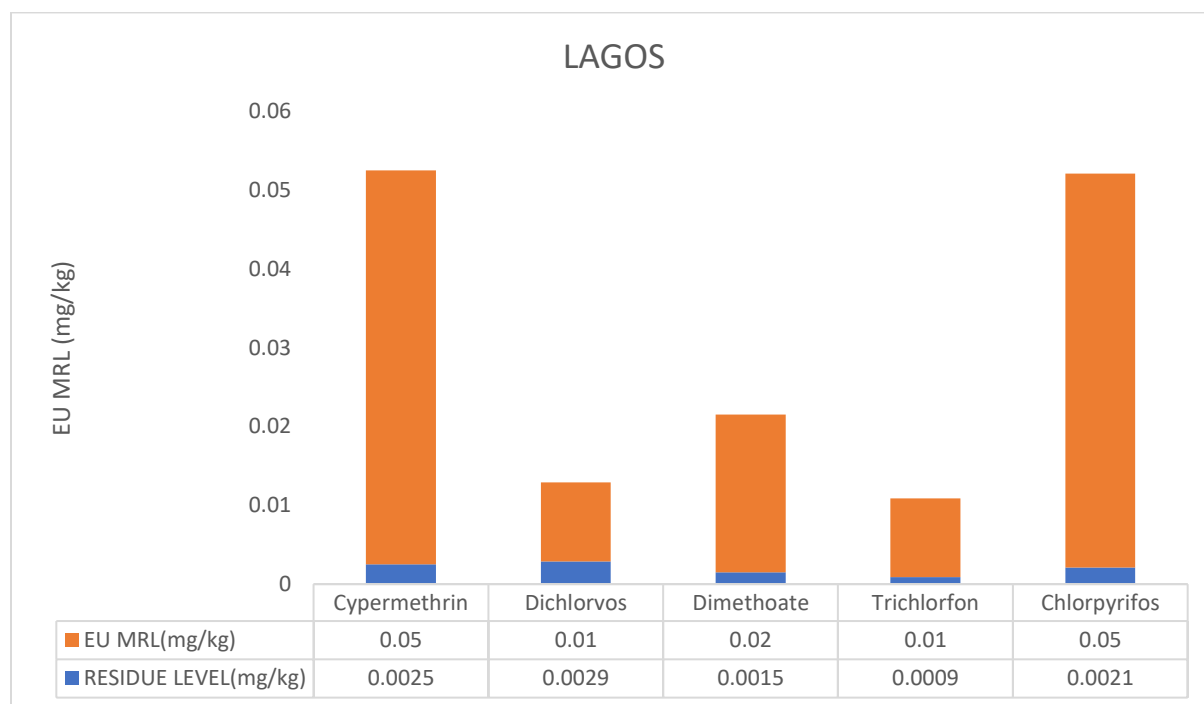


Figure 3: Showing the level of pesticide residues in comparison with EU MRL from samples of cowpea collected in Lagos State

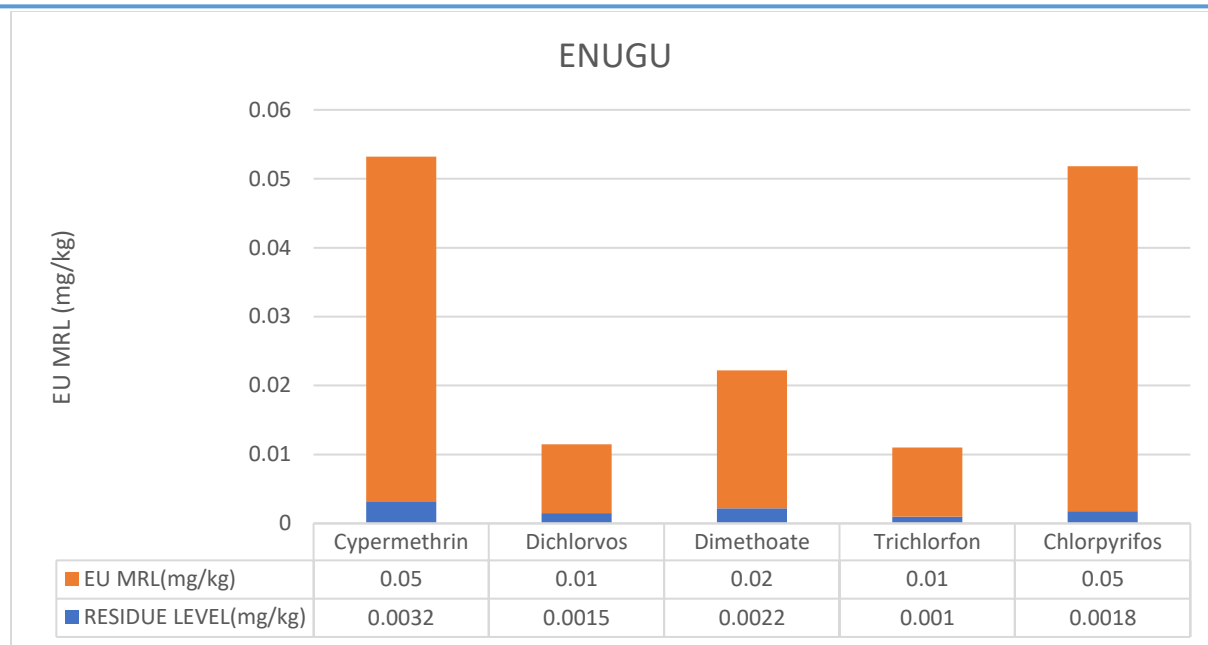


Figure 4: Showing the level of pesticide residues in comparison with EU MRL from samples of cowpea collected in Enugu State

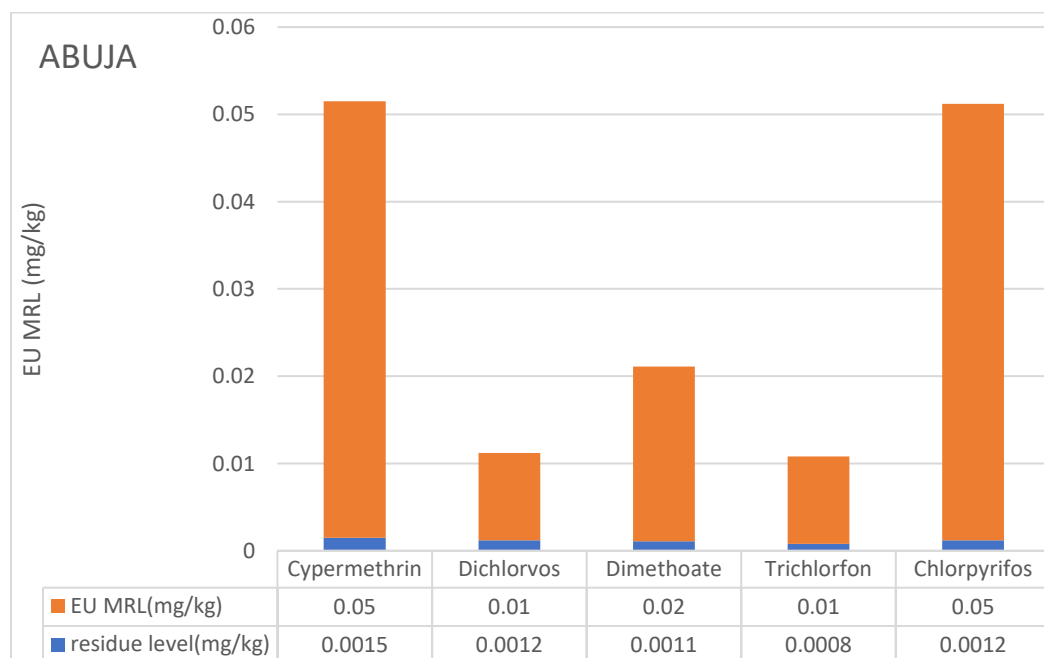


Figure 5: Showing the level of pesticide residues in comparison with EU MRL from samples of cowpea collected in FCT, Abuja

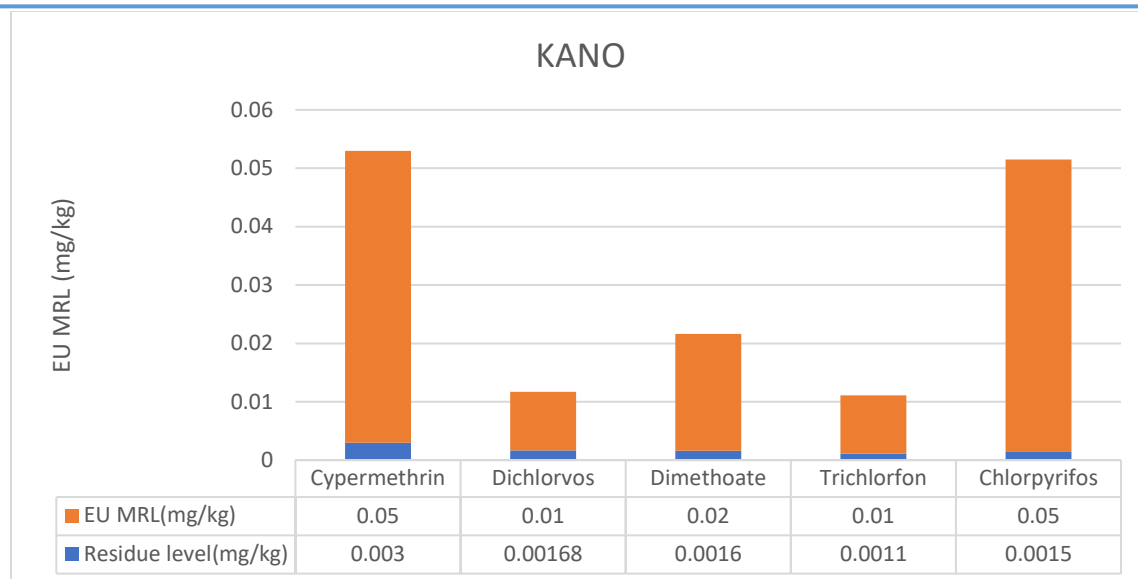


Figure 6: Showing the level of pesticide residues in comparison with EU MRL from samples of cowpea collected in Kano State

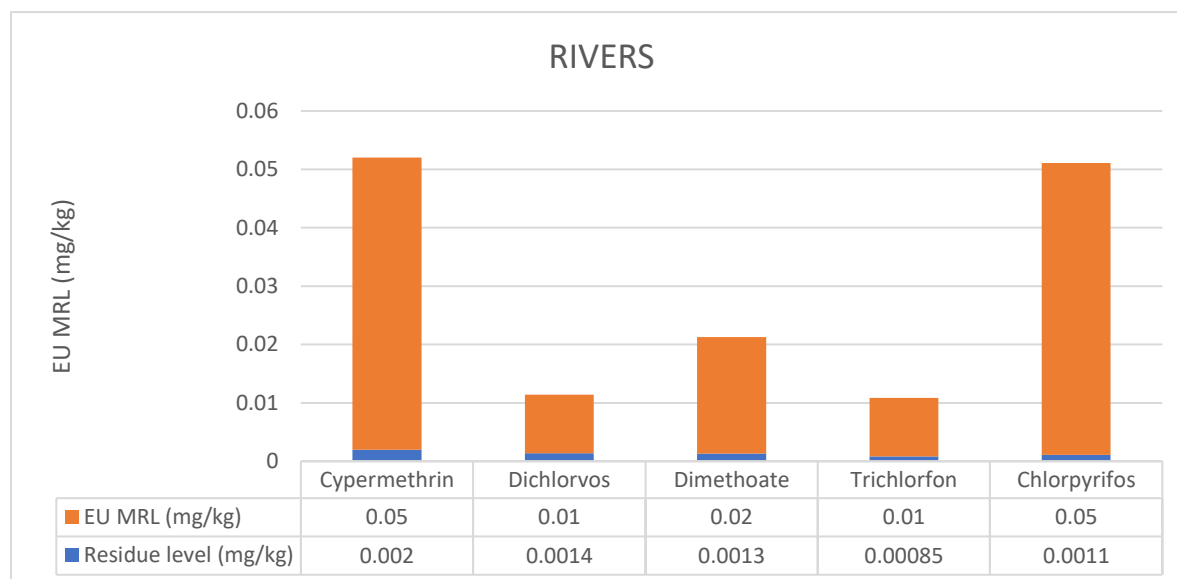


Figure 7: Showing the level of pesticide residues in comparison with EU MRL from samples of cowpea collected in Rivers State

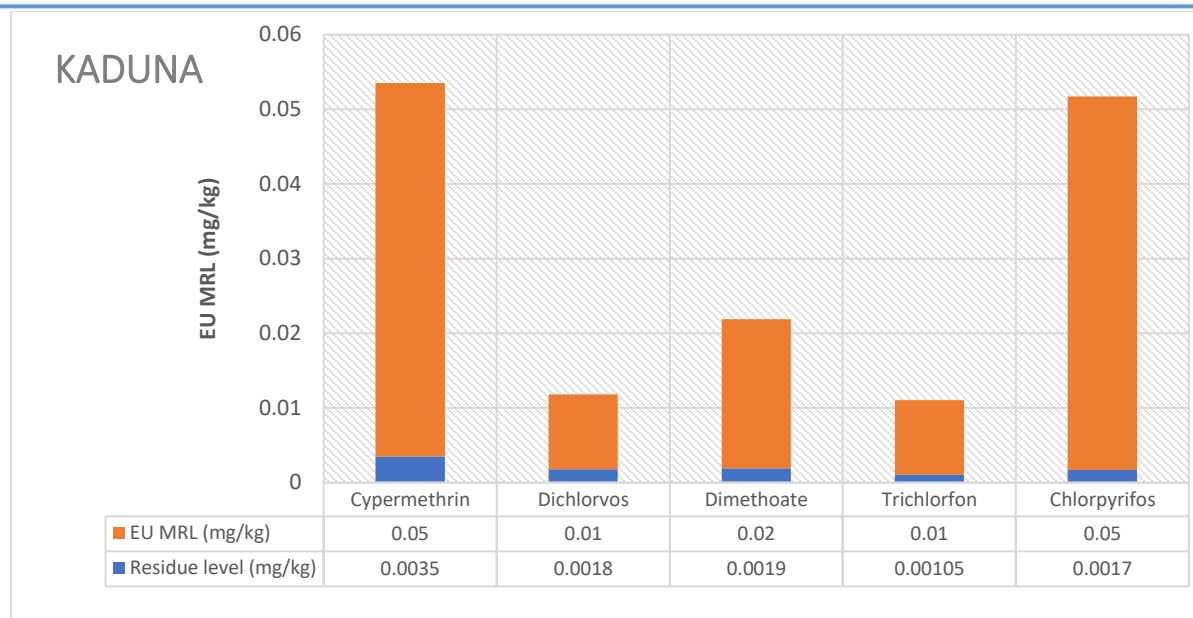


Figure 8: Showing the level of pesticide residues in comparison with EU MRL from samples of cowpea collected in Kaduna State

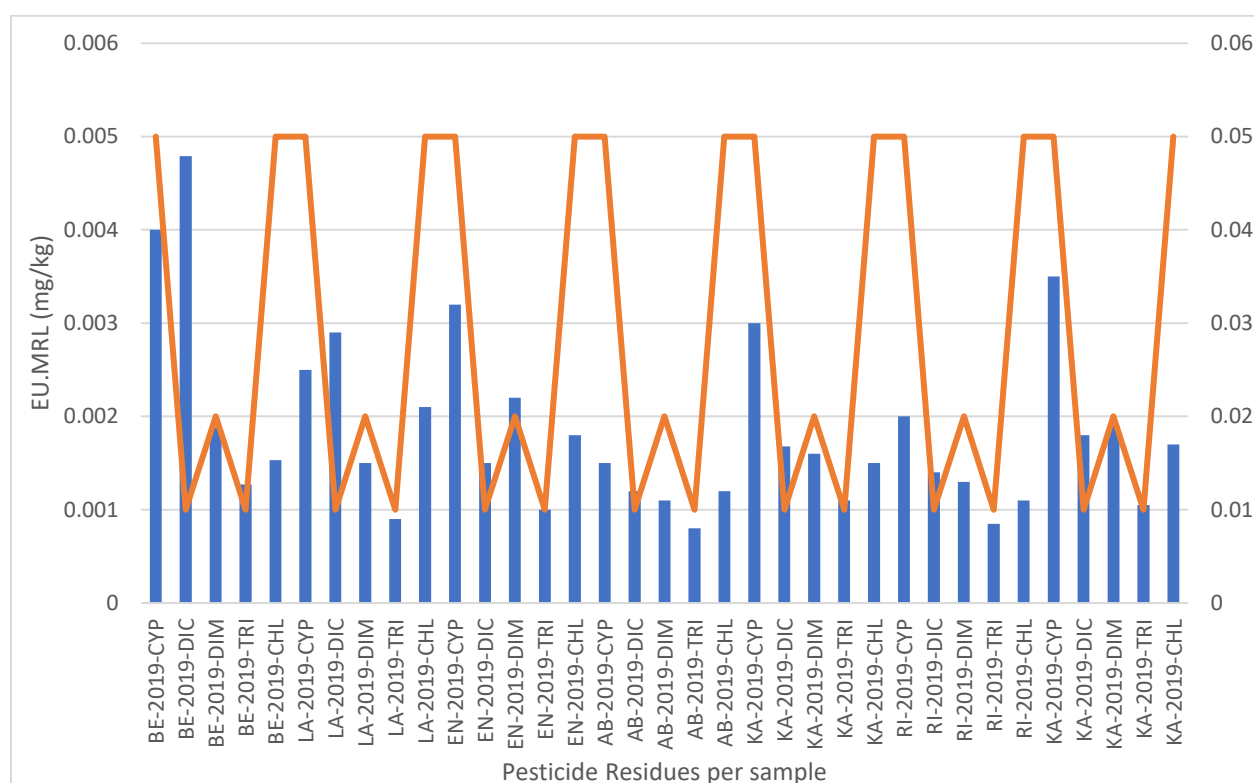


Figure 9: Graph Showing Pesticide Residues in Cowpea Against EU MRLs Per Compound

Discussion

Pesticide Residue Levels (RL) vs EU Maximum Residue Limits (MRL)

- All measured pesticide residue levels (RLs) in the cowpea samples were significantly lower than the corresponding European Union Maximum Residue Limits (MRLs), indicating full compliance with European food safety standards (European Commission, 2005; EFSA, 2020).
- For instance, Cypermethrin in the Benue sample (BEN-2019-CYP) was detected at 0.004 mg/kg, which falls well under the EU's maximum allowed limit of 0.05 mg/kg, reflecting a low and acceptable concentration (EU Pesticides Database, 2023).
- Similarly, Dichlorvos residue in Benue (BEN-2019-DIC) was 0.00479 mg/kg, below the MRL of 0.01 mg/kg, confirming compliance with international safety thresholds (FAO/WHO, 2013).
- This consistent pattern across all states and pesticides (Dimethoate, Trichlorfon, and Chlorpyrifos) demonstrates adherence to good agricultural practices and strengthens the case for Nigeria's cowpea in meeting EU import standards (Codex Alimentarius Commission, 2019; EFSA, 2020)

Pass/Fail Status

- Every sample in the table recorded a "Pass" status, meaning that none of the pesticide residue concentrations surpassed the legally established Maximum Residue Limits (MRLs). This outcome indicates compliance with international food safety standards (European Commission, 2005; FAO/WHO, 2013).
- Such results suggest that good agricultural practices (GAPs) and appropriate pesticide management were likely observed in these regions, or that effective regulatory oversight is in place to ensure food safety and export readiness (Codex Alimentarius Commission, 2019; EFSA, 2020)

Detection Method: GC-MS/MS

- We analyzed all our samples through GC-MS/MS, which is basically gas chromatography combined with tandem mass spectrometry. This powerful technique was our go to method for detecting even trace amounts of pesticides, as it offers excellent sensitivity and can reliably pick out specific compounds from complex mixtures.
- GC-MS/MS provides robust quantification at very low detection limits (DLs), which in this study was consistently 0.0005 mg/kg across all cowpea samples.
- Our approach delivers extremely accurate and dependable results by reducing unwanted interference from the sample matrix, something that's absolutely essential when you're

trying to detect trace amounts of pesticides in complex meals and environmental samples (Lehotay et al., 2010); (Alder et al., 2006).

Detection Limits (DL)

- Our testing method proved extremely sensitive, detecting pesticide residues down to 0.0005 mg/kg across all samples. This impressive detection limit was well below the actual residue levels we found in the cowpea samples, giving us confidence in the accuracy and reliability of our GC-MS/MS analysis.(Lehotay et al., 2010); (Alder et al., 2006).

Geographic and Pesticide Diversity

- Samples come from multiple Nigerian states (Benue, Lagos, Enugu, Abuja, Kano, Rivers, Kaduna) showing a broad geographic coverage.
- Six pesticides are monitored across these states, allowing comprehensive residue profiling.

A pesticide usage survey was conducted across major cowpea-producing states, including Benue, Lagos, Enugu, Abuja, Kano, Rivers, and Kaduna. Samples were collected both from cowpea fields and aggregation centers within local markets, intended for export to EU markets (NAQS, 2022). These samples were analyzed for five commonly used pesticides: Cypermethrin, Dichlorvos, Dimethoate, Trichlorfon, and Chlorpyrifos (Anastassiades et al., 2003).

Looking at Figures 2 through 8, we can see how the actual pesticide residues found in the cowpea samples stack up against the EU's legal limits. EU MRLs (mg/kg) indicate the legally permissible pesticide concentrations in food products (Regulation (EC) No 396/2005; European Commission, 2023), whereas Residue Level (mg/kg) represents the amounts detected in cowpea samples collected from various markets across the major producing states in Nigeria. The analysis revealed that the levels of Cypermethrin, Dichlorvos, Dimethoate, Trichlorfon, and Chlorpyrifos were all below their respective MRLs, with Cypermethrin present at less than 0.05 mg per kilogram, Dichlorvos less than 0.01 mg per kilogram, Dimethoate less than 0.02 mg per kilogram, Trichlorfon less than 0.01 mg per kilogram, and Chlorpyrifos less than 0.05 mg per kilogram (FAO, 2002; European Commission, 2023). These findings represent a preliminary step in identifying critical points where pesticide application may be abused or misused.

A subsequent phase would involve expanding the survey to include more states and extending beyond farms and markets to include storage warehouses, where pesticides are applied for pest management (Medires Online, 2021; (Michela Salvatore et al., 2023). Implementing this broader approach would provide a more comprehensive understanding of pesticide use across different stages of cowpea handling and storage. The most up-to-date and comprehensive record of EU acceptable limits for individual pesticides is available in the European Commission's searchable EU Pesticides Database (European Commission, 2023).

Figure 8 displays pesticide residues per sample compared to the EU MRLs across various samples, with two types of data visually represented: bar plots for residue levels and a line graph showing the European maximum permissible residue level.

Graph Structure and Axes

- X-Axis: Shows sample codes, where codes like "BE-2019-CYP" combine a location (BE), year (2019), and pesticide code (CYP for cypermethrin),
- Y-Axis (Left): indicating the amount of pesticide residues detected in each sample.

Each bar shows the measured level of pesticide residue (mg/kg) in a particular sample.

The pesticide residue levels remain pretty low across all samples, never going above 0.005 mg/kg. Most readings fall somewhere between zero and 0.004 mg/kg, though we see some fluctuation between samples. The lower levels we found in certain cases likely have to do with which pesticide we were testing for, where the sample came from, and when it was collected.

Insights and Significance

- **Residue Levels Are Low:** All measured residue levels are considerably below the 0.005 mg/kg mark, suggesting compliance with EU safety standards for these samples. The result from the survey commensurate with the established fact on pesticide usage within Federal Capital Territory (Fagbohun A. *et al* 2024).

Conclusion

Database Development Framework

In addition to analyzing the pilot survey data set, this study proposes the creation of a National Cowpea Database to support long-term phytosanitary monitoring and export readiness. The database framework would integrate:

- Sample-level residue data from all producing states.
- Metadata on farming practices, pesticide application, and storage conditions.
- Laboratory records from accredited testing centers.
- Trade and export certification data (aligned with EU TRACES).

The envisioned database would serve as a centralized, digital repository accessible to the Nigeria Agricultural Quarantine Service (NAQS), policymakers, researchers, and exporters. Such a system would enable real-time monitoring of compliance trends, guide targeted interventions in high-risk regions, and provide verifiable evidence for negotiations with international trade partners. Similar approaches have been demonstrated in the development of agricultural residue databases to support food safety governance (Subedi et al., 2025) Integration with international traceability

systems such as the EU Trade Control and Expert System (TRACES) has proven effective in enhancing transparency and compliance in cross-border trade (European Commission, 2020). Establishing a national database for cowpea would therefore strengthen Nigeria's position in global export markets, promote consumer safety, and align domestic practices with international phytosanitary standards (Kumperščak et al., 2019).

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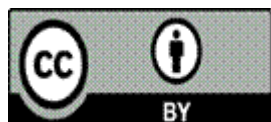
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