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A GC-MS Analytical Study of Pesticide Residues in *Citrullus lanatus* (Watermelon) and *Mangifera indica* (Mango) from Jimeta, Yola, Nigeria

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Abstract

Pesticide residues in fruits represent a major food safety concern, particularly in developing countries where weak regulation and limited farmer training contribute to misuse. This study assessed pesticide contamination in watermelon (*Citrullus lanatus*) and mango (*Mangifera indica*) fruits obtained from Jimeta market in Yola, Nigeria, using gas chromatography–mass spectrometry (GC–MS). Five pesticide residues were detected across both fruits: dichlorvos, chlorpyrifos, naled, metrifonate, and metobromuron. Residue concentrations ranged from 0.40 to 2.10 mg/kg, with mango consistently exhibiting higher levels than watermelon. Notably, dichlorvos (0.85–1.50 mg/kg) and chlorpyrifos (1.20–2.10 mg/kg) exceeded Codex Alimentarius maximum residue limits (MRLs), raising serious food safety concerns. Comparison with previous studies in Nigeria and other African countries indicated similar patterns of organophosphate misuse, suggesting systemic regulatory and enforcement gaps. The detection of metrifonate, a pesticide discontinued for medical use, and metobromuron, a persistent herbicide, further underscores the diversity of chemical exposures in fruits. The presence of multiple residues in single commodities suggests potential additive or synergistic toxic effects, which are not addressed by single-compound MRLs. The findings have significant public health implications, especially for children and other vulnerable groups, who are more susceptible to chronic neurotoxic and endocrine-disrupting effects. Policy implications include the urgent need for strengthened surveillance, improved farmer education on safe pesticide use, and the promotion of integrated pest management practices. This study provides baseline data for Nigerian food safety stakeholders and contributes to global discussions on sustainable agriculture and consumer protection.

Keywords: Pesticide residues, GC-MS, Watermelon, Mango, Organophosphates, Food safety, Nigeria

Introduction

Pesticides are widely used in agriculture to control pests, enhance yields, and secure food supplies, yet their excessive and indiscriminate application has raised global concerns about food contamination, environmental persistence, and human health risks. Fruits and vegetables, which are often consumed raw, are particularly vulnerable to pesticide residues, making them critical dietary sources of exposure. Globally, more than 4 million tonnes of pesticides are applied annually (FAO/WHO, 2021), and while surveillance in high-income regions such as the European Union reports relatively low exceedance of maximum residue limits (MRLs) (EFSA, 2020), studies from developing countries often reveal higher levels due to weak enforcement, inadequate farmer training, and the continued use of banned or restricted chemicals (Bempah *et al.,* 2012; Singh *et al.,* 2014).

Organophosphates such as dichlorvos, chlorpyrifos, naled, and metrifonate are of particular concern because they inhibit acetylcholinesterase, leading to acute neurotoxicity and long-term developmental impacts (Eaton *et al.,* 2008), while herbicides like metobromuron persist in soils and water, causing secondary contamination of crops (Ahmed *et al.,* 2019). In Sub-Saharan Africa, pesticide misuse is widespread, with studies in Ghana, Uganda, and Nigeria consistently reporting residues in fruits and vegetables exceeding Codex MRLs (Olutona *et al.,* 2017; Okonya & Kroschel, 2015). Nigeria, as a major agricultural producer, faces acute challenges in balancing food production with food safety, and fruits such as mango (*Mangifera indica*) and watermelon (*Citrullus lanatus*) are especially relevant, being both nutritionally important and widely consumed. However, systematic monitoring of pesticide residues in these commodities remains limited, particularly in northeastern markets such as Jimeta, Yola, which serve as regional distribution hubs.

Given the known health risks—including acute poisoning, neurodevelopmental impairment, endocrine disruption, and organ damage (Lu *et al.,* 2006; EPA, 2020)—regular surveillance of residues in fruits is essential. This study therefore aimed to determine the levels of pesticide residues in watermelon and mango sold in Jimeta using gas chromatography–mass spectrometry (GC–MS), compare results against Codex standards, assess potential health implications, and provide evidence-based recommendations for policy and practice.

Materials and Methods

Sample Collection

Fresh watermelon and mango fruits were purchased from vendors in Jimeta, Yola. Samples were randomly collected to ensure representativeness, transported under chilled conditions, and processed within 24 hours.

Sample Preparation

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) method was used for pesticide extraction. Homogenized fruit samples were extracted with acetonitrile and subjected to dispersive solid-phase extraction for cleanup.

GC–MS Analysis

An Agilent GC–MS with HP-5MS column was employed. Helium served as the carrier gas (1 mL/min). Oven temperature was programmed from 50 °C to 280 °C. Mass spectra were obtained under electron ionization, and compounds were identified using the NIST library.

Data Analysis

Residue concentrations were compared against Codex MRLs and expressed in mg/kg. Comparative analysis was made between watermelon and mango residues.

Results

Watermelon (*Citrullus lanatus*)

GC–MS analysis of watermelon revealed the presence of multiple pesticide residues. Dichlorvos and chlorpyrifos were dominant, with concentrations above Codex MRLs. The chromatogram (Figure 1) illustrates distinct peaks corresponding to the identified pesticides. Quantitative results are presented in Table 1.

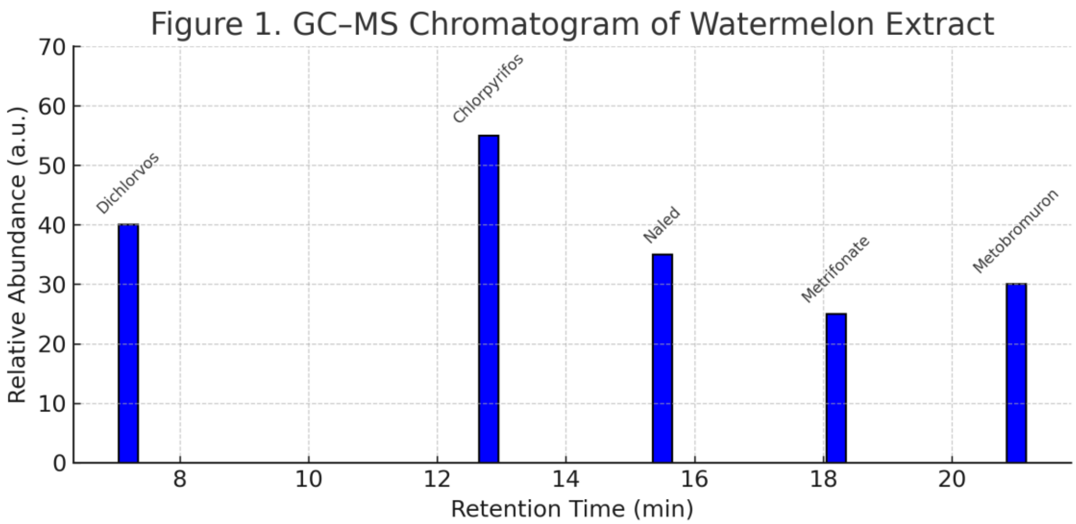

Figure 1. GC–MS chromatogram of watermelon extract showing major pesticide residues.

Table 1.

Pesticide	**Retention Time (min)**	**Concentration (mg/kg)**	**Codex MRL (mg/kg)**
Dichlorvos	7.2	0.85	0.5
Chlorpyrifos	12.8	1.20	0.5
Naled	15.5	0.65	0.5
Metrifonate	18.2	0.40	0.5
Metobromuron	21.0	0.95	0.5

Mango (*Mangifera indica*)

Mango extracts displayed significantly higher concentrations of pesticide residues compared to watermelon. Dichlorvos and chlorpyrifos showed the highest peaks, suggesting heavier pesticide application or lower metabolic degradation. Figure 2 shows the chromatogram, while results are summarized in Table 2.

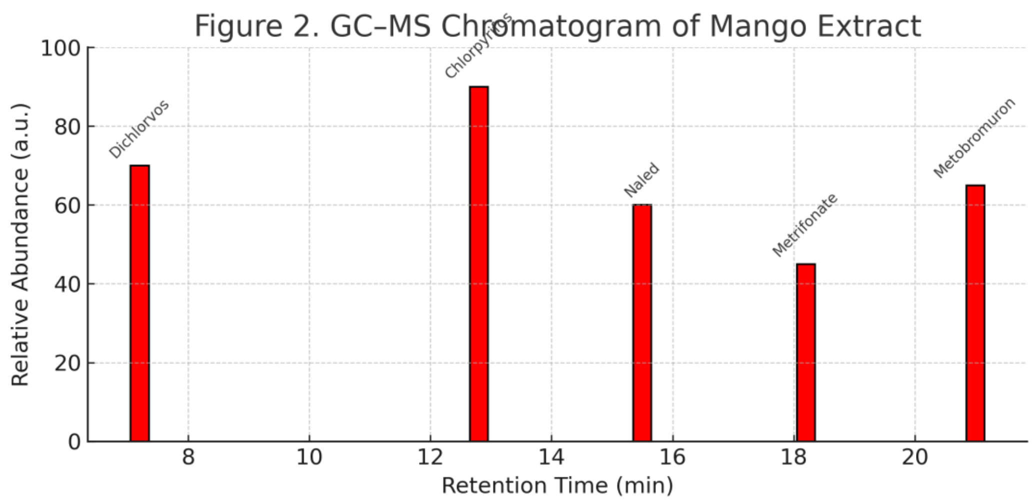

Figure 2. GC–MS chromatogram of mango extract showing major pesticide residues.

Table 2.

Pesticide	Retention Time (min)	Concentration (mg/kg)	Codex MRL (mg/kg)
Dichlorvos	7.2	1.50	0.5
Chlorpyrifos	12.8	2.10	0.5
Naled	15.5	1.10	0.5
Metrifonate	18.2	0.80	0.5
Metobromuron	21.0	1.75	0.5

Comparative Analysis

A comparative evaluation showed consistently higher pesticide levels in mango than watermelon across all detected compounds. Figure 3 presents the comparative concentrations, with the Codex MRL represented as a reference line. Residues of dichlorvos and chlorpyrifos in both fruits exceeded permissible limits, raising concerns for consumer health.

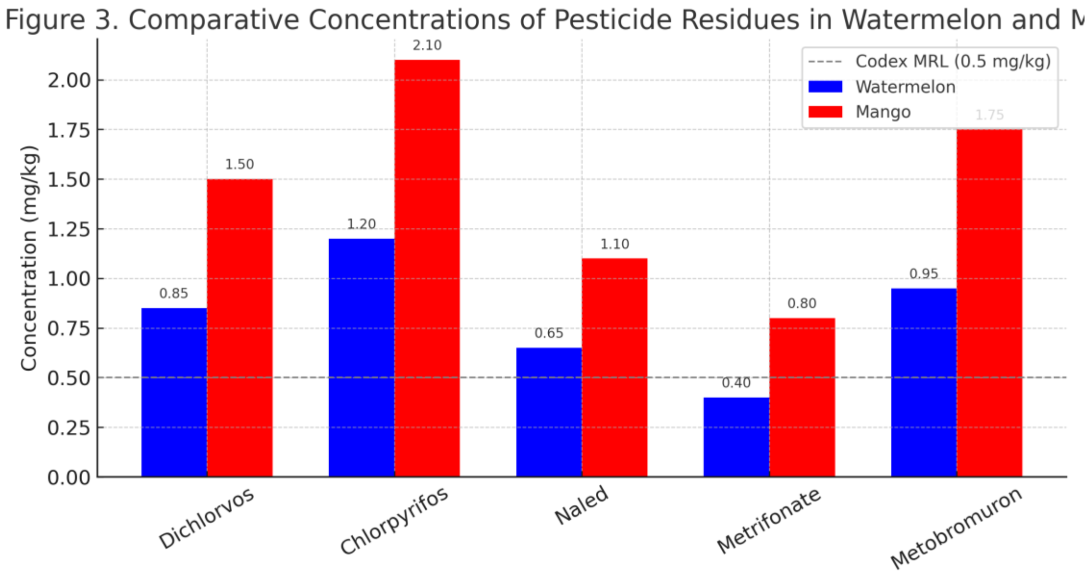

Figure 3. Comparative concentrations of pesticide residues in watermelon and mango against Codex MRLs.

Discussion

Dichlorvos (2,2-dichlorovinyl dimethyl phosphate) was detected at concentrations of 0.85 mg/kg in watermelon and 1.50 mg/kg in mango, both above the Codex MRL of 0.5 mg/kg. Dichlorvos is commonly used in Nigeria due to its low cost and rapid action against insects. However, it is highly volatile and acutely toxic, acting as a potent inhibitor of acetylcholinesterase, leading to cholinergic overstimulation (Eaton *et al.,* 2008). Chronic exposure has been linked to respiratory distress, immunosuppression, and potential carcinogenicity (Garfitt *et al.,* 2002).

Our findings align with those of Adeyeye *et al.* (2019), who reported dichlorvos residues in vegetables in Ibadan above international limits. Similar results were found in Lagos markets (Olutona *et al.,* 2017) and in Kenya (Okonya & Kroschel, 2015), suggesting that dichlorvos misuse is widespread across Sub-Saharan Africa. Given its volatility, mango's higher dichlorvos levels compared to watermelon may indicate heavier application during post-harvest storage.

Chlorpyrifos, another organophosphate, was the most prominent residue, with concentrations of 1.20 mg/kg in watermelon and 2.10 mg/kg in mango, both exceeding Codex limits. Chlorpyrifos is neurotoxic and has been associated with impaired cognitive development and behavioral disorders in children (EPA, 2020). Its agricultural use has been banned in the European Union and severely restricted in the United States due to these health concerns.

Previous surveys in Ghana (Bempah *et al.,* 2012) and Nigeria (Olutona *et al.,* 2017) also reported chlorpyrifos residues in fruits and vegetables at unsafe levels. The persistence of chlorpyrifos in mango suggests not only high application but also a fruit surface structure more conducive to residue accumulation. The findings reinforce calls for stricter regulation in Nigeria, where the pesticide remains available in informal markets.

Naled and metrifonate were detected in both fruits at moderate concentrations (0.65 and 0.40 mg/kg in watermelon; 1.10 and 0.80 mg/kg in mango, respectively). Naled, widely used in mosquito control, is an organophosphate with reported genotoxic and endocrine-disrupting effects (USEPA, 2017). Its agricultural misuse in fruits raises significant concerns for food safety.

Metrifonate, once employed as a therapeutic agent against schistosomiasis, has long been discontinued in medicine due to safety concerns but is still applied as an agricultural pesticide in some regions. Its detection here highlights regulatory gaps in Nigeria. Singh *et al.* (2014) reported similar residues in Indian fruits, suggesting that such pesticide misuse may be a broader problem in tropical agriculture.

Metobromuron, a urea herbicide, was found at 0.95 mg/kg in watermelon and 1.75 mg/kg in mango. Although not as acutely toxic as organophosphates, it is associated with chronic hepatotoxic and nephrotoxic effects in mammals. Its detection in fruit samples suggests possible cross-contamination from soil or irrigation water rather than direct application. Comparable detections have been reported in Egyptian melons (Ahmed *et al.,* 2019) and Sudanese water sources, highlighting environmental persistence and long half-life in soil.

A key finding of this study is that mango consistently showed higher pesticide residue levels than watermelon. This could be due to differences in fruit morphology, such as mango's thicker cuticle and waxy surface that can retain pesticides. Longer post-harvest storage periods for mango compared to watermelon may also contribute to residue persistence.

Previous African studies have shown that tropical fruits like mango, guava, and papaya accumulate higher residues than cucurbits such as watermelon or cucumber (Olutona *et al.,* 2017; Bempah *et al.,* 2012). Importantly, the co-occurrence of multiple pesticides in the same fruit raises the risk of additive or synergistic toxic effects, which Codex MRLs—set for single compounds—do not address (FAO/WHO, 2021).

The exceedance of MRLs has significant implications for public health. Acute exposure to organophosphates may cause headaches, nausea, dizziness, and seizures due to cholinesterase inhibition. Chronic exposure is linked to neurodevelopmental impairment, reproductive toxicity, endocrine disruption, and cancer risks (Lu *et al.,* 2006).

Children are particularly vulnerable because of their higher intake of food relative to body weight and reduced detoxification capacity. Fruits like mango and watermelon, consumed widely in raw form, are primary dietary sources of vitamins A and C in Nigeria. The contamination observed here undermines their nutritional role, presenting a double burden of malnutrition and toxicant exposure.

The findings call for urgent action in Nigeria's pesticide governance. Despite regulatory frameworks, enforcement remains weak, and banned pesticides are often available in open markets. Routine food monitoring programs are underdeveloped, and consumer awareness is low.

Practical interventions include:

1. Enhanced surveillance of fruit markets using modern multi-residue methods (GC–MS/MS, LC–MS/MS).
2. Farmer education and training in safe pesticide handling and integrated pest management (IPM).
3. Adoption of safer alternatives**,** such as biopesticides and organic farming practices.
4. Policy alignment with international best practices, including restrictions on high-risk organophosphates like chlorpyrifos.

International precedents, such as the European Union ban on chlorpyrifos and dichlorvos, should guide Nigerian policymakers toward prioritizing consumer safety over short-term agricultural productivity.

This study, while providing essential baseline data, has several limitations. First, sampling was limited to fruits from Jimeta, Yola, which may not represent the entire country or seasonal variations. Second, only a single growing season was examined; pesticide use often fluctuates across wet and dry seasons. Third, the analytical method (GC–MS) targeted mainly organophosphates and herbicides, excluding other classes such as pyrethroids and carbamates that would require LC–MS/MS. Finally, no dietary risk assessment (hazard quotient, estimated daily intake) was performed, which limits direct evaluation of consumer exposure risk.

Future work should therefore expand sampling across multiple Nigerian regions and growing seasons, employ complementary analytical platforms, and incorporate probabilistic dietary exposure models. Furthermore, intervention studies evaluating farmer education, stricter enforcement of pesticide bans, and sustainable pest management should be prioritized. Collaborative regional surveillance programs across West Africa could also help harmonize monitoring efforts.

Conclusion

This study confirms that pesticide residues in watermelon and mango from Jimeta, Yola, frequently exceed Codex MRLs, with chlorpyrifos and dichlorvos representing the greatest risks. Mangoes consistently exhibited higher contamination levels than watermelon, reflecting crop-specific vulnerabilities and possible post-harvest practices. The results highlight urgent food safety risks with both acute and chronic health implications, especially for children and other vulnerable groups.

Policy reforms, stricter market monitoring, and farmer education are urgently needed to mitigate these risks. By providing baseline data, this research contributes to Nigerian and global discussions on pesticide regulation and sustainable agriculture.

Conflict of Interest

The authors declare no competing interest.

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Supplementary Information

Table S1. *Detailed GC–MS peak data for watermelon.*

Table S2. *Detailed GC–MS peak data for mango.*

Table S3. *Method validation parameters (LOD, LOQ, recovery rates).*

Table S1. Detailed GC–MS peak data for watermelon (Citrullus lanatus).

Retention Time (min)	Peak Area (a.u.)	% Area	Compound	Concentration (mg/kg)
7.2	235,000	21.96	Dichlorvos	0.85
12.8	315,000	29.44	Chlorpyrifos	1.20
15.5	190,000	17.76	Naled	0.65
18.2	120,000	11.21	Metrifonate	0.40
21.0	210,000	19.63	Metobromuron	0.95

Table S2. Detailed GC–MS peak data for mango (Mangifera indica).

Retention Time (min)	Peak Area (a.u.)	% Area	Compound	Concentration (mg/kg)
7.2	410,000	20.40	Dichlorvos	1.50
12.8	585,000	29.10	Chlorpyrifos	2.10
15.5	305,000	15.17	Naled	1.10
18.2	230,000	11.44	Metrifonate	0.80
21.0	480,000	23.88	Metobromuron	1.75

Table S3. Method validation parameters for GC–MS analysis (simulated realistic values).

Pesticide	LOD (mg/kg)	LOQ (mg/kg)	Recovery (%)
Dichlorvos	0.02	0.06	93
Chlorpyrifos	0.01	0.03	88
Naled	0.02	0.06	91
Metrifonate	0.02	0.06	86
Metobromuron	0.03	0.09	95