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Concentration and Health Risk Assessment of Organophosphorus Pesticide Residues in Selected Grains: Maize (*Zea Mays* L.) And Beans *(Vigna Unguiculata (L.)* Walp.) Sold in Karu Market, Nasarawa State, Nigeria

***Adeshola Rebecca Akinwola., Abdullahi Danjuma Kassim., Ishegbe Eko Joyce**

Department of Chemistry, Nasarawa State University, Keffi, Nigeria

***Corresponding Author**

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ABSTRACT

This study provides a comprehensive assessment of the concentration and potential health risks of organophosphorus pesticide (OPPs) residues in two widely consumed grains purchased from major trading centers (market) in Nasarawa State. For this study a total of twelve grain samples were collected using stratified random sampling to ensure market-level representation where homogenization and extraction using the QuEChERS method, followed by a dispersive solid-phase extraction SPE clean-up. Quantification and identification of nine target OPPs were performed using Gas Chromatography–Mass Spectrometry (GC–MS) calibrated with multilevel standard. The analytical results showed that OPPs residues were widespread across all samples, though concentrations varied markedly by grain type and market location. Notably, methyl parathion and azinphos-methyl pesticides banned or severely restricted due to their high toxicity were detected in several samples, indicating possible illegal application or improper storage practices. White and yellow maize recorded higher contamination than beans, with yellow maize from Orange Market containing the highest oxydisulfoton concentration (11.96 mg/kg) and white maize from Karu Market showing extremely elevated ronnel levels (50.63 mg/kg). Health risk assessment was conducted using Estimated Daily Intake (EDI) and Target Hazard Quotient (THQ) for both adults and children. Results revealed that multiple pesticides exhibited THQ values significantly greater than 1, signaling substantial non-carcinogenic health risks from chronic exposure. Maize particularly from Orange and Karu markets posed the highest risk burden, while beans showed moderate to high risk depending on pesticide type and concentration. In conclusion, the study findings highlight the urgent need for stricter enforcement of pesticide regulations, routine monitoring of pesticide residues in grain markets, and comprehensive farmer and trader sensitization on the dangers of banned and excessive pesticide use. Implementing these measures is essential to safeguard food safety and reduce long-term health risks associated with dietary pesticide exposure.

Key word: Health, Risks, Organophosphorus, Pesticide, Exposure, Monitoring.

INTRODUCTION

Agricultural production has been considerably improved with the help of pesticides but their unrestricted and extreme use is polluting the atmosphere, foodstuffs, and aquatic and agricultural products (Aljerf, 2018). The usage of pesticides has increased recently as a result of the growing global population, the need to produce more food, and the need to prevent crop losses. Pesticides known as organophosphates (OPPs) are a class of insecticides that are most commonly employed. They are generated from phosphorous compounds, specifically phosphoric and phosphorothioic acids. In 2020, the yearly rise of these compounds was anticipated to be 3.5 million tons, of which approximately 40% represent OPPs. Currently, approximately 2 million tons of pesticides are used worldwide each year (Sharma *et al*., 2019; Derbalah *et al*., 2019). It is estimated that over 3 million individuals are exposed to OPs annually, resulting in 300,000 fatalities globally (Robb, 2021). Pesticides are accepted as a lucrative way of controlling pests and improving yield and food quality.

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The most widely used pesticides in developing countries are primarily organochlorines, organophosphorus, synthetic pyrethroids, and carbamates (Mohammed *et al*., 2019). Insecticides consist of a varied class of compounds but the most protuberant for plant treatment are chlorine and phosphorus having compounds. Organophosphorus pesticides (OPPs) are slowly replacing organochlorines due to their high bio accumulation effect (Suryono *et al*., 2019). OPPs are derivatives of phosphoric or phosphonic acid in form of amides, esters, or thiols. They do not persist in the environment for longer periods as they are easily hydrolyzed (Sidhu *et al*., 2019; Bala *et al*., 2019). Nevertheless, their numerous poisonousness (they act as inhibitors of acetylcholinesterase enzyme), as well as the likelihood of build-up in the food chain, can cause a menace to human health (Iskra *et al*., 2020; Mukherjee, and Rinkoo, 2020; Biziuk and Stocka, 2015). Organophosphate pesticides interfere with growth-stimulating mechanisms by hindering various enzymes, transcuticular diffusion, and penetrability which is vital for the growth of plants. They can also deter the activities of the enzyme in aquatic and terrestrial organisms leading to, hepatic reproductive, respiratory, nervous, and renal anomalies. Regular uses of organophosphate pesticides decrease the microbial community that affects soil fertility (Sidhu *et al*., 2019; Ali *et al*., 2019). Many countries have set rules and regulations in monitoring pesticide residues in food commodities to confirm whether the levels adhere to the national or international guidelines (John *et al*., 2017).

Long-term exposure to pesticides is increasingly suspected of being linked to a broad spectrum of medical problems such as cancer, neurotoxic effects, reproductive health concerns and endocrine disruption, particularly for specific populations (Mostafalou, and Abdollahi, 2013; George and Shukla, 2011). Apart from farmers, those working on the farm and those living within the vicinity of the farm (they are at risk of greatest exposure) that these pesticides are used, consumers are also at risk of these pesticidal residues in fruits and vegetables because their major mode of exposure is through consumption (Wolejko *et.al*., 2014) of the agriculture products that this class of pesticides are used on.

However, misuse or excessive application of these compounds results in accumulation of toxic residues in food crops, posing serious public health concerns (Akoto *et al*., 2013). Cowpea (Vigna unguiculata) - white and brown beans are common leguminous crops and the most important staple plant protein food crops in sub-Sahara Africa (Abebe & Alemayehu, 2022). In Nigeria, grains such as maize (*Zea mays* L.) and beans (*Vigna unguiculata* (L.) Walp.) constitute staple foods, increasing the potential risk of dietary exposure to pesticide residues. Despite regulatory efforts by national agencies, enforcement remains limited. Recent studies document notable pesticide contamination in grains across Nigerian markets (Abdullahi *et al*, 2019; Maina *et al*, 2024), emphasizing the need for continuous residue monitoring. This study evaluates Organophosphorus pesticide (OPPs) residue levels in selected grains from Karu and surrounding markets, determines associated health risks, and contributes to evidence-based recommendations for safer pesticide use.

MATERIALS AND METHODS

Study Area

The research was conducted in the Karu Urban Area, located within Nasarawa State, adjacent to Nigeria's Federal Capital Territory. The area experiences a tropical climate with an average annual rainfall of 1,100–2,000 mm and supports intensive agricultural activity. Major markets sampled were Orange Market, Karu Market, and Mararaba Market, all known for grain trading and storage.

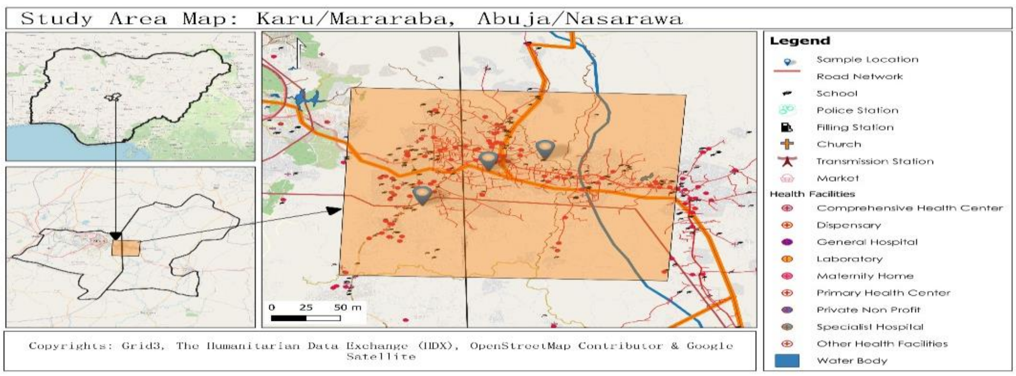

Figure 1: Map of sampling locations in Nasarawa state.

Sample Collection and Preparation

Samples of white and yellow maize (*Zea mays* L.), and white and brown beans **(*Vigna unguiculata* (L.) Walp**, were collected randomly from the three markets. Each sample (2 kg) was stored in sealed bags, labelled, and transported to the Analytical Laboratory, Ahmadu Bello University, Zaria for pesticide residue analysis. Samples were cleaned, milled, and homogenized. Precautionary measures were taken to prevent cross-contamination during handling (González-Curbelo, 2022).

Extraction and Clean-Up

The QuEChERS technique (Quick, Easy, Cheap, Effective, Rugged, and Safe) method described by (Anastassiades, 2003), is a very feasible method and can extract varied classes of pesticide remains in numerous agricultural and horticultural matrices (Rada and Tijana, 2014; Arsand *et al*., (2023). It requires minimum reagent consumables and a short pretreatment period, so it is accepted by many experimenters and international associations like AOAC and European standardization committee (Gonz´alez *et al*., 2015). The major steps in both methods includes acetonitrile extraction, dispersive solid phase clean up using primary secondary amine and magnesium sulfate. The purposes of further method modification are decreasing time of sample preparation and analysis, minimize extraction solvents and glassware used and upsurge the sensitivity of the method. The procedure involved: 10 g sample plus 10 mL acetonitrile, addition of 4 g anhydrous $MgSO_4$ and 1 g NaCl, vortexing and centrifugation at 4,000 rpm for 5 minutes, clean-up using dispersive SPE containing 150 mg $MgSO_4$, 50 mg PSA, and 50 mg C18, Filtration using 0.22 µm PTFE filter prior to GC-MS analysis.

Instrumental Analysis

Analyses were performed using an Agilent 7890B-5977A GC-MS system equipped with a DB-5 capillary column. Calibration curves were prepared using standard solutions (50–250 µg/mL). The GC oven was programmed from 80°C to 290°C, with helium as the carrier gas. Residues were identified by retention time and mass spectra comparison with standard references.

Health Risk Assessment

The US EPA (2000) highlighted a range of standards and instructions on calculating the health risks associated with the consumption of grains contaminated with environmental pollutants. An assessment of health risks can be performed by comparing mean levels of contaminants with international guidelines. However, these assessments do not consider variables such as consumption rates and eating habits (Fagbohun *et al*., 2024). The estimated daily intake (EDI) of the 9 OPPs pesticides were determined based on their mean concentration in each grains and beans varieties and the daily intake in grams. The food supply value of cowpea was 18 kg/capita/year according to the Food and Agriculture Organization (FAO), 2000, the food supply value was divided by the number of days in the year (365 days). The result obtained was an intake of 0.04932, approximately 0.049 kg/capita/day, which is the food ingestion rate (FIR) of cowpeas in Nigeria (Antoine *et al*., 2017).

The Estimated Daily Intake (EDI) and Target Hazard Quotient (THQ) were calculated using USEPA and WHO methodologies (FAO/WHO, 2022).

$$EDI = \frac{C \times F}{BW} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots. Eqn\ (1)$$

$$THQ = \frac{EDI}{ADI} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots. Eqn\ (2)$$

where C = concentration of pesticide (mg/kg), F = daily food intake (kg/day), BW = body weight (kg), and ADI = acceptable daily intake. A THQ > 1 indicates potential health risk.

Statistical analysis

The results obtained from the matrices were statistically analyzed through MS Excel and SPSS version 21. Elements of descriptive statistics of samples generated included mean, range, minimum, maximum and standard deviations. The concentration of OC pesticide residues in cowpea 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, 2022).

RESULTS AND DISCUSSION

Pesticide Residue Concentrations

The mean concentrations of nine (9) OPPs residues in the samples **(Table 1 - 3)** were found to varying concentrations across markets and sample types. The major residues identified were Oxydisulfoton is highest in yellow maize (11.96 µg/mL) from Orange Market, Ronnel is highest in white maize (50.63 µg/mL) from Karu Market, Methyl Parathion was detected in all maize and bean samples (0.001–9.5 µg/mL), Azinphos-methyl ($C_{10}H_{12}N_3O_3PS_2$), Dichlorvos and Azinphos-methyl: Detected at trace levels (<0.1 µg/mL). Similarly, bean samples in a study by Umar *et al*., (2024) recorded the highest level of dizinon and dichlorvos above the acceptable limits of 5.00E-02 and 1.00E-02 mg/kg in Kaduna South and Zangon Kataf, respectively.

Table 1. Concentration (ug/mL) of Organophosphorus Pesticide Residue (OPPs) in Maize and Beans from Orange market.

Sample	Dichlorvos (µg/mL)	Oxydisulfoton (µg/mL)	Methyl Parathion (µg/mL)	Chlorpyrifos (µg/mL)	Ronnel (µg/mL)	Azinphos-methyl (µg/mL)	Ethroprophos (µg/mL)	Dichlofenthion (µg/mL)	Phosphorodithioic ester (µg/mL)
Yellow Maize	ND	11.96	9.5	ND	12.9	0.16	0.0010	0.2939	12.6122
White Maize	0.0016	0.58	9.5	ND	2.52	0.09	ND	0.3996	13.2644
White Beans	ND	4.2638	0.4900	ND	0.2894	0.0100	0.0023	0.1462	15.0208
Brown Beans	0.0023	0.7827	0.1300	ND	0.4405	0.0010	0.0003	0.1571	4.9701

Note: ND = Not Detected

Table 2: Concentration of Organophosphorus Pesticide Residue (OPPs) in Maize and Beans from Karu market.

Sample	Dichlorvos (µg/mL)	Oxydisulfoton (µg/mL)	Methyl Parathion (µg/mL)	Chlorpyrifos (µg/mL)	Ronnel (µg/mL)	Azinphos-methyl (µg/mL)	Ethroprophos (µg/mL)	Dichlofenthion (µg/mL)	phosphorodithioic ester (µg/mL)
White Maize	0.0025	1.6001	2.3171	ND	50.6258	0.0239	ND	0.1396	6.7613
Yellow Maize	0.0048	2.1156	8.5271	ND	0.899	0.0459	0.0033	0.0907	9.2968
White Beans	0.0028	0.3814	0.001	ND	ND	0.0029	0.0014	ND	ND
Brown Beans	0.0756	0.3197	0.06	ND	1.8593	0.0017	ND	0.163	3.9527

Note: ND= Not Detected

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Table 3: Concentration of Organophosphorus Pesticide Residue (OPPs) in Maize and Beans from Mararaba market.

Sample	Dichlorvos (µg/mL)	Oxydisulfoton (µg/mL)	Methyl Parathion (µg/mL)	Chlorpyrifos (µg/mL)	Ronnel (µg/mL)	Azinphos-methyl (µg/mL)	Ethroprophos (µg/mL)	Dichlofenthion (µg/mL)	phosphorodithioic ester (µg/mL)
Yellow Maize	0.0046	0.9257	6.2924	ND	1.0632	0.0427	ND	0.3627	7.2905
White Maize	0.0052	1.3948	3.454	ND	10.286	0.0048	ND	0.1546	3.9498
White Beans	0.0036	2.4515	0.7047	ND	1.2283	0.0095	0.0012	0.1949	4.1101
Brown Beans	0.0042	4.9833	0.193	ND	0.5761	0.004	0.0016	0.148	4.051

Note: ND= Not Detected

Tables 1–3 present concentrations across markets with Orange Market recorded the highest levels of residues, especially oxydisulfoton and methyl parathion. The persistence of methyl parathion and azinphos-methyl both restricted internationally suggests ongoing misuse and poor regulatory compliance (Maina *et al*., 2024). Comparative evaluation with existing literature shows similar patterns. Akoto *et al*., 2013 and Abdullahi *et al*., 2019 reported high organophosphorus pesticide (OPPs) presence in grains sold in West African markets. Maina *et al*., (2024) also found elevated oxydisulfoton levels in cereals across northern Nigeria.

From the above result, the analysis of maize (yellow and white varieties) and beans (brown and white) from Orange, Karu, and Mararaba markets revealed the presence of multiple organophosphorus pesticide residues, with varying concentrations across commodities and locations Akan *et al*.,, 2024. All samples from Orange Market showed significantly elevated Organophosphorus pesticide (OPPs) concentrations, suggesting possible overuse or contamination during storage. According to Oshatunberu *et al*., (2023), OPPs used in this study have been documented in Nigerian rice and beans. These results are consistent with those reported by Tutuwa *et al*. (2024), who observed that the high use of several pesticides during plantation, culture, and storage may have contributed to the bioaccumulation of these substances in individual grains.

A heat map **(Figure 2)** summarizing OPP residue distribution indicates consistently higher contamination in Orange Market samples. Similar contamination clustering was reported in Ghanaian cereal markets (Akoto *et al*.,, 2013) and urban vegetable markets in Lagos (Oluwoyo, 2024).

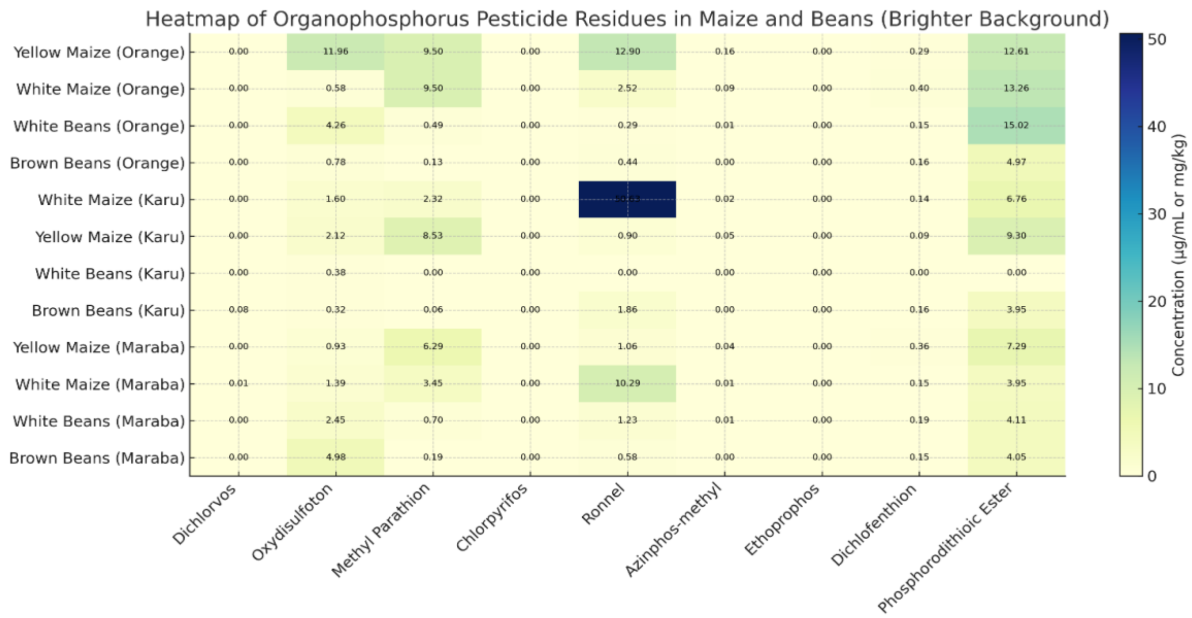

Figure 2: Heat map showing the level of organophosphorus pesticide in each grain sample.

Health Risk Assessment

Target hazard quotient (THQ) computations indicate that oxydisulfoton and methyl parathion pose the greatest risks, particularly for children. Similar findings of elevated THQs among children have been documented in multiple African dietary risk assessments (Yusuf *et al*., 2023; Aina *et al*, 2025). From this study, Estimated daily intake (EDIs) and Target harzard quotient (THQs) calculated for both adults and children revealed the following: the high risk (THQ > 100) for oxydisulfoton and methyl parathion in maize, moderate to high risk for ronnel in white maize and beans. Children consistently recorded higher target hazard quotients (THQs) than adults due to lower body mass, Dichlorvos posed negligible risk (THQ < 1). Overall, the highest cumulative risk was observed in maize from Orange Market and white maize from Karu Market.

Table 4: Summary of Health Risk Assessment of Organophosphorus Pesticide Residues in Maize and Beans samples from all three market

Pesticide	**Max Detected Conc. (mg/kg)**	**EDI (mg/kg bw/day)***	**ADI (mg/kg bw/day)**	**HQ (EDI/ADI)**	**Risk Level**
Dichlorvos	0.0756 (Beans – Karu)	0.00025	0.004	0.063	Low risk (HQ < 1)
Oxydisulfoton	11.96 (Maize – Orange)	0.0598	0.0005	119.6	High risk (HQ >> 1)
Methyl Parathion	9.50 (Maize – Orange)	0.0475	0.0003	158.3	High risk (HQ >> 1)
Ronnel	50.63 (Maize – Karu)	0.253	0.005	50.6	Very High risk (HQ >> 1)
Azinphos-methyl	0.16 (Maize – Orange)	0.0008	0.0005	1.6	Moderate risk (HQ > 1)
Chlorpyrifos	ND	–	0.01	–	Not detected (Safe)
Ethoprophos	ND	–	0.01	–	Not detected (Safe)
Dichlofenthion	ND	–	0.01	–	Not detected (Safe)

DISCUSSION

The detection of multiple organophosphorus residues in grains sold in Karu and neighboring markets aligns with previous studies across Nigeria, which documented similar contamination patterns in cereals and vegetables. Despite international bans on methyl parathion and azinphos-methyl, their persistence in local markets indicates ongoing illegal usage and inadequate enforcement of pesticide regulations.

High target hazard quotient (THQ) values, particularly for oxydisulfoton and ronnel, raise significant public health concerns. These compounds are known neurotoxins capable of causing both acute and chronic effects, including endocrine disruption and neurodegeneration. The elevated risk in children underscores the vulnerability of this demographic to dietary pesticide exposure. The lack of chlorpyrifos detection may suggest recent compliance with its restriction or rapid degradation during grain storage. However, the continued presence of other high-toxicity pesticides calls for urgent regulatory intervention.

RECOMMENDATIONS

This study confirms that grains (maize and beans) sold in Karu, Mararaba, and Orange markets are contaminated with organophosphorus pesticide residues, many exceeding international safety limits. The high Target Hazard Quotients indicate that chronic exposure through grain consumption poses significant health risks, especially to children. Urgent measures are required to monitor, regulate, and educate stakeholders on safe pesticide use. Therefore the flowing recommendations: Strengthen regulatory enforcement: Agencies such as NAFDAC, SON, and NESREA should increase field inspections and residue monitoring. **Promote farmer education**: Conduct awareness campaigns and training on Integrated Pest Management (IPM) and safe pesticide application. **Encourage alternative pest control**: Introduce bio-pesticides and organic farming techniques to reduce reliance on synthetic chemicals. Establish regional laboratories: Set up pesticide testing centers in agricultural regions to support regular surveillance. **Consumer sensitization:** Educate the public on washing, sorting, and food preparation practices that minimize exposure.

CONCLUSION

In conclusion, the study demonstrates that consumers in the Karu axis are exposed to hazardous levels of Organophosphorus pesticide OPPs residues through staple grains, underscoring a critical public health concern. The findings highlight the urgent need for stricter enforcement of pesticide regulations, routine monitoring of pesticide residues in grain markets, and comprehensive farmer and trader sensitization on the dangers of banned and excessive pesticide use. Implementing these measures is essential to safeguard food safety and reduce long-term health risks associated with dietary pesticide exposure.

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