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Concentrations and Health Risk Assessment of Organophosphorus Pesticides Residues in Common Cereal Samples from Ngalda Agricultural Area, Yobe State, Northeastern Nigeria

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ABSTRACT
Organophosphorus pesticides (OPPs) are a well-known class of man-made chemicals frequently used to protect crops against pests giving rise to accumulation of these chemicals in foods. In the present study, the residual levels and potential health risk associated with four (4) organophosphorus pesticides (diazinon, dichlorvos, chlorpyrifos and fenitrothion) in cereals (maize, millet, wheat and rice) samples collected from Ngalda agricultural area, Fika Local Government Area, Yobe State were analyzed. Extractions and clean-up of the samples were carried out using standard procedures. The levels of the OPPs in cereals samples were determined using GC-MS coupled with flame photometric detector. The analysis of OPPs residues in maize, millet, wheat and rice revealed that the residual levels of the studied OPPs were highest in maize samples ranging from 3.97E-01 – 6.83E-01 mg/kg. On the contrary, rice samples had the least concentrations range of 1.09E-02 - 1.06E-01 mg/kg. The detected OPPs in all the maize, millet and wheat samples exceeded corresponding maximum residue limits (MRLs). However, the levels in rice samples were all lower than the recommended MRLs. The hazard index (HI) through consumption of maize for adults (1.60E+01 - 2.23E+03) and children (2.87E+01 - 3.99E+03); millet for adults (1.11E+01 - 5.96E+02) and children (1.99E+01 - 1.07E+03); wheat for adults (7.72E+00 - 1.71E+03) and children (1.39E+01 - 4.20E+01); and rice for adults (1.46E+00 - 3.61E+02) and children (2.62E+00 - 6.47E+02). HI values were all > 1, suggesting that consumption of these cereals by both age groups from the study area might pose danger of potential health risk. Furthermore, higher risk indices were recorded for children through crop consumption than adults.

Keywords: Maize, Millet, Ngalda, Organophosphates, Rice, Risk assessment, Wheat

INTRODUCTION

Agriculture has traditionally been a vital part of the Nigerian economy, employing over 70 % of the population and providing a means of subsistence (NOUN, 2016). There are biotic and abiotic components to Nigeria's agricultural difficulties. Abiotic factors include pests and diseases that harm crops in storage or the field, reducing crop yield and causing low productivity. Since the 1940s, pesticides have been used more frequently to address this problem (Kolani *et al.*, 2017).

Food losses can be significantly attributed to pests, and controlling them is essential to attaining food security throughout all geographical ranges. The use of pesticides has led to control of weeds, diseases, pests, and various plant pathogens, giving rise to significant reduction in crop losses and increased food production while upholding high-quality yields (Iya and Kwaghe, 2007). Considerable attention has been directed towards the health risks associated with pesticide residues in food, despite the extremely stringent control procedures used in the production of pesticides to guarantee logical certainty and little impact on human health and the environment (Damalas and Eleftherohorinos, 2011).

Pesticides are classified into several chemical classes, with organochlorines, organophosphates, carbamates, and pyrethroids as the most prevalent (Ogah and Coker, 2012). Organochlorine pesticides have been in use for long time but due to their long persistence, they have been banned for agricultural practices in many countries. This measure has led to the use of alternative pesticides with lower persistence like organophosphate and carbamates (Fantunsi *et al.*, 2020). Despite their advantages over organochlorine pesticides, organophosphates are hazardous chemicals with negative impacts upon accumulation in the food chain and persistent in long-term risks to human and animal health, as well as negative effects on the environment (Akinneye *et al.*, 2006; Leong *et al.*, 2007). These dangers are increased by improper storage procedures, poorly maintained or completely incorrect spraying equipment, and ineffective application methods (Al-wabel *et al.*, 2011). It may not be possible to completely prevent pesticide residues in plants, even when they are applied in accordance with proper agricultural procedures (Iya and Kwaghe, 2007). OPPs can inhibit the activity of cholinesterase, leading to overstimulation of neurotransmission. Exposure to OPPs was associated with a range of adverse health effects, including neurological function disorder and the male reproductive system. Some studies reported that OPs exposure could be affected by childhood IQ and brain anomalies. Moreover, they can affect the endocrine system and prostate cancer (Naksen *et al.*, 2023). Human exposure route to pesticides occurs mainly via dermal absorption, inhalation and oral ingestion. Generally, ingestion of pesticides is often from residues in food (Fantunsi *et al.*, 2020).

Grains which consist of Cereals and legumes generally constitute the most vital diet component for the majority of people in the world by providing the calories and protein consumed majorly by poor people (Saheed *et al.*, 2020).

Ngalda is primarily agriculture area with excessive usage of pesticides to enhance the production of vegetables, cereals and fruits, as well as the control of pests. Cereals from Ngalda agricultural area also constitute an important source of food for the inhabitants in and around Fika Local Government in Yobe State, and farming cereal is a major source of income for the inhabitants. However, the agricultural activities have impacted negatively on the soil because bioaccumulation and bio concentration of the pesticides in the cereals are capable of reaching toxic levels even at low exposure.

Limited studies on pesticide residues in cereals have been carried out in this part of Nigeria (Atsen *et al.*, 2021). To the best of our knowledge, no studies have been carried out in Ngalda agricultural area despite the fact that pesticides of high toxicity are used through ground application on the farms. The aim of this research is therefore to quantify the levels of organophosphorus pesticide residues in some common cereals (maize, wheat, millet and rice) grown in the area and assess the human health risk associated with their consumption.

MATERIALS AND METHODS

Study Area

Ngalda is located in Fika Local Government Area, Yobe State, Nigeria (Fig. 1). It is located on Latitude 11° 6' 23.19" Nand Longitude 11° 22' 27.52" E, and it is about 79 kilometres from Potiskum Local Government Area in Yobe State, bordering with Gombe State. Ngalda is an agricultural and commercial community. Rice, wheat, onion maize, millet, beans, tomatoes, pepper, red bell pepper, and other crops are grown in the area.

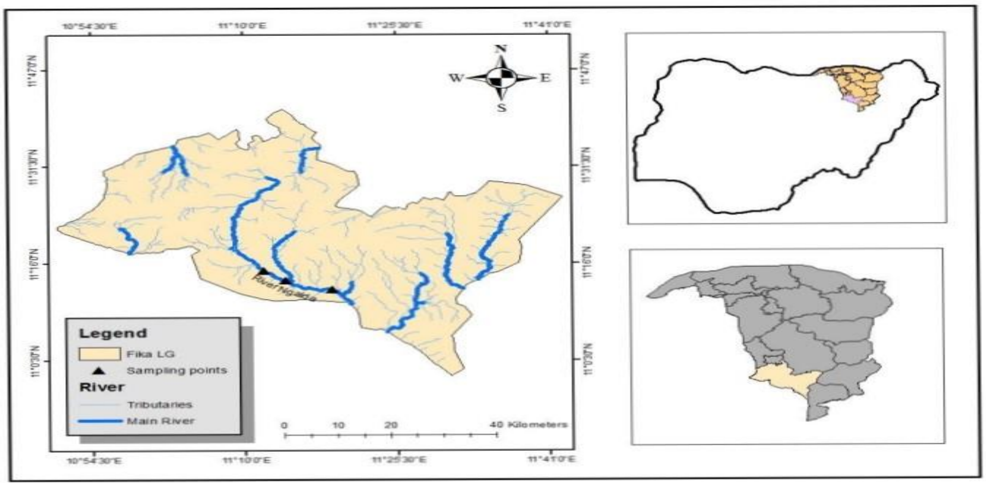

Fig. 1: Map of Fika local government showing sampling location

Sample Collection

Cereal samples which are maize (*Zea mays*), millet (*Pennisetum glaucum*), wheat (*Triticum aestivum*), and rice (*Oryza sativa*), were collected from four different farm locations in Ngalda agricultural area, Fika Local Government Area, Yobe state. To keep the samples safe from moisture and contamination, they were kept in specific bottles with tight lids. After that, the samples were stored in the refrigerator at a temperature of 4 °C.

Samples Preparation

To ensure the samples were clean, stones and other extraneous materials were removed. After thoroughly mixing each sample to produce an appropriate homogenate, 200 g of the mixture was removed and ground to a particle size of 20 mesh. After milling, the samples were appropriately labelled and stored in glass bottles at a temperature of 4 °C. Additionally, 200 g portions of the samples were preserved as whole grains in labelled glass bottles in the refrigerator for backup.

Sample Extraction

Sample (1 g) was extracted with 10 mL of acetonitrile. The mixture was mixed at high speed with vortex mixer for 1 minutes. Exactly 0.1 g NaCl and 0.2 g of activated anhydrous $MgSO_4$ were added to the mixture, and mixing was continued for an additional 1 minute. The mixture was then centrifuged for 5 minutes at 5000 rpm at 5 °C. The supernatant was transferred to a 15 mL tube containing 2 g of $MgSO_4$. The mixture was then shaken for 1 minute, centrifuged for 5 minutes at 5000 rpm. The supernatant (4 mL) was transferred to a 5 mL vial and evaporated to dryness. The residue was reconstituted by acetonitrile to obtain 1 mL solution, and after shaking for 3 minutes, 2 µL of the solution was injected in to gas chromatograph for the analysis (Usher and Majors, 2012).

Determination of Pesticides Residues

The Agilent GCMS model GC 7890B and MSD 5977A utilized helium as the carrier gas, maintaining a constant flow rate of 1 mL/min. The initial oven temperature was set to 80 °C for 3 minutes, followed by a ramp at a rate of 15 °C/min up to 290 °C, where it was held for 5 minutes. The injection port temperature was adjusted to 250 °C, and a split less injection mode was employed. Upon acquiring the total ion chromatogram for the mixed stock standard solutions in scan mode, peaks were identified based on their retention time and mass spectra. The ion with the highest signal-to-noise ratio and no chromatographic interference was chosen for quantification purposes (Usher and Majors, 2012).

Health Risk Assessment of OPPs

Estimated Daily Intake (EDI)

The EDI depend on both OPPs levels and the daily consumption rate of cereal (Akoto *et al*., 2013). The EDI (mg kg^{-1} d^{-1}) was calculated using Equation (1).

$$EDI = \frac{C\ X\ C_{On}}{Bw} \qquad (1)$$

where *C* (mg kg^{-1}) is average OPPs concentrations on dry basis in Cereal, C_{on} (mg kg^{-1}) is the daily average consumption, *Bw* (kg $person^{-1}$) represents body weight. Average body weights for adults and children was 60 kg and 15 kg respectively.

Target hazard quotient (THQ)

According to standard USEPA methods, the risk of non-carcinogenic effect is expressed as the ratio of the dose resulting from exposure to site media compared to a dose that was believed to be without risk of effects (Darko and Akoto 2008; USEPA, 2007). This ratio was referred to as THQ. The THQ for consumer through cereal can be assessed by food chain and the acceptable daily intake (ADI) for OPPs. The applied ADI is obtained from National food safety standard-Maximum residue limits for pesticides in food (2019). The THQ was calculated using Equation (2).

$$\text{THQ} = \frac{\text{EDI}}{\text{ADI}} \qquad (2)$$

If THQ is less than one, the exposed people are unlikely to experience obvious toxic effects. If it exceeded one, there is a possibility of obvious toxic effects, with an increasing probability as THQ increased.

Estimation of Short-term Intake (ESTI)

The ESTI (mg kg^{-1} d^{-1}) was calculated using Equation (3).

$$\text{ESTI} = \frac{\text{HR x F}}{\text{Bw}} \qquad (3)$$

where HR (mg kg^{-1}) is the OPPs highest residue on dry basis in cereal, *F* (kg $person^{-1}$ d^{-1}) is the daily highest consumption of the cereal in the region, Bw (kg $person^{-1}$) represents body weight. Based on the report by GEMS/FOOD (2012), the highest maize consumptions for adults and children were 242.0 and 108.5 g d^{-1}, respectively, and the average body weights for adults and children (1–6 years) were 60 and 15 kg, respectively.

Acute Hazard Index (aHI)

Acute Reference Dose (ARfD) values will be referred to a Joint FAO/WHO Meeting on Pesticide Residues. The aHI was calculated using Equation (4) (Li *et al.*, 2015).

$$aHI = \frac{ESTI}{\text{AR}f\text{D}} \qquad (4)$$

If aHI is less than one, the exposed people are unlikely to experience obvious toxic effects. If it exceeds one.

RESULTS AND DISCUSSION

Table 1 presents the concentrations (mg/kg) of organophosphorus pesticide residue (OPPs) in maize samples obtained from Ngalda agricultural area. The residual levels in the four (4) samples are as follows: diazinon (2.81E-01 - 8.71E-01 mg/kg), dichlorvos (8.50E-02 - 1.01E+00 mg/kg), chlorpyrifos (2.73E-01 - 5.30E-01 mg/kg) and fenitrothion (3.50E-01 - 9.73E-01 mg/kg). The mean residual levels are as follows: diazinon (6.05E-01 mg/kg), dichlorvos (5.52E-01 mg/kg), chlorpyrifos (3.97E-01 mg/kg) and fenitrothion (6.83E-01 mg/kg).

Table 2 shows the concentrations of OPPs residues in millet samples obtained from Ngalda area. The detection frequency of all OPPs was 100 % except chlorpyrifos which has 75 %. The levels ranged: diazinon (2.50E-02 - 4.71E-01 mg/kg), dichlorvos (1.27E-01 - 1.67E-01 mg/kg), chlorpyrifos (ND - 4.80E-01 mg/kg) and fenitrothion (1.39E-01 - 5.80E-01 mg/kg). The mean concentrations of the OPPs are as follows: diazinon (1.51E-01 mg/kg), dichlorvos (1.48E-01 mg/kg), chlorpyrifos (2.76E-01 mg/kg) and fenitrothion (3.62E-01 mg/kg).

Table 3 shows the frequency of detection and concentration of the OPPs studied in wheat grains collected from Ngalda area. Overall, 100 % of wheat samples were detected with OPPs. The range of concentrations for the OPPs in the wheat samples was: diazinon (1.70E-02 - 6.31E-01 mg/kg), dichlorvos (3.90E-02 - 7.83E-01 mg/kg), chlorpyrifos (1.00E-02 - 7.23E-01 mg/kg) and fenitrothion (2.80E-02 - 6.87E-01 mg/kg). The mean concentrations were: diazinon (1.74E-01 mg/kg), dichlorvos (4.23E-01 mg/kg), chlorpyrifos (1.92E-01 mg/kg) and fenitrothion (1.91E-01 mg/kg).

Table 4 shows the residual concentrations of OPPs in rice samples from Ngalda area. The concentration of the studied OPPs across the sampling points are as follows: diazinon (3.40E-03 - 2.00E-02 mg/kg), dichlorvos (1.00E-02 - 1.80E-01 mg/kg), chlorpyrifos (2.70E-03 - 4.00E-01 mg/kg) and fenitrothion (3.00E-03 - 2.80E-02 mg/kg). The mean concentrations are as follows: diazinon (1.09E-02 mg/kg), dichlorvos (8.95E-02 mg/kg), chlorpyrifos (1.06E-01 mg/kg) and fenitrothion (1.60E-02 mg/kg).

Risk Assessment

Table 5 shows the estimated dietary intakes (EDI) and target hazard quotients (THQ) of the OPPs for adults and children of OPPs through maize consumption. The EDI of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were estimated to be 6.05E-01, 5.52E-01, 3.97E-01 and 6.83E-01 mg kg/day respectively for adults; and 2.42E+00, 2.21E+00, 1.59E+00 and 2.73E+00 mg kg/day for children. The target hazard quotients (THQ) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were 1.21E+02, 1.38E+02, 3.97E+01 and 1.14E+02 respectively for adults. For children, the THQ are 4.84E+02, 5.52E+02, 1.59E+02 and 4.56E+02 mg kg/day for diazinon, dichlorvos, chlorpyrifos and fenitrothion respectively. The hazard index (HI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were 8.13E+01, 2.23E+03, 1.60E+01 and 6.89E+01 respectively for adults. For children, the HI values were 1.46E+02, 3.99E+03, 2.87E+01 and 1.24E+02 mg kg/day for diazinon, dichlorvos, chlorpyrifos and fenitrothion respectively.

Table 6 shows the ADI, EDI, THQ, ESTI and HI of OPPs for children and adults. The dietary intakes (EDI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through millet consumption were estimated to be 1.51E-01, 1.48E-01, 2.76E-01 and 3.62E-01 mg kg/day respectively for adults; and 6.02E-01, 5.91E-01, 1.10E+00 and 1.45E+00 mg kg/day for children. The THQ of diazinon, dichlorvos, chlorpyrifos and fenitrothion through millet consumption were 3.01E+01, 3.69E+01, 2.76E+01 and 6.04E+01 respectively for adults. For children, the values were diazinon (1.20E+02), dichlorvos (1.48E+02), chlorpyrifos (1.10E+02) and fenitrothion (2.42E+02). The hazard index (HI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were 2.02E+01, 5.96E+02, 1.11E+01 and 3.65E+01 respectively for adults. For children, the HI values were 3.63E+01, 1.07E+03, 1.99E+01 and 6.55E+01 for diazinon, dichlorvos, chlorpyrifos and fenitrothion respectively.

Table 7 presents the ADI, EDI, THQ, ESTI, ARfD and HI of OPPs for children and adults. The dietary intakes (EDI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through wheat consumption were estimated to be 1.74E-01, 4.23E-01, 1.92E-01 and 1.91E-01 mg kg/day respectively for adults; and 6.97E-01, 1.69E+00, 7.66E-01 and 7.64E-01 mg kg/day for children. The THQ of diazinon, dichlorvos, chlorpyrifos and fenitrothion through wheat consumption were 3.49E+01, 1.06E+02, 1.92E+01 and 3.18E+01 respectively for adults. For children, the THQ were diazinon (1.39E+02), dichlorvos (4.23E+02), chlorpyrifos (7.66E+01) and fenitrothion (1.27E+02). The hazard index (HI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were 2.34E+01, 1.71E+03, 7.72E+00 and 1.93E+01 respectively for adults. For children, the HI values were 4.20E+01, 3.06E+03, 1.39E+01 and 3.45E+01 for diazinon, dichlorvos, chlorpyrifos and fenitrothion respectively.

Table 8 shows the ADI, EDI, THQ, ESTI, ARfD and HI of OPPs for children and adults. The dietary intakes (EDI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through rice consumption were estimated to be 1.09E-02, 8.95E-02, 1.06E-01 and 1.60E-02 mg kg/day respectively for adults; and 4.34E-02, 3.58E-01, 4.23E-01 and 6.40E-02 mg kg/day for children. The THQ of diazinon, dichlorvos, chlorpyrifos and fenitrothion through rice consumption were 2.17E+00, 2.24E+01, 1.06E+01 and 2.67E+00 respectively for adults. For children, the THQ were diazinon (8.68E+00), dichlorvos (8.95E+01), chlorpyrifos (4.23E+01) and fenitrothion (1.07E+01). The hazard index (HI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were 1.46E+00, 3.61E+02, 4.26E+00 and 1.61E+00 respectively for adults. For children, the HI values were 2.62E+00, 6.47E+02, 7.64E+00 and 2.89E+00 for diazinon, dichlorvos, chlorpyrifos and fenitrothion respectively.

Table 1: Concentrations (mg/kg) of Organophosphorus Pesticide Residue (OPPs) in Maize Samples from Ngalda Agricultural Area

Pesticides	Sampling points				Range	Mean	S.D	D. F.	MRL
	S1	S2	S3	S4				(%)	
Diazinon	2.81E-01	4.73E-01	8.71E-01	7.95E-01	2.81E-01 - 8.71E-01	6.05E-01	0.28	100	0.02
Dichlorvos	8.50E-02	9.70E-02	1.01E+00	1.01E+00	8.50E-02 - 1.01E+00	5.52E-01	0.53	100	0.2
Chlorpyrifos	2.73E-01	3.12E-01	4.73E-01	5.30E-01	2.73E-01 - 5.30E-01	3.97E-01	0.12	100	0.05
Fenitrothion	3.50E-01	5.31E-01	9.73E-01	8.79E-01	3.50E-01 - 9.73E-01	6.83E-01	0.29	100	0.01
Total	**9.89E-01**	**1.41E+00**	**3.33E+00**	**3.22E+00**		**2.24E+00**			

D. F. = Detection Frequency, S.D = Standard Deviation, MRL = Maximum Residue Limit (FAO/WHO, 2020)

Table 2: Concentrations (mg/kg) of Organophosphorus Pesticide Residues (OPPs) in Millet Samples from Ngalda Agricultural Area

Pesticides	Sampling points				Range	Mean	S.D	D. F.	MRL
	S1	S2	S3	S4				(%)	
Diazinon	2.50E-02	5.30E-02	4.71E-01	5.30E-02	2.50E-02 - 4.71E-01	1.51E-01	0.21	100	**0.02**
Dichlorvos	1.58E-01	1.67E-01	1.39E-01	1.27E-01	1.27E-01 - 1.67E-01	1.48E-01	0.02	100	**NA**
Chlorpyrifos	ND	4.80E-01	3.12E-01	3.10E-01	ND - 4.80E-01	2.76E-01	0.20	75	**0.05**
Fenitrothion	4.20E-01	3.10E-01	5.80E-01	1.39E-01	1.39E-01 - 5.80E-01	3.62E-01	0.19	100	**NA**
Total	**6.03E-01**	**1.01E+00**	**1.50E+00**	**6.29E-01**		**9.36E-01**			

D. F. = Detection Frequency, S.D = Standard Deviation, MRL = Maximum Residue Limit (FAO/WHO, 2020)

Table 3: Concentrations (mg/kg) of Organophosphorus Pesticide Residues (OPPs) in Wheat Samples from Ngalda Agricultural Area

Pesticides	Sampling points				Range	Mean	S.D	D. F.	MRL
	S1	S2	S3	S4				(%)	
Diazinon	6.31E-01	2.80E-02	2.10E-02	1.70E-02	1.70E-02 - 6.31E-01	1.74E-01	0.30	100	0.1
Dichlorvos	7.83E-01	6.31E-01	2.40E-01	3.90E-02	3.90E-02 - 7.83E-01	4.23E-01	0.34	100	0.1
Chlorpyrifos	7.23E-01	2.10E-02	1.00E-02	1.20E-02	1.00E-02 - 7.23E-01	1.92E-01	0.35	100	0.5
Fenitrothion	6.87E-01	2.80E-02	2.10E-02	2.80E-02	2.80E-02 - 6.87E-01	1.91E-01	0.33	100	5.0
Total	**2.82E+00**	**7.08E-01**	**2.92E-01**	**9.60E-02**		**9.80E-01**			

D. F. = Detection Frequency, S.D = Standard Deviation, MRL = Maximum Residue Limit (FAO/WHO, 2020)

Table 4: Concentrations (mg/kg) of Organophosphorus Pesticide Residues (OPPs) in Rice Samples from Ngalda Agricultural Area

Pesticides	Sampling points				Range	Mean	S.D	D. F. (%)	MRL
	S1	S2	S3	S4					
Diazinon	2.00E-02	1.00E-02	1.00E-02	3.40E-03	3.40E-03 - 2.00E-02	1.09E-02	0.00	100	0.1
Dichlorvos	1.80E-01	1.31E-01	1.00E-02	3.70E-02	1.00E-02 - 1.80E-01	8.95E-02	0.08	100	0.1
Chlorpyrifos	4.00E-01	1.00E-02	2.70E-03	1.00E-02	2.70E-03 - 4.00E-01	1.06E-01	0.20	100	0.5
Fenitrothion	1.00E-02	2.30E-02	3.00E-03	2.80E-02	3.00E-03 - 2.80E-02	1.60E-02	0.01	100	1.0
Total	**6.10E-01**	**1.74E-01**	**2.57E-02**	**7.84E-02**		**2.22E-01**			

D. F. = Detection Frequency, S.D = Standard Deviation, MRL = Maximum Residue Limit (FAO/WHO, 2020)

Table 5: Health Risk Estimation of Organophosphorus Pesticide Residue (OPPs) in Maize from Ngalda Agricultural Area

Pesticides	Conc. (mg/kg)	EDI		ADI (mg/kg bw)	THQ		ESTI		ARfD	HI	
		Adult	Children		Adult	Children	Adult	Children		Adult	Children
Diazinon	6.05E-01	6.05E-01	2.42E+00	0.005	1.21E+02	4.84E+02	2.44E+00	4.38E+00	0.03	8.13E+01	1.46E+02
Dichlorvos	5.52E-01	5.52E-01	2.21E+00	0.004	1.38E+02	5.52E+02	2.23E+00	3.99E+00	0.001	2.23E+03	3.99E+03
Chlorpyrifos	3.97E-01	3.97E-01	1.59E+00	0.01	3.97E+01	1.59E+02	1.60E+00	2.87E+00	0.1	1.60E+01	2.87E+01
Fenitrothion	6.83E-01	6.83E-01	2.73E+00	0.006	1.14E+02	4.56E+02	2.76E+00	4.94E+00	0.04	6.89E+01	1.24E+02

Table 6: Health Risk Estimation of Organophosphorus Pesticide Residue (OPPs) in Millet from Ngalda Agricultural Area

Pesticides	Conc. (mg/kg)	EDI		ADI (mg/kg bw)	THQ		ESTI		ARfD	HI	
		Adult	Children		Adult	Children	Adult	Children		Adult	Children
Diazinon	1.51E-01	1.51E-01	6.02E-01	0.005	3.01E+01	1.20E+02	6.07E-01	1.09E+00	0.03	2.02E+01	3.63E+01
Dichlorvos	1.48E-01	1.48E-01	5.91E-01	0.004	3.69E+01	1.48E+02	5.96E-01	1.07E+00	0.001	5.96E+02	1.07E+03
Chlorpyrifos	2.76E-01	2.76E-01	1.10E+00	0.01	2.76E+01	1.10E+02	1.11E+00	1.99E+00	0.1	1.11E+01	1.99E+01
Fenitrothion	3.62E-01	3.62E-01	1.45E+00	0.006	6.04E+01	2.42E+02	1.46E+00	2.62E+00	0.04	3.65E+01	6.55E+01

ADI Source: National Food Safety Standard (2019)

Table 7: Health Risk Estimation of Organophosphorus Pesticide Residues (OPPs) in Wheat from Ngalda Agricultural Area

Pesticides	Conc. (mg/kg)	EDI		ADI (mg/kg bw)	THQ		ESTI		ARfD	HI	
		Adult	Children		Adult	Children	Adult	Children		Adult	Children
Diazinon	1.74E-01	1.74E-01	6.97E-01	0.005	3.49E+01	1.39E+02	7.03E-01	1.26E+00	0.03	2.34E+01	4.20E+01
Dichlorvos	4.23E-01	4.23E-01	1.69E+00	0.004	1.06E+02	4.23E+02	1.71E+00	3.06E+00	0.001	1.71E+03	3.06E+03
Chlorpyrifos	1.92E-01	1.92E-01	7.66E-01	0.01	1.92E+01	7.66E+01	7.72E-01	1.39E+00	0.1	7.72E+00	1.39E+01
Fenitrothion	1.91E-01	1.91E-01	7.64E-01	0.006	3.18E+01	1.27E+02	7.70E-01	1.38E+00	0.04	1.93E+01	3.45E+01

Table 8: Health Risk Estimation of Organophosphorus Pesticide Residue (OPPs) in Rice from Ngalda Agricultural Area

Pesticides	Conc. (mg/kg)	EDI		ADI (mg/kg bw)	THQ		ESTI (mg kg^{-1} d^{-1})		ARfD	HI	
		Adult	Children		Adult	Children	Adult	Children		Adult	Children
Diazinon	1.09E-02	1.09E-02	4.34E-02	0.005	2.17E+00	8.68E+00	4.38E-02	7.85E-02	0.03	1.46E+00	2.62E+00
Dichlorvos	8.95E-02	8.95E-02	3.58E-01	0.004	2.24E+01	8.95E+01	3.61E-01	6.47E-01	0.001	3.61E+02	6.47E+02
Chlorpyrifos	1.06E-01	1.06E-01	4.23E-01	0.01	1.06E+01	4.23E+01	4.26E-01	7.64E-01	0.1	4.26E+00	7.64E+00
Fenitrothion	1.60E-02	1.60E-02	6.40E-02	0.006	2.67E+00	1.07E+01	6.45E-02	1.16E-01	0.04	1.61E+00	2.89E+00

Occurrence of Organophosphorus Pesticides in Maize

The concentrations (mg/kg) of organophosphorus pesticide residue (OPPs) in maize samples obtained from Ngalda agricultural area are presented in Table 1. The OPPs were detected with 100 % detection frequency implicative of their ubiquity in the samples obtained from the four sampling points. This could be attributed to absorption of highly volatile pesticides by the foliar of non-target crops through spray drift as well as take up by crop roots under dry soil conditions (Jara and Winter, 2019). Furthermore, contamination could arise from contaminated surfaces due to storage and distribution practices (Mekonnen *et al.*, 2021). The residual levels in the four (4) samples are as follows: diazinon (2.81E-01 - 8.71E-01 mg/kg), dichlorvos (8.50E-02 - 1.01E+00 mg/kg), chlorpyrifos (2.73E-01 - 5.30E-01 mg/kg) and fenitrothion (3.50E-01 - 9.73E-01 mg/kg). In all OPPs evaluated, the residual level increases from sampling point S1 through S4 with either point S3 or S4 having the highest concentration. The OPPs mean concentrations in the maize samples were fenitrothion (6.83E-01 mg/kg) > diazinon (6.05E-01 mg/kg) > dichlorvos (5.52E-01 mg/kg) > chlorpyrifos (3.97E-01 mg/kg). The level of dichlorvos in this study is in agreement with the work of Yusuf and Bolaji (2017) who observed mean concentration of 0.58 to 0.62 ppm. Wholesomely, the findings are in agreement with the study conducted by Oshatunberu *et al.* (2023) on maize samples from selected markets of southwest Nigeria. According to their results, the concentrations of dichlorvos in both yellow and white maize samples were higher than MRL of 0.01 mg/kg. Diazinon levels in maize collected from Osun had a mean value of 0.046 mg/kg which was very much higher that the stipulated MRLs. In addition to Ibadan, the concentration of chlorpyrifos in yellow maize from Ado-Ekiti was higher than MRLs. On the other hand, its concentrations in white maize across all markets were lower than the MRL (0.05 mg/kg). However, the mean concentrations reported by Ogah and Coker (2012) for maize samples obtained from markets across Lagos State, viz diazinon (0.0212 mg/kg), chlorpyrifos (0.0487 mg/kg) and fenitrothion (0.012 mg/kg) are by far lower than the present study. However, the level of dichlorvos (1.1272 mg/kg) was about doubly higher than value recorded in this study. In a study conducted on fresh maize samples from farms within Enugu state, Nigeria, the concentration of dichlorvos pesticide ranged from 0.0263 to 0.0483 ppm (Onyia *et al.*, 2022). Similarly, Atsen *et al.* (2021) recorded concentration of ND - 0.46 ppm for maize samples stored in warehouses of some local government areas of Plateau state. Furthermore, in the work of Das and Hasan (2022) carried out in Dhaka, Bangladesh, the concentration of dichlorvos (964.38±143.21 µg/kg) was documented. Besides, Salihu *et al.* (2023) obtained concentration of 0.038 mg/kg for chlorpyrifos in maize sold in some markets in Gbako local government, Niger State. These values are all lower than the current study. The detection of OPPs above their maximum residue limits suggests the possibility of misapplication and abuse of the OPPs. The detection could also originate from environmental contamination as a result of pre-harvest agricultural activities such as chemical spraying against weeds and insects (Nyarko *et al.*, 2021).

Occurrence of Organophosphorus Pesticides in Millet

The concentrations of OPPs residues in millet samples obtained from Ngalda are presented in Table 2. The results revealed that the distribution of the OPPs in the four sampling points was uneven with chlorpyrifos and others occurring with 75 % and 100 % detection frequency. The levels ranged: diazinon (2.50E-02 - 4.71E-01 mg/kg), dichlorvos (1.27E-01 - 1.67E-01 mg/kg), chlorpyrifos (ND - 4.80E-01 mg/kg) and fenitrothion (1.39E-01 - 5.80E-01 mg/kg). The statistical mean concentrations (mg/kg) of individual OPPs was in a decreasing order of fenitrothion (3.62E-01) > chlorpyrifos (2.76E-01) > diazinon (1.51E-01) > dichlorvos (1.48E-01). It could be observed that the levels of OPPs residues in millet grains were higher than the recommended MRL (Ezeani *et al.*, 2022).

Occurrence of Organophosphorus Pesticides in Wheat

Table 3 shows the frequency of detection and concentration of the OPPs studied in wheat grains collected from Ngalda area. Overall, 100 % of wheat samples were detected with OPPs. The ranges of concentrations for the OPPs in the wheat samples are as follows: diazinon (1.70E-02 - 6.31E-01 mg/kg), dichlorvos (3.90E-02 - 7.83E-01 mg/kg), chlorpyrifos (1.00E-02 - 7.23E-01 mg/kg) and fenitrothion (2.80E-02 - 6.87E-01 mg/kg). As shown in Table 3, the detection concentrations of OPPs decreased in this order: dichlorvos (4.23E-01 mg/kg) > chlorpyrifos (1.92E-01 mg/kg) > fenitrothion (1.91E-01 mg/kg) > diazinon (1.74E-01 mg/kg). The detected OPPs in all the wheat samples exceeded corresponding maximum residue limit (MRL). However, Akinneye *et al.* (2018) reported a lower dichlorvos level of 0. 15 ±0.001 mg/kg in stored wheat samples in Ondo State. Nardelli *et al*. (2021) recorded the concentration range of 0.011–0.113 mg/kg for OPPs in Italy.

Occurrence of Organophosphorus Pesticides in Rice

The residual concentration of OPPs in rice samples from Ngalda area have been analyzed. The results are presented in Table 4. The concentration of the studied OPPs across the sampling points are as follows: diazinon (3.40E-03 - 2.00E-02 mg/kg), dichlorvos (1.00E-02 - 1.80E-01 mg/kg), chlorpyrifos (2.70E-03 - 4.00E-01 mg/kg) and fenitrothion (3.00E-03 - 2.80E-02 mg/kg). The average concentrations of the these OPPs in the samples were observed in the decreasing order: chlorpyrifos (1.06E-01 mg/kg) > dichlorvos (8.95E-02 mg/kg) > fenitrothion (1.60E-02 mg/kg) > diazinon (1.09E-02 mg/kg). These residual levels are all lower than the recommended maximum residual limit. Contrary to Ghanbari *et al.* (2020) who reported 96.66 % detection of diazinon in the total samples in Iran, with average level of diazinon (0.4±0.43 mg/kg) higher than the MRL. Similarly, the concentration of diazinon in rice samples from a study conducted in Rasht Area, Guilan Province, Iran by Ghanbari *et al.* (2017) indicated that the total average concentration (31.91 mg/kg) is by far higher than the maximum residue limit.

The mean residual level for chlorpyrifos in the current study is 1.06E-01 mg/kg (below MRL). The analysis of Shettima *et al.* (2023) conducted on rice samples grown around Hadejia-Komadugu-Yobe river area in Yobe State shows no detectable level of chlorpyrifos. Similarly, Adepoju-Bello *et al.* (2019) recorded no detectable concentration for chlorpyrifos and diazinon for rice samples collected from some markets in Lagos, Nigeria. However, the results from this study were dramatically lower than the chlorpyrifos residue in rice samples from markets of Makurdi, Benue state as analyzed by Adah *et al.* (2020). Their findings revealed that all the concentrations determined were above MRLs for chlorpyrifos in foodstuffs. Specifically, chlorpyrifos was found in rice samples from Wurukum Market as follows: RAS (6.3960 mg/kg), CCR (2.8537 mg/kg), Faro-61 (3.2745 mg/kg) and Faro-44 (3.1884 mg/kg). The rice samples from Wadata Market showed varying concentrations of chlorpyrifos as follows: RAS (6.3960 mg/kg), Faro-61 (3.2475 mg/kg) and Faro-44 (3.8280 mg/kg) while Faro-61 and Faro-44 samples from Modern Market contained chlorpyrifos at 3.2744 mg/kg and 3.8280 mg/kg respectively. Furthermore, a chlorpyrifos concentration of 0.037±0.051 mg/kg was reported by Divyashree and Tomar (2021) for rice samples obtained from Thiruvananthapuram location, India. Cao *et al.* (2022) recorded highest concentrations for chlorpyrifos (4.22 mg/kg) in rice-growing regions in China during 2016–2020.

Analysis of OPPs residues in maize, millet, wheat and rice have been carried out and the mean concentrations of each cereal are compared as depicted in Fig. 2. Generally, the residual levels of the studied OPPs are highest in maize samples ranging from 3.97E-01 – 6.83E-01 mg/kg. On the contrary, rice samples had the least concentrations range of 1.09E-02 - 1.06E-01 mg/kg.

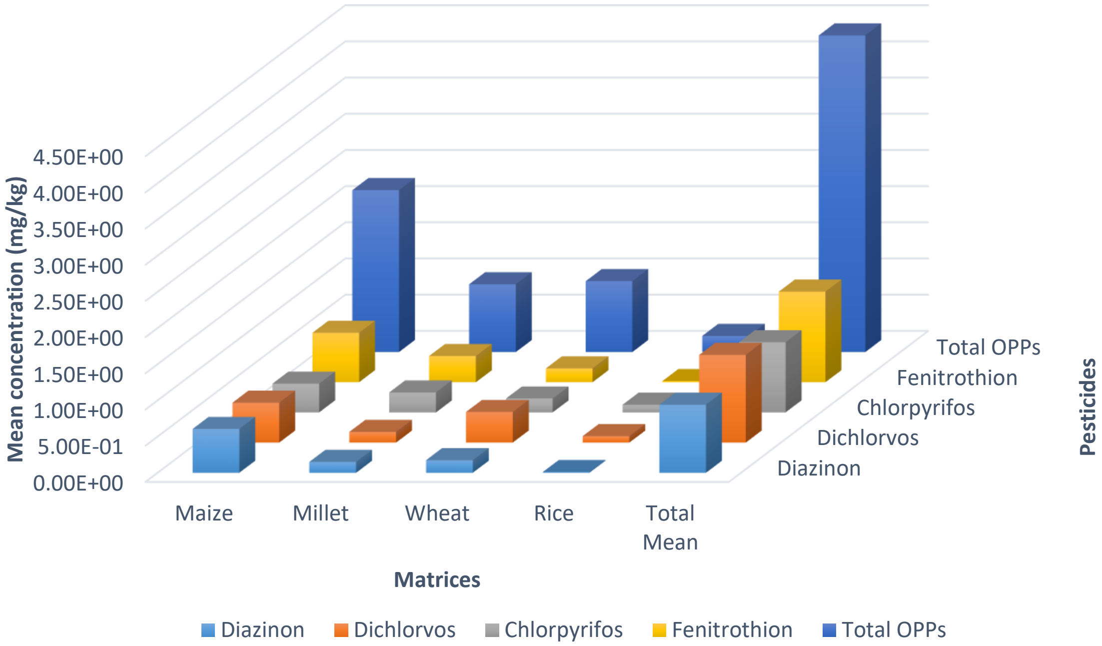

Fig. 2: Comparison of the Pesticides Concentration in the cereals samples from Ngalda Agricultural Area

Potential Health Risk of OPPs through Maize Consumption

Hazards related with pesticides exposure through food consumption have been evaluated. Since maximum residual limits are not toxicological limits, the amounts of OPPs exposure obtained in this study were compared to standard acceptable daily intake (ADI) to estimate the human health risk from consumption of foodstuffs. Health risk index was obtained from the ratio of estimated daily intake (EDI) and their corresponding values of acceptable daily intake (ADI). The ADI, EDI, THQ, ESTI and HI of OPPs for children and adults are presented in Table 5. As shown in Table 5, the estimated dietary intakes (EDI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were estimated to be 6.05E-01, 5.52E-01, 3.97E-01 and

6.83E-01 mg kg/day respectively for adults; and 2.42E+00, 2.21E+00, 1.59E+00 and 2.73E+00 mg kg/day for children. The values of EDI for children were higher than those for adults. The EDI of the studied OPPs exceeded the corresponding ADI for both adults and children indicating that the current cereal consumption posed a health risk to the local inhabitants (Wang *et al.,* 2022).

The target hazard quotients (THQ) of the OPPs for adults and children are presented in Table 5. The THQ of diazinon, dichlorvos, chlorpyrifos and fenitrothion through maize consumption were 1.21E+02, 1.38E+02, 3.97E+01 and 1.14E+02 respectively for adults. For children, the THQ decreased in the order: dichlorvos (5.52E+02) > diazinon (4.84E+02) > fenitrothion (4.56E+02) > chlorpyrifos (1.59E+02). Similarly, the HI values for adults (1.60E+01 - 2.23E+03) and children (2.87E+01 - 3.99E+03) alongside THQ for both age groups are > 1, suggestive of potential health risk. This is in agreement to the findings of Sosan *et al.* (2020) for some organochlorine pesticides in maize for different age groups.

Potential Health Risk of OPPs through Millet Consumption

The ADI, EDI, THQ, ESTI and HI of OPPs for children and adults are presented in Table 6. The dietary intakes (EDI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through millet consumption were estimated to be 1.51E-01, 1.48E-01, 2.76E-01 and 3.62E-01 mg kg/day respectively for adults; and 6.02E-01, 5.91E-01, 1.10E+00 and 1.45E+00 mg kg/day for children. The EDI of the studied OPPs exceeded the corresponding ADI for both adults and children.

The target hazard quotients (THQ) of the OPPs for adults and children are presented in Table 6. The THQ of diazinon, dichlorvos, chlorpyrifos and fenitrothion through millet consumption were 3.01E+01, 3.69E+01, 2.76E+01 and 6.04E+01 respectively for adults. For children, the THQ decreased in the order: fenitrothion (2.42E+02) > dichlorvos (1.48E+02) > diazinon (1.20E+02) > chlorpyrifos (1.10E+02). The HI values were 1.11E+01 - 5.96E+02 and 1.99E+01 - 1.07E+03 for adults and children respectively. The values are > 1 which suggests potential health risk to the consumers (Adeleye *et al.,* 2019).

Potential Health Risk of OPPs through Wheat Consumption

The ADI, EDI, THQ, ESTI and HI of OPPs for children and adults are presented in Table 7. The dietary intakes (EDI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through wheat consumption were estimated to be 1.74E-01, 4.23E-01, 1.92E-01 and 1.91E-01 mg kg/day respectively for adults; and 6.97E-01, 1.69E+00, 7.66E-01 and 7.64E-01 mg kg/day for children. The EDI of the studied OPPs exceeded the corresponding ADI for both adults and children.

The target hazard quotients (THQ) of the OPPs for adults and children are presented in Table 7. The THQ of diazinon, dichlorvos, chlorpyrifos and fenitrothion through wheat consumption were 3.49E+01, 1.06E+02, 1.92E+01 and 3.18E+01 respectively for adults. For children, the THQ decreased in the order: dichlorvos (4.23E+02) > diazinon (1.39E+02) > fenitrothion (1.27E+02) > chlorpyrifos (7.66E+01). HI value ranges of 7.72E+00 - 1.71E+03 and 1.39E+01 - 4.20E+01 were obtained for adults and children respectively. These values > 1 are suggestive of posing potential health hazard to the consumers of wheat from the study area. This study is in line with the work of Sosan *et al*. (2020).

Potential Health Risk of OPPs through Rice Consumption

The ADI, EDI, THQ, ESTI and HI of OPPs for children and adults are presented in Table 8. The dietary intakes (max EDI) of diazinon, dichlorvos, chlorpyrifos and fenitrothion through rice consumption were estimated to be 1.09E-02, 8.95E-02, 1.06E-01 and 1.60E-02 mg kg/day respectively for adults; and 4.34E-02, 3.58E-01, 4.23E-01 and 6.40E-02 mg kg/day for children. The EDI of the studied OPPs exceeded the corresponding ADI for both adults and children.

The target hazard quotients (THQ) of the OPPs for adults and children are presented in Table 8. The THQ of diazinon, dichlorvos, chlorpyrifos and fenitrothion through rice consumption were 2.17E+00, 2.24E+01, 1.06E+01 and 2.67E+00 respectively for adults. For children, the THQ decreased in the order: dichlorvos (8.95E+01) > chlorpyrifos (4.23E+01) > fenitrothion (1.07E+01) > diazinon (8.68E+00). The HI values for adults (1.46E+00 - 3.61E+02) and children (2.62E+00 - 6.47E+02) as well as the THQ are all > 1, suggesting that both the adults and children consuming rice from this study area might be at danger of health risk (Hossain *et al.,* 2015). Furthermore, the high risk index for children through crop consumption might be attributed to their low body weight (Oyinloye *et al.,* 2021).

CONCLUSION

The OPPs residues in maize, millet, wheat and rice samples from Ngalda agricultural area have been analyzed. The study reveals varying degree pesticide residues in the four cereals samples. The concentrations were in most cases found to be above the maximum residual limits. The Hazard index for all the cereals were above 1, indicating that consumption of the cereals from the study area might pose serious health risk. There is a need for the relevant agencies to strictly control the use and disposal of these hazardous pesticides.

REFERENCES

Adah, C. A., Asemave, K. and Ado, O. (2020). Determination of some herbicides in foreign and local rice brands obtained from Makurdi, Benue, Nigeria. *International Journal of Chemistry Studies,* 4(1): 01-05.

Adeleye, O. A., Soasn, M. B. and Oyekunle, J. A. O. (2019). Dietary Exposure Assessment of Organochlorine pesticides in Two Commonly Grown Leafy Vegetables in South-western Nigeria. *Heliyon*, 5:e01895.

Adepoju-Bello, A. A., Oyawaluja, B. O. and Sosanya, D. G. (2019). Analysis of Some Selected Toxic Metals and Pesticide Residues in Ofada Rice (*Oryza Sativa L.*) Samples Produced in South Western Region of Nigeria. *Journal of Industrial Research and Technology,* 8(1):27-44.

Akinneye, J. O., Adedire, C. O. and Arannilewa, S. T. (2006) Potential of Cleisthopholis patens Elliot asa maize protectant against the stored product moth, Plodia Interpunctella (Hubner)(Lepidoptera; Pyralidae). *African Journal of Biotechnology*, 5(1):2510-2515.

Akinneye, J. O., Adedolapo, A. O. and Adesina, F. P. (2018). Quantification of organophosphate and carbamate residue on stored grains in Ondo State, Nigeria. *Journal of Biology and Medicine,* 2(1):001-006. DOI: 10.17352/ aur.000003

Akoto, O., Andoh, H., Darko, G., Eshun, K and Osei-fosu, P. (2013) Health risk assessment of pesticides residue in maize and cowpea from Ejura. *Ghana Journal of Chemosphere*, 92(1):67-73.

Al-Wabel, M. I., El-Saeid, M. H., Al-Turki, A. M. and Abdel Nasser, G. (2011). Monitoring of pesticide residues in Saudi Arabia agricultural soils. *Research Journal of Environmental Science,* 5(1):269-278.

Atsen, S. N., Mafuyai, G. M. and Eneji, I. S. (2021). Pesticide Residue in Maize Stored in Bassa, Bokkos, Jos – North and Mangu Local Government Area Warehouses, Plateau State. *African Journal of Agriculture and Food Science*, 4(1):79-88.

Cao, Z., Zheng, X., Guan, M., Zhang, W., Lin, X., Zhao, X. and Chen, M. (2022). Cumulative Risk Assessment of Dietary Exposure to Pesticide Residues in Brown Rice (*Oryza sativa* L.) from the Three Main Rice-Growing Regions in China during 2016–2020. *Journal of Food Quality*, Article ID 5902540. https://doi.org/10.1155/2022/5902540

Damalas, C. A. and Eleftherohorinos, I. G. (2011). Pesticide exposure, safety issues and risk assessment indicators. *International Journal of Environmental Research and Public Health*, 8(1):1402-1419.

Darko, G. and Akoto, O. (2008). Dietary intake of Organophosphorus pesticide residues through vegetables from Kumasi, Ghana. *Journal of Food Chemical Toxicology*, 46(12):3703-3706.

Das, A. K. and Hasan, G. M. M. A. (2022). Analysis of Pesticide Residual Levels in Maize (Zea mays L.) Grain, Flour and Processed Items from Selected Areas of Dhaka, Bangladesh. *Oriental Journal of Chemistry,* 8(3):681-687.

Divyashree, J. and Tomar, S. (2021). Analysis of Pesticide Residues in Water, Rice, Fruits and Vegetables. *Journal of Pharmaceutical Research International*, 33(60B):1462-1473. DOI: 10.9734/JPRI/2021/v33i60B34768

Ezeani, U. U., Afonne, O. J., Ezeani, M. C. and Ehigiator, B. E. (2022). Determination of pesticide residue levels in some common food crops: The suitability for human consumption. *Journal of Toxicology and Environmental Health Sciences*, 14(1):10-19. DOI: 10.5897/JTEHS2021.0493

FAO/WHO (2020). Joint FAO/WHO Food standards programme codex alimentarius commission pesticide residues in food and feed and maximum residue limits.

Fatunsin, O. T., Oyeyiola, A. O., Moshood, M. O., Akanbi, L. M. and Fadahunsi, D. E. (2020). Dietary Risk Assessment of Organophosphate and Carbamate Pesticide Residue in Commonly Eaten Food Crops. *Scientific African*, 8:e00442. https://doi.org/10.1016/j.sciaf.2020e00442

GEMS/FOOD. (2012). Global Environment Monitoring System-Food Contamination Monitoring and Assessment Programme. GME/Food Cluster Diets. World Health Organization, New York, USA

Ghanbari, F., Moattar, F., Monavari, S. M. and Arjmandi, R. (2017). Human health risk assessment of organophosphorus pesticide in rice crop from selected districts of Anzali International Wetland basin, Iran. *Human and Experimental Toxicology,* 36(5):438–444. DOI: 10.1177/0960327116657603

Ghanbari, F., Monavari, S. M., Sadr, M. K., Rahimi, R. and Mirbolooki, H. (2020). Pesticide in Soil and Rice Crop from North of Iran: Concentration and Risk Assessment. *Advanced Journal of Chemistry-Section A,* 3(2), 211-220. DOI: 10.33945/SAMI/AJCA.2020.2.9

Hossain, M. S., Fakhruddin, A. N., Chowdhury, A. Z., Rahman, M. A. and Alam, M. K. (2015). Health risk assessment of selected pesticide residues in locally produced vegetables of Bangladesh. *International Food Research Journal*, 22 (1): 110–115.

Iya, I. B. and Kwaghe, T. T. (2007). The economic effect of spray pesticides on cowpeaproduction in Adamawa state of Nigeria. *International Journal of Agricultural Research*, 2(1):637-650.

Jara, E. A. and Winter, C. K. (2019). Safety levels for organophosphate pesticide residues on fruits, vegetables, and nuts. *Journal of Food Contamination*, 6(1):1–8.

Kolani. L., Mawussi G., Devault D. A. and Sanda K. (2017). Organochlorine Pesticide Residues in Agricultural Soils from Region Des Plateaux in Togo. *International Journal of Agricultural Research*, 33(1):33-42.

Leong, K. H., Tan, L. B. and Mustafa, A. M. (2007). Contamination levels of selected organochlorine and organophosphate pesticides in the Selangor River, Malaysia between 2002 and 2003. *Journal of Chemosphere*, 66(1):53-59.

Li, R., He, L., Wei, W., Hao, L., Ji, X., Zhou, Y. and Wang, Q. (2015). Chlorpyrifos residue levels on field crops (rice, maize and soybean) in China and their dietary risks to consumers. *Food Control*, **5**:212–7.

Mekonnen, B., Siraj, J. and Negash, S. (2021). Determination of Pesticide Residues in Food Premises Using QuECHERS Method in Bench-Sheko Zone, Southwest Ethiopia. *BioMed Research International*, Article ID 6612096, 13 pages https://doi.org/10.1155/2021/6612096

Naksen, W., Hongsibsong, S., Xu, Z., Kosashunhanan, N., Kerdnoi, T., Prapamontol, T. and Patarasiriwong, V. (2023). Health Risk Assessment from Organophosphate Insecticides Residues in Commonly Consumed Vegetables of Local Markets, Northern Thailand. *Journal of Health Research*, 37(3):153e162. https://doi.org/10.56808/2586-940X.1009

Nardelli, V., D'Amico, V., Ingegno, M., Della Rovere, I., Iammarino, M., Casamassima, F., Calitri, A., Nardiello, D. and Li, D. (2021). Quinto, M. Pesticides Contamination of Cereals and Legumes: Monitoring of Samples Marketed in Italy as a Contribution to Risk Assessment. *Applied Science*, 11: 7283. https://doi.org/10.3390/app11167283

National Food Safety Standard Maximum Residue Limits for Pesticides in Foods (2019). (GB 2763-2019)

NOUN (National Open University of Nigeria) Stored Produce Protection, Published by National Open University of Nigeria (2016).

Nyarko, S. K., Akyereko, Y. G., Akowuah, J. O., Wireko-Manu, F. D. (2021). Comparative Studies on Grain Quality and Pesticide Residues in Maize Stored in Hermetic and Polypropylene Storage Bags. *Agriculture*, 11, 772. https://doi.org/10.3390/agriculture11080772

Ogah C. O., Coker, H. B. (2012). Quantification of Organophosphate and Carbamate Pesticide Residues in Maize. *Journal of Applied Pharmaceutical Science*, 2(1): 093- 097.

Onyia, V. C., Chime, C. C., Udeozo, P. I., Lotanna, O. C. and Ude, U. J. (2022). Determination of pesticide residue in maize treated with mixed pesticides against stem borer disease in selected farms within Enugu state, Nigeria. *International Journal of Phytology Research*, 2(4):01-08.

Oshatunberu, M. A., Oladimeji, A., Henry, S. O., Olaniyan, O. A. and Raimi, M. O. (2023). Concentrations of Pesticides Residues in Grain Sold at Selected Markets of Southwest Nigeria. *Natural Resources for Human Health*, 3(4): 387-402. https://doi.org/10.53365/nrfhh/171368

Oyinloye, J. A., Oyekunle, J. A. O., Ogunfowokan, A. O., Msagati, T., Adekunle, A. S. and Netty, S. S. (2021). Human Health Risk Assessment of Organochlorine Pesticides in Some Food Crops from Esa-Oke farm Settlement, Osun State, Nigeria. *Heliyon*, 7:e07470. https://doi.org/10.1016/j.heliyon.2021.e07470

Saheed, O. A., Olawale, S. H., Tolulope, S. J. (2020). Assessment of Pesticide Residues in Grains Sold in Major Commercial Markets in Ilorin, Kwara State, Nigeria. *International Journal of Research and Innovation in Applied Science*, V(IX): 227 – 231.

Salihu, A. M., Ndamitso, M. M., Mathew, J. T., Etsuyankpa, M. B., Gwadabe, N. K. and Ajai, A. I. (2023). Determination of Pesticide Residues in Selected Cereals Crops Sold in SomeMarkets in Gbako Local Government, Niger State. *Lapai Journal of Applied and Natural Sciences*, 8(1): 25-30.

Shettima, S. A., Gashinge, A. M., Baffa, A. A., Akinlabi, A. K. and Abdulkadir, A. S. (2023). Investigation into the Pesticides Residues in Sesame, Millet, Rice, and Guinea Corn Grown Around the Hadejia-Komadugu-Yobe River Area in Yobe State, Nigeria. *Journal of Chemical Society of Nigeria*, 48(3):449 – 397.

Sosan, M. B., Adeleye, A. O., Oyekunle, J. A. O., Udah, O., Oloruntunbi, P. O., Daramola, M. O. and Saka, W. T. (2020). Dietary Exposure Assessment of Organochlorine pesticides in in Maize-based Complimentary Breakfast Food Products in Nigeria. *Heliyon*, 6:e05803.

USEPA. (2007). Integrated risk information system-database. Philadelphia PA; Washington, DC

Usher, K. and Majors, R. E. (2012). Analysis of Pesticide Residues using GCMS. Agilent, Inc. Wilmington. DE 19808 ISA

Wang, N., Cui, Z., Wang, Y. and Zhang, J. (2022). Characteristics and Residual Health Risk of Organochlorine Pesticides in Fresh Vegetables in the Suburb of Changchun, Northeast China. *International Journal of Environmental Research in Public Health*, 19, 12547. https://doi.org/10.3390/ijerph191912547

Yusuf, B. I. and Bolaji, O. M. (2017). Concentration of Pesticide Residue in Beans and Maize Grains in Bodija Market, Ibadan, Nigeria. *African Journal of Environmental Health Sciences*, 4:69-77.