

Human Health Risk Assessment of Pesticide Residues in Some Cereals Sold in Nigerian Markets

Oladapo, F. O*, Adeboye, A & Ogundele, R.

Department of Chemical Sciences, Tai Solarin University of Education, Ijagun, Ogun State, Nigeria

*Corresponding Author: oladapofo@tasued.edu.ng

ABSTRACT: The occurrence of pesticide residues in agricultural produce and processed foodstuffs can be detrimental to global efforts aimed at ensuring food security. This study evaluated the presence of some pesticide residues in rice and oats sold in Nigerian markets. Samples collected were homogenized, extracted with a hexane-acetone mixture and analyzed using the gas chromatography coupled with mass spectrometry (GC-MS) technique. Results of the analysis showed that the concentrations of lindane isomers in rice and oats ranged from 0.00 mg.kg⁻¹ to 0.13 mg.kg⁻¹. Heptachlor concentrations in rice and oats ranged between 0.02 mg.kg⁻¹ and 0.18 mg.kg⁻¹ whereas the concentration ranges of endosulphan and endrin aldehyde were 0.03-0.40 mg.kg⁻¹ and 0.00-0.16 mg.kg⁻¹ respectively. The levels of dichlorodiphenyltrichloroethane (DDT) and derivatives in rice ranged between 0.13 and 0.23 mg.kg⁻¹, whilst methoxychlor had 0.08 mg.kg⁻¹ in rice. Some pesticide residues (6 in oats and 5 in rice) were detected at levels higher than their maximum residue levels (MRLs) prescribed by the Food and Agricultural Organization/World Health Organization (FAO/WHO) and the European Union (EU). The health quotient (HQ) levels of heptachlor in rice for both adults (HQ = 1.40) and children (HQ = 6.82) exceeded the threshold of health risk safety level (HQ <1) and hence may constitute non-carcinogenic health problems. The health index (HI) of pesticide residues in oat for children (HI = 2.957), as well as rice samples for both adults (HI = 1.576) and children (HI = 7.695) exceeded the threshold health safety mark (HI <1) and hence would be described as not safe for consumption over a lifetime.

KEYWORDS: Pesticide residues, Cereals, Maximum residue levels, Health index, Non-carcinogenic risk assessment.

1. INTRODUCTION

Pesticides are common chemical agents widely used to control or kill pests on agricultural farmlands, grain storage warehouses, animal husbandry, and poultry farms (NIEHS, 2013). Pesticides, as used throughout this paper, refer to all categories of pesticides, namely herbicides, insecticides, rodenticides, avicides, molluscides, nematocides, and fungicides (NAFDAC, 1996). According to Njoku and coworkers (2017), pesticide residues can be defined as the components of pesticides that remain or are contained in food substances after pesticide application. The World Health Organization (2016) defined pesticide residues as substances or a mixture of substances found in food prepared for human or animal consumption resulting from the use and biotransformation of pesticides in the environment. Pesticide applications have helped increase the agricultural turnout of commercial farmers, promote food security globally, lower food costs, minimize the spread of diseases, and sustain the viability of agricultural businesses (Omoyajowo *et al.*, 2018).

In modern farm practices, pesticides are applied at different stages of cropping, starting from pre-planting, planting, emergence, leafing, and fruiting to storage (Naylor, 2003). Pesticides belong to different chemical classifications, and the prominent ones used by farmers include organochlorines, organophosphates, carbamates, and pyrethroids (Ogah & Coker, 2012). Pesticides can be applied directly to food plants such as vegetables, cereals, and fruits, as well as non-food plants such as cotton, flowers, and fields (Njoku *et al.*, 2017). Pesticides' interaction with the plant's surfaces

occurs immediately after their application under the influence of the wind and the sun, while some proportions are washed off by rain into the soil (Keikotlhaile & Spanoghe, 2011). They enter the plant tissue system after absorption or get sequestered to form pesticide residues (Keikotlhaile & Spanoghe, 2011). The proportion washed into the soil is drained or leached into the lower part of the soil profile (Keikotlhaile & Spanoghe, 2011). According to studies, about 80% of pesticides used in agriculture have a direct negative impact on the environment (Ali *et al.*, 2021).

The fate of pesticides in the environment may be completely unknown; however, the biological transport mechanism of pesticides in the environment is more likely to be determined by their physicochemical properties (Lewis *et al.*, 2021). Factors, such as the chemical structures of pesticides, especially the number of benzene-ring linkages, the position and type of halogen appendages on a benzene ring, the physicochemical properties of their post-biodegradation chemical adduct, their solubility, mobility, volatility, and persistence, are predictive of their behaviour and toxic activities in the environment (Lewis *et al.*, 2021). Pesticides' persistence in the environment and bioaccumulation in the food chain are responsible for their toxicity, making them detrimental to human and animal health (Leong *et al.*, 2007; El-Shahawi *et al.*, 2010). Other human pesticide exposure sources include contaminated drinking water and sediment, consumption of contaminated fish, and nasal inhalation of pesticide dust (Qu *et al.*, 2015)

The many advantages of pesticides, especially their potential for improved agricultural output, capacities for large-scale production, maximization of profits, and the possibility of raising disease-free farm produce, would have encouraged the widespread application of pesticides to diverse agricultural farmlands. Today, cereal farmers have seized these benefits to increase their yearly turnouts and reduce the risks of losing their grains before, during, and after storage (Sulaiman & Rosentrater, 2022). The application of pesticides on crops and animal farms can leave potentially toxic pesticide residues, which may induce health risks through dietary and non-dietary exposure pathways (Botwe *et al.*, 2011). Ogbeide and co-workers (2016) reported that there were potential exposure risks to humans and fish after assessing the health risks of heptachlor epoxide, aldrin, dieldrin, DDT, endosulphan I, endosulphan II, and endosulphan aldehyde residues on rice farms in Edo State, Nigeria. Studies revealed that plants' pollinating insects are at risk of extinction due to pesticide exposure (Lundgren & Fausti, 2015, Alkassab & Kirchner, 2017).

Clinical studies have associated pesticide exposure with health conditions, such as cancer, and dysfunction of the neurological, reproductive, immunological, and nervous systems (Sanborn *et al.*, 2007; WHO, 2016). Pesticide toxicity has been linked to declining sperm quality and quantity (Chiu *et al.*, 2015), cases of infertility, such as azoospermia, oligospermia, and increased follicle-stimulating hormone (FSH) and luteinizing hormone (LH) (Perry *et al.*, 2011). Acute pesticide poisoning can also result in seizures, rashes, and gastrointestinal conditions (Guidotti & Gosselin, 1999). Due to their acetylcholinesterase inhibition potential, carbamates' exposure can cause reversible neurological illness (Bogialli *et al.*, 2004). These pesticides, and many others have been reported as putative carcinogenic, mutagenic and teratogenic agents (Bogialli *et al.*, 2004; Ragnarsdottir, 2000; Rocha & Grisolia, 2019; Sternberg, 1979).

Administration of some pesticides may have been reduced or outrightly banned, safer alternatives recommended for use are excessively consumed (Taylor *et al.*, 2002; Sharma *et al.*, 2017). Although there are existing pesticide data in

several published papers, the continued use of pesticides across the globe will require periodic review. This study was, therefore, conducted to assess the levels of some pesticide residues in selected cereals along the food distribution chain and to assess the health risks associated with long-term dietary exposure to such food items.

2. MATERIALS AND METHODS

2.1 Materials: Chemicals/Reagents

All chemicals used for this study were of analytical grade. Hydrogen peroxide, n-hexane, nitric acid, and acetone were obtained from Rathburn (Scotland, United Kingdom). The OCP standard (2000 ppm), containing twenty-one different components of organochlorine pesticides, was purchased from AccuStandard Incorporated, USA. GC-MS was purchased from Agilent Technologies Incorporated, USA.

2.2 Methods

2.2.1 Sample Collection and Treatment

Samples of local and foreign cereals (3 brands of rice, and 5 brands of oat) used for this analysis were collected across markets and malls in Lagos and Ibadan in South-West Nigeria. The Double T Ofada rice and Bigbull rice used for this study were produced locally, while Maharani rice was made in India. Similarly, all the oat samples were produced locally, except the Quaker Cooking Oat, which was produced in Canada. The samples were homogenized using a pestle and mortar. 5 g of a homogenized sample was dissolved in a 250 mL conical flask with a 20 mL hexane-acetone (1:1; v/v) mixture, and the resulting solution was ultrasonicated at 27°C for 20 min. The solution was removed, allowed to cool down, and filtered afterwards using filter paper and a funnel. The filtrate was concentrated to 2 mL, ready for analysis using Gas Chromatography coupled with Mass Spectrometry (GC-MS). The other cereal samples were given similar treatment.

2.2.2. Sample Analysis Using Gas Chromatography-Mass Spectrometry (GC-MS)

Five (5) point serial dilution calibration standards, namely, 0.10, 1.00, 5.00, 10.00, and 100.00 ppm, were prepared from the organochlorine pesticide stock and were used to calibrate the GC-MS. An Agilent 7820A gas chromatograph coupled to a triple-axis detector-carrying 5975C inert mass spectrometer with an electron-impact source was used. Attached to the GC was the stationary phase, comprising the HP-5 capillary column coated with 5% phenylmethyl siloxane (30 m length x 0.32 mm diameter x 0.25 µm film thickness), where sample separation was carried out. 1 µL of the samples was injected into the GC in splitless mode, aided by helium as the carrier gas. The Helium carrier gas was operated at a constant flow rate of 1.2 mL/min at an initial pressure of 0.26 psi and an average velocity of 40.00 cm/sec. The oven programme was initiated and kept at 50 °C for 1 min, then ramped at 25 °C/min to 100 °C temperature (3 min), and then 5 °C/min to 300 °C (5 min). The run-time was 51 min with a 3 min solvent delay. The mass spectrometer was operated with an electron-impact ionization mode at 70 eV with an ion-source temperature of 230 °C, quadrupole temperature of 150 °C and a transfer line temperature of 300 °C.

2.2.3 Health Risk Assessment of Pesticide Residues

Human health risk assessment correlates environmental pollutants' concentrations with their likelihood of toxic effects on the exposed human population (Jiang *et al.*, 2017). The health risks of taking pesticide residues in cereals were evaluated based on estimated daily intake (EDI), health risk index (HRI) and cancer risk (CR) using the Integrated Risk Information System model of the United States Environmental Protection Agency (USEPA, 2001, 2002 & 2007).

Estimated Daily Intake (EDI)

The EDI can be calculated using Equation 1

$$EDI = \frac{C \times FCR}{BW} \quad (1)$$

Where EDI represents the estimated daily pesticide intake in cereals ($\text{mg.kg}^{-1}.\text{day}^{-1}$), C stands for the concentration of pesticide residues in cereals (mg.kg^{-1}), FCR is the food consumption rate set at 0.41 kg.day^{-1} and 0.19 kg.day^{-1} for cereal in adults and children, respectively, in (Fatunsin *et al.*, 2020), and BW represents the average body weight (given as 60 kg for adult and 16.7 kg for children) set by USEPA and WHO in (Fatunsin *et al.*, 2020).

The health quotient (HQ) of pesticide residues in cereal can be calculated using Equation 2, where EDI is the estimated daily intake in cereals, and ADI stands for the acceptable daily intake. ADI refers to the level of a potentially toxic substance present in food or drinking water that can be ingested over a lifetime without suffering any health consequences. This value has been equated to the tolerable daily intake (TDI) and is also referred to as the reference dose (RfD) by the United States Environmental Protection Agency (USEPA) (Becking *et al.*, 2007)

$$HQ = \frac{EDI}{ADI} \quad (2)$$

The HI is the sum of all the HQs of the pesticides and is expressed in Equation 3

$$HI = \sum_1^n HQ \quad (3)$$

The sum of the HQs of each pesticide residue adds up to make the HI of the cereals of interest. The HQ value <1 suggests no health risk, whilst the HQ value >1 suggests a potentially adverse health risk, especially with long-term exposure (Singh & Kumar, 2017)

3. RESULTS AND DISCUSSION

This study examined the levels of pesticide residues in oat and rice samples. Twelve pesticide residues were investigated in the rice and oat samples analyzed, and these pesticide residues are alpha-lindane, beta-lindane, gamma-lindane, delta-lindane, heptachlor, endosulphan I, endosulphan II, endrin aldehyde, p,p-DDD, p,p-DDE, p,p-DDT and methoxychlor (Table 1).

Table 1: The levels of pesticide residues in different oat samples compared with maximum residue levels (MRLs)

Pesticide	Concentration of Oat Samples (mg.kg ⁻¹), N =5										
	OFO	CUO	LWO	MTWO	QCO	Mean	Samples with residues	Samples with residues > MRL	Samples with residues < MRL	MRL (mg.kg ⁻¹) ^a FAO/WHO ^b EU	
Alpha-lindane	0.00	0.00	0.00	0.00	0.00	0.00	Nil	NA	5	0.01	0.01
Beta-lindane	0.00	0.00	0.13±0.00	0.00	0.00	0.03	1	1	4	0.01	0.01
Gamma-lindane	0.00	0.00	0.00	0.00	0.00	0.00	Nil	NA	5	0.01	0.01
Delta-lindane	0.07±0.00	0.00	0.05±0.00	0.00	0.00	0.02	2	2	3	0.01	0.01
Heptachlor	0.00	0.00	0.00	0.00	0.00	0.00	Nil	NA	5	0.02	0.01
Endosulphan I	0.00	0.34±0.00	0.00	0.40±0.00	0.00	0.15	2	Nil	3	1.0	0.05
Endosulphan II	0.38±0.00	0.05±0.00	0.05±0.00	0.21±0.00	0.05±0.00	0.15	5	Nil	5	1.0	0.05
Endrin aldehyde	0.16±0.00	0.00	0.00	0.01±0.00	0.05±0.00	0.04	3	3	2	0.05	0.01
p,p'-DDE	0.00	0.00	0.00	0.00	0.00	0.00	Nil	NA	5	0.1	0.01
p,p'-DDD	0.00	0.00	0.00	0.00	0.00	0.00	Nil	NA	5	0.1	0.01
p,p'-DDT	0.00	0.00	0.00	0.00	0.00	0.00	Nil	NA	5	0.1	0.01
Methoxychlor	0.00	0.00	0.00	0.00	0.00	0.00	Nil	NA	5	0.01	0.01

OFO: Old Fashioned Oat, CUO: Cheer-Up Oat, LWO: Lecker White Oat, MTWO: Morning Time White Oat, and QCO: Quaker Cooking Oat, MRL: Maximum Residue Limit, ^aFAO/WHO, 2018, ^bEU, 2017.

The result analysis revealed the presence of pesticide residues in all the oat and rice samples studied. Although alpha-lindane, gamma-lindane, heptachlor, p,p-DDT, p,p-DDD, p,p-DDE and methoxychlor were below the detection limit in all the oat samples, two pesticide residues were present in all five oat samples. The other seven pesticide residues occurred below the detection limit. Cheer-Up Oat (CUO) and Quaker Cooking Oat (QCO) both had two pesticide residues, whereas Lecker White Oat (LWO), Morning Time White Oat (MTWO) and Old-Fashioned Oat (OFO) had three pesticide residues each. The results from the rice samples indicated the presence of all the pesticide residues under investigation in Bigbull Rice (BBR), a locally produced rice, except four pesticides, namely delta-lindane, heptachlor, endosulphan I, and endrin aldehyde (Table 2).

Table 2: The levels of pesticide residues in different rice samples compared with maximum residue levels (MRLs)

Pesticide	Concentration of Rice Samples (mg.kg ⁻¹), N=3								
	DTOR	BBR	MR	Mean	Samples with residues	Samples with residues >MRL	Samples with residues <MRL	MRL (mg.kg ⁻¹) ^a FAO/WHO ^b EU	
Alpha-lindane	0.00	0.02±0.00	0.00	0.01	1	Nil	2	0.01	0.01
Beta-lindane	0.00	0.07±0.00	0.00	0.02	1	1	2	0.01	0.01
Gamma-lindane	0.00	0.03±0.00	0.00	0.01	1	1	2	0.01	0.01
Delta-lindane	0.07±0.00	0.00	0.07±0.00	0.05	2	2	1	0.01	0.01
Heptachlor	0.02±0.00	0.00	0.18±0.00	0.06	2	1	2	0.02	0.01
Endosulphan I	0.03±0.00	0.00	0.08±0.00	0.04	2	Nil	3	1.0	0.05
Endosulphan II	0.14±0.00	0.28±0.00	0.16±0.00	0.19	2	Nil	3	1.0	0.05
Endrin aldehyde	0.00	0.00	0.00	0.00	Nil	NA	3	0.05	0.01
p,p'-DDE	0.00	0.13±0.00	0.00	0.04	1	Nil	3	0.1	0.01
p,p'-DDD	0.00	0.23±0.00	0.00	0.08	1	Nil	3	0.1	0.01
p,p'-DDT	0.00	0.13±0.00	0.00	0.04	1	Nil	3	0.1	0.01
Methoxychlor	0.00	0.08±0.00	0.00	0.03	1	Nil	3	0.01	0.01

DTOR: Double T Ofada Rice, BBR: Big Bull Rice, MR: Maharani, MRL: Maximum Residue Limit, ^aFAO/WHO, 2018. ^bEU, 2017.

Results obtained from rice analysis show the presence of pesticide residues in the three samples analyzed. For Double T Ofada (DTOR) and Maharani rice (MR), four pesticide residues only were detected, namely delta-lindane, heptachlor, and endosulphan I and II. The concentrations of delta-lindane in DTOR and MR exceeded the maximum residue limit (MRL) values set by FAO/WHO (2018) and the EU (2017). The heptachlor level in DTOR equals the MRL in the FAO/WHO database but exceeds that of the EU (2017). The endosulphan I and II contents in DTOR and MR occurred below the MRLs set by the FAO/WHO (2018). The concentrations of various isomers of lindane in BBR exceeded the MRLs of both the FAO/WHO (2018) and the EU (2017). DDT and its metabolites occur at concentrations slightly higher than the FAO/WHO's and the EU's MRLs. The methoxychlor concentration reported in rice was higher than the MRLs recommended by FAO/WHO and EU (Table 2). In a study conducted by Omeje and coworkers (2021), the concentrations of pesticides in rice were reported as follows: lindane and its isomers, 0.0833 mg.kg⁻¹; heptachlor, 0.2175 mg.kg⁻¹; endosulphan I and II, 0.0796 mg.kg⁻¹; DDT and its congeners, 0.1168 mg.kg⁻¹.

Health Risk Assessment of Pesticide Residues in Cereals

The health quotients (HQ) of all the pesticide residues were calculated for both children and adults to determine the non-carcinogenic risks which individual pesticide residue, and, by extension, cumulative pesticide residues may constitute to oats and rice consumers (Tables 3 and 4). The HQs of alpha-lindane, gamma-lindane, heptachlor, p,p-DDD, p,p-DDE, p,p-DDT and methoxychlor residues in oats calculated for adults are zero, indicating no health risk

to consumers. The HQs of other pesticide residues in oat samples for adults occur in ascending order of beta-lindane, endosulphan II < delta-lindane < endosulphan I < endrin aldehyde. However, the cumulative HQ of all the pesticide residues (HI=10.02) in oat samples for adults is far greater than 1, suggesting chronic health risks to consumers. Similarly, all the HQs of the pesticide residues evaluated in oats for children individually suggest no health risk, except for that of endrin aldehyde, with an HQ of 3.98. Going by the health risk reference scale, $HQ > 1$ indicates serious health effects for consumers.

Table 3: Health Quotient and Non-carcinogenic Risk Assessment of Pesticide Residues in Oat Samples

Pesticide	Adult				Children		
	Conc;n (mg.kg ⁻¹)	EDI (x 10 ⁻⁵) (mg.kg ⁻¹ .d ⁻¹)	ADI (mg.kg ⁻¹ .d ⁻¹)	HQ	EDI (x 10 ⁻⁴) (mg.kg ⁻¹ .d ⁻¹)	ADI (mg.kg ⁻¹ .d ⁻¹)	HQ
Alpha-lindane	0	0	0.005	0	0	0.005	0
Beta-lindane	0.03	7.00	0.005	0.014	3.413	0.005	0.068
Gamma-lindane	0	0	0.005	0	0	0.005	0
Delta-lindane	0.02	4.67	0.005	0.009	2.275	0.005	0.046
Heptachlor	0	0	0.0001	0	0	0.0001	0
Endosulphan I	0.15	35.00	0.006	0.058	17.066	0.006	0.284
Endosulphan II	0.15	35.00	0.006	0.058	17.066	0.006	0.284
Endrin aldehyde	0.04	9.33	0.0002	0.467	4.551	0.0002	2.275
p,p'-DDE	0	0	0.01	0	0	0.01	0
p,p'-DDD	0	0	0.01	0	0	0.01	0
p,p'-DDT	0	0	0.01	0	0	0.01	0
Methoxychlor	0	0	0.01	0	0	0.01	0
			HI = ∑HQs =	0.606		HI = ∑HQs =	2.957

EDI: Estimated Daily Intake, ADI: Average Daily Intake, HQ: Health Quotient, HI: Health Risk Index

The HQ values recorded for pesticide residues in rice samples for adults are below the unity mark, except for heptachlor, which recorded an HQ value of 2.33 (Table 4). This implies that heptachlor may induce non-carcinogenic health concerns in rice consumers if pesticide application continues unabated. For rice, a cumulative HQs of 2.649 for pesticide residues in adults indicates a slight likelihood that such food consumption may lead to a potential health effect. For Children, the HQ values for the pesticide residues in rice samples are slightly higher than those reported for adults. Heptachlor recorded an HQ value of 11.38, but HQ values for others are below 1. In summary, the HI for pesticide residues in rice for children suggests a high probability that such a cumulative pesticide level can modulate non-carcinogenic health risks in potential consumers.

Table 4: Health Quotient and Non-carcinogenic Risk Assessment of Pesticide Residues in Rice Samples

Pesticide	Adult				Children		
	Conc'n (mg.kg ⁻¹)	EDI (x 10 ⁻⁵) (mg.kg ⁻¹ .d ⁻¹)	ADI (mg.kg ⁻¹ .d ⁻¹)	HQ	EDI (x 10 ⁻⁴) (mg.kg ⁻¹ .d ⁻¹)	ADI (mg.kg ⁻¹ .d ⁻¹)	HQ
Alpha-lindane	0.01	2.33	0.005	0.005	1.14	0.005	0.023
Beta-lindane	0.02	4.67	0.005	0.009	2.28	0.005	0.046
Gamma-lindane	0.01	2.33	0.005	0.005	1.14	0.005	0.023
Delta-lindane	0.05	11.66	0.005	0.023	5.69	0.005	0.114
Heptachlor	0.06	14.00	0.0001	1.400	6.83	0.0001	6.836
Endosulphan I	0.04	9.33	0.006	0.016	4.55	0.006	0.076
Endosulphan II	0.19	44.33	0.006	0.074	21.62	0.006	0.360
Endrin aldehyde	0	0	0.0002	0	0	0.0002	0
p,p'-DDE	0.04	9.33	0.01	0.009	4.55	0.01	0.046
p,p'-DDD	0.08	18.67	0.01	0.019	9.10	0.01	0.091
p,p'-DDT	0.04	9.33	0.01	0.009	4.55	0.01	0.046
Methoxychlor	0.03	7.00	0.01	0.007	3.41	0.01	0.034
			HI=∑HQs	1.576		HI=∑HQs	7.695

EDI: Estimated Daily Intake, ADI: Average Daily Intake, HQ: Health Quotient, HI: Health Risk Index

A statistical study of the five Oat brands analyzed showed that the mean concentrations of each pesticide residue reduced drastically compared to that of an individual brand in which pesticide residue was reported. These mean levels are expected to reduce further when large samples are used. These results can be extrapolated to the three rice brands analyzed. Generally, it is necessary to state that pesticide residues found in each cereal brand occurred at microgram levels per kilogram except in a few others which were slightly below the milligram per kilogram levels. Nevertheless, the maximum residue limits (also known as maximum permissible level, MPL) published by regulatory organizations are at the sub-micro level, indicating how detrimental pesticide residue bioaccumulation would be in humans, and the environment.

4. CONCLUSION

This study showed that continued human dietary exposure to pesticides could modulate serious health risks in the future, especially where pesticide regulation is poor. The contents of six pesticide residues in oat samples, and five in rice, were found to exceed their MRL values. The endrin aldehyde content in oat consumed by children suggest possible health risk to such individual. Similarly, the levels of heptachlor in rice consumed by both children and adults can trigger non-carcinogenic health risks. In summary, the cumulative HQs of pesticide residues in both oat and rice samples for children and adults can cause non-carcinogenic health problems for consumers.

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