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Monitoring and health risk assessment of organochlorine pesticide residue in some leafy and fruiting vegetables from Lagos State, Southwestern Nigeria

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Abstract

This study investigated the health risk of pesticide residue in some leafy and fruiting vegetables namely fluted pumpkin (*f. Telfairia occidentalis*), green amaranth (*Amaranthus viridis*) and scotch bonnet (*capsicum chinense*) which are commonly eaten in Nigeria. Pesticide can impart harmful effect on health, it can be a source of exposure to health issues to Nigerians. Fluted pumpkin, green amaranth and scotch bonnet were purchased from the major wholesale market in Lagos state, Nigeria through which food stuffs are distributed in Lagos to retailers. Pesticide residues including alpha lindane, delta lindane, heptachlor, heptachlor epoxide, aldrin, trans – chlordane, p,p'- DDE, dieldrin, endrin, endrin ketone, endosulfan, endosulfan sulfate, endosulfan ether and methoxychlor were extracted from each sample using dichloromethane, and anhydrous sodium sulphate and sodium chloride to enhance liquid-liquid partitioning. Solid phase extraction using agilent cartridges was used to clean-up the extracts. The extracts were analysed for pesticide residues using gas chromatography coupled to mass spectrometer (GC-MS). From the results of the study, most of the pesticide residues were below detectable limit. Endosulfan ether was the only pesticide detected in all the vegetables from all the sources of the vegetables. The mean concentration (mg/kg) of the pesticide residues ranged from 0.0003-4.06 mg/kg. The mean concentration of some of the pesticide residues in the leafy and fruiting vegetables were either above, within and below the maximum residue limit mg/kg (MRL) recommended by FAO/WHO. The estimated daily intake (EDI) mg/kg bw/day of the pesticide residues ranged from 4.25×10^{-7} to 5.76×10^{-3} mg/kg bw/day. The EDI of all the pesticide residues in the samples are below the acceptable daily intake (ADI) mg/kg bw/day of the pesticide residues as recommended by FAO/WHO, except the EDI of heptachlor in all the samples which are above the recommended ADI. The hazard quotient and hazard index of the pesticide residues in the vegetables ranged from 8.50×10^{-7} - 2.50 for hazard quotient and 0.07 - 3.2 for hazard index. Incremental lifetime cancer risk (ILCR) values for carcinogenic pesticide residues in the vegetables ranged from 1.3×10^{-5} - 2.1×10^{-3}. Few of the vegetables had ILCR values for the carcinogenic pesticide residues above acceptable values recommended for ILCR. Consumption of the vegetables with high EDI, HQ, HI and ILCR values may pose health issues to consumers. There is need for continuous monitoring of pesticide residues in food samples in Nigeria.

Keywords: Fluted pumpkin, green amaranth, hazard index, incremental lifetime cancer risk, pesticide residue

1. Introduction

Agriculture is the main occupation of about 36.5% of Nigeria population through which they earn their livelihood (FAO, 2016). It is the food crops produced by the farmers that feed Nigerians mainly. However, the means of livelihood of farmers and food security in Nigeria is usually threatened by pest infestation which makes the Nigeria farmer resort to use of pesticides to control pest so as to ensure quality and higher crop yield and income generation from sale of their produce. The danger inherent in the use of pesticides is that pesticides are toxic; they can cause acute and chronic health issues at certain concentration; while some are highly toxic even at low concentration and have been banned from use in agricultural practices, however, due to their low cost are still patronized by developing countries (WHO, 2018). Pesticides act faster in elimination of pest, that is why most farmers resort to their use. In order to safeguard public health, the World Health Organization (WHO) recommends the use of non-genotoxic pesticides because they do not have any effect on the DNA. These pesticides will not cause cancer or mutation at certain

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concentration, but, above a certain safe level of exposure will cause adverse health effects such as cancer and reproduction problems (WHO, 2018). For this reason, WHO also recommended maximum residue limit of these pesticides on or in crops (WHO, 2018). Pesticide that remains on or in food after application of pesticide is termed pesticide residue (McNaught and Wilkinson, 2019). Pesticide residue can be a source of exposure of humans to pesticide when food containing pesticide residue is ingested. Food safety can be affected when the pesticide residue in food crop is above maximum residue limit (MRL). To keep pesticide residue on or in crops at acceptable maximum residue limit means applying exact recommended concentration of pesticides on crops (WHO, 2018). However, in Nigeria, some farmers apply pesticides on their crops before and after harvest for pest control indiscriminately without compliance to recommended concentration, this might bring the concentration of the applied pesticide to above acceptable maximum residue limit. Indiscriminate application of pesticides by some Nigeria farmers may be attributed largely to the fact that greater percentage of the farmers are illiterates.

Pesticides are of different classes which include among others organochlorine, organophosphorus and carbamate pesticides (Nicolopouslou-Stamati *et al*., 2016; Jayaraj et al., 2016). Organochlorine (OC) pesticides are group of chlorinated compounds while organophosphate (OP) pesticides are esters of phosphoric acid and carbamate pesticides are organic compounds derived from carbamic acid (NH_2COOH) (Jayaraj *et al.*, 2016). Unlike organophosphate pesticides which degrade rapidly by hydrolysis when expose to sunlight, air and soil and are also biodegraded by soil bacteria though small amounts may persist in food and drinking water (Amir, 2019; Jayaraj *et al.*, 2016) and carbamates which break down in the environment within weeks or months (Goel and Aggarwal, 2007), organochlorine pesticides are very persistent because of which they are classified among the group of chemical compounds called persistent organic pollutants (POPs) (Jayaraj *et al.*, 2016). Due to their persistent nature organochlorine pesticides accumulate in the environment, bioaccumulate in lipid- rich tissues and biomagnify in the food chain (Rogan and Chen, 2005; Dietz *et al.*, 2000). Generally, organochlorine pesticides are characterized by high lipophilicity, bioaccumulation, long half – life and potential of long-range transport, all of which increase the chances of organochlorine pesticides contaminating the environment even after many years of application (Jayaraj *et al.*, 2016). Organochlorine pesticides are toxic compounds linked with short term (chronic) and long-term (acute) health issues such as convulsions, headache, dizziness, nausea, vomiting, tremors, confusion, slurred speech, endocrine disruption, neurological damage, reproductive disorder, fetal defects, thyroid dysfunction, and more others (Mnif *et al.*, 2011; Jayaraj *et al.*, 2016; Witcza *et al.*, 2021). Due to their toxicity the Stockholm Convention a global treaty on protection of human health and the environment from POPs banned the use of many organochlorine pesticides such as aldrin, chlordane, dieldrin, endrin, heptachlor, lindane, endosulfan, mirex, toxaphene etc (Moy, 2014; Jayaraj *et al.*, 2016). However, they are still been used in many developing countries because of their low cost (Jayaraj *et al.*, 2016; FAO, 2005; WHO, 2018).

In the present study, the pesticide residue of interest are organochlorine pesticides namely alpha-lindane, delta-lindane, endosulfan ether, heptachlor, aldrin, heptachlor epoxide, trans- chlordane, p,p'-DDE, dieldrin, endrin, endosulfan, endosulfan sulfate, endrin ketone and methoxychlor. Studies have shown that aldrin and dieldrin cause neurotoxic, reproductive, developmental, immunological, genotoxic and other health effects in human (USEPA, 2003), while chlordane causes hepatotoxicity and neurotoxicity in humans and is considered a possible human carcinogen (ATSDR, 1997; CDC, 2022; Koshlukova and Reed, 2014) and endosulfan causes DNA damage and mutation in humans, decreases white blood cell count, affects semen quality, sperm count, spermatogenial cells, sperm morphology and other defects in male sex hormones in humans (Pendey *et al*., 1990; Singh *et al*., 2007). Lindane (α, β, γ, δ) affects kidney, immune system, induces birth defects, causes cancer, neurotoxicity, reproductive toxicity and hepatoxicity in humans (ATSDR, 1999; Sahoo *et al.*, 2008; Bano and Bhatt, 2010; Vijaya *et al.*, 2011). Methoxychlor is linked with neurotoxicity and reproductive toxicity in rats (ATSDR, 2002; Cummings and Gray, 1989), heptachlor and heptachlor epoxide can alter the immune system during development, cause hepatotoxicity, neurotoxicity, developmental toxicity and they are classified as possible human carcinogen (ATSDR, 1993; Reed and Koshlukova, 2014). High levels of endrin causes neurotoxicity and sudden collapse or death (ATSDR, 2021). p, p'- DDE can cause reduction in the duration of lactation and increased chance of having a premature baby (ATSDR, 2002), it may cause endocrine disruption and is carcinogenic (Eskenazi *et al.*, 2006) and neurodevelopment problems in children (Turusoy *et al.*, 2002).

The use of pesticides in Nigeria is regulated by the National Agency for Food and Drug administration and Control (NAFDAC), National Environmental Standards and Regulation Enforcement Agency (NESREA) and Federal Ministry of Agriculture and Rural Development (NAFDAC, 2016). Among the functions of the National Agency for Food and Drug Administration and Control (NAFDAC) includes regulation and control of the manufacture, importation, exportation, distribution, advertisement, sale and use of chemicals, in addition, it registers chemicals and review chemical regulations in accordance with international conventions while National Environmental Standards and Regulation Enforcement Agency (NESREA) enforce laws, policies and standards relating to the environment, international agreements, including chemicals, hazardous waste and legislation on sound chemical management, safe use of pesticides and disposal of spent packages among other functions. In response to its mandate, NAFDAC adopted the Stockholm Convention agreement of 2014 and 2017 which banned the use of some OC pesticides previously mentioned, and included these pesticides among the list of chemicals banned for usage in Nigeria (NAFDAC, 2018). The list of approved pesticides is not available on NAFDAC website, however, market survey on the current types of pesticides (insecticide and herbicide) sold in the Nigeria market shows that the active ingredients in the pesticides, though, sold under different trade name are glyphosate, chlorpyfos, dichlorvos, cypermethrin, deltamethrin, permethrin, dimethoate, paraquat dichloride (paraquat), mancozeb, atrazine, butachlor, alachlor and propanil. This finding is in agreement with the findings of Dugje *et al.* (2008) and report of Abdulkareem (2021). These pesticides belong to the organophosphate, pyrethroids, carbamate, chloracetanilide and triazine class of pesticides. However, several studies on pesticide residues in vegetables, grains, cereals and other types of food crops have revealed the presence of organochlorine pesticide residues in them. Such as the works of Sosan *et al*. (2018), Dada *et al.* (2020), Ibrahim *et al. (*2018), Unyimadu *et al*. (2018), Adeleye, *et al*. (2019), Sosan, *et al.* (2020), Olufade, *et al*. (2014), Oyeyiola *et al.* (2017), and Otitoju and Lewis (2021) just to mention a few. The presence of organochlorine pesticide residue detected in recent studies may be due to the persistent nature of this class of pesticide and its ability to travel long range distance. Although drawing his conclusion from Bhuiyan *et al.* (2009) who reported that banned OC pesticides in Bangladesh are still in use under different names or labels, Olufade *et al.* (2014) presume that banned OC pesticides may likely be sold under different names or label in Nigeria or added as one of the active ingredients to other insecticides currently sold and used by Nigeria farmers.

Arising from the fact that OC pesticides have potent public health issues, the need for periodic monitory and establishment of data for the contamination levels of OC in Nigeria, the present study was designed to investigate the levels of concentrations of common OC pesticides (alpha and delta lindane, heptachlor and heptachlor epoxide, aldrin, trans-chlordane, p,p'- DDE, dieldrin, endrin and endrin ketone, endosulfan, endosulfan sulfate, endosulfan ether and methoxychlor) in Nigeria and predict their health risk factor using various approved indices.

2. Methodology

2.1. Chemicals and reagents

High purity 99% reference standard mixed organochlorine (OC) pesticides containing fourteen (14) pesticides namely, heptachlor epoxide, heptachlor, methoxychlor dieldrin, endrin, endrin ketone, alpha-lindane, delta-lindane, endosulfan ether, aldrin, trans- chlordane, p,p'-DDE, endosulfan and endosulfan sulfate were purchased from AccuStandard (New Haven, USA). Dichloromethane, n-hexane and methanol were purchased from Merck, Darmstadt (Germany). Anhydrous sodium sulphate and sodium chloride were purchased from Sigma – Aldrich (USA).

2.2. Sample collection and preparation

Fluted pumpkin (*f. Telfairia occidentalis)*, green amaranth (*Amaranthus Viridis)* and scotch bonnet (*capsicum chinense*) were purchased in January 2021 from Mile 12 market, Ketu, Lagos state, Nigeria. Mile 12 market is a wholesale market for food crops where farm products from all parts of Nigeria are brought and purchased by retailers who retail these crops in all parts of Lagos state, Nigeria. The fluted pumpkin, green amaranth and scotch bonnet were from different sources, and the sources as provided by the retailers are as follows; fluted pumpkin: Sabo – Ikorodu, Adamo – Ikorodu, Ita – Oluwo and Odongunyan abbreviated as SIP, AIP, IP and OP respectively. Green amaranth: Ilorin, Sango – ota and Abeokuta abbreviated as IG, SG and AG, while scotch bonnet from Sokoto, Abeokuta, Niger and Oyo abbreviated as SS, AS, NS and OS. The samples which were randomly selected were package in clean dark properly labelled polyethene bags and taken immediately to the laboratory, stored in refrigerator preset at 4^0C and analysed within three days as prescribed in the document SANTE/11813/2017 (European Commission, 2017). The samples were prepared as in Kelle, (2020); the samples were washed with water, milled and homogenised and stored in the refrigerator at temperature of - 20^0 C. 2g of each homogenised sample was transferred in a 50 ml conical flask, after which 10 ml of dichloromethane was added, stoppered and sonicated for two hours at 270 rpm. Then vortexed for 1 minute. 4g of anhydrous sodium sulphate and 1g of sodium chloride were added to the mixture and sonicated for 20 minutes. The mixture was allowed to stand for 5 minutes and centrifuged for 5 minutes at 2500 rpm. The supernatant was removed and cleaned – up by solid phase extraction using agilent cartridges initially conditioned with 10 ml methanol. The supernatant was eluted with 5 ml dichloromethane, and the eluate concentrated using nitrogen concentrator at room temperature to 1 ml and transferred into GC vials for GC – MS analysis.

2.3 Standard preparation

100 $\mu g\ ml^{-1}$ stock solution of standard mixed organochlorine (OC) pesticide was prepared in n – hexane and stored in dark flasks at -20 °C in refrigerator until use. 2.5 µg ml^{-1}, 5 µg $ml^{-1}$and 10 µg ml^{-1}working standard solutions were prepared in n - hexane from the stock solution daily (Kelle, 2020). These were used for the calibration of the instrument.

2.4 GC–MS analysis and method validation

The eluates were analysed using GC 7890A Agilent coupled with an electron capture detector and interfaced with mass selective detector model 5975 C (MSD). The stationary phase of the GC was HP 5 column (30 m ×320 µm ×0.25 µm film thickness) with helium gas (99.9 %) as carrier gas at a constant flow rate of 0.5 $ml\ min^{-1}$. 1 µL standard or sample was injected in split less mode at 250^0 C, the GC oven was operated at an initial temperature of 80^0C for 4 minutes and then heated at a rate of 5^0C min^{-1} to 240^0C and heated again at a rate of 11^0C to 280^0C and held for 5 minutes. Electron ionization of the mass spectrometer was at 70 ev and the ion source temperature was at 250^0C. Constituents of the mixed organochlorine pesticide reference standard were identified by comparing the mass spectra with a known standard using 5975 MSD (mass detector) with Chemstation software library. The analytical process was performed in triplicate.

Validation of the method was done by weighing 10g of the samples and dividing the 10g into two equal portions. One portion was spiked with 5 µg ml^{-1} of mixed OC pesticide reference standard and mixed thoroughly. The other portion was left un-spiked. Both the spiked and un-spiked samples were subjected to the same extraction, clean-up and GC – MS analysis as the test samples previously described. The concentrated extracts were analysed by GC-MS. The linearity, limits of detection and quantification, range, accuracy, precision and sensitivity were the analytical parameters validated. From the analytical curve plots for each pesticide the linearity, limits of detection and quantification, sensitivity and range were determined. The limits of detection (LOD) and quantification (LOQ) were calculated from equation 1 and 2 respectively, (Escarlet *et al*., 2018).

$$\text{LOD} = 3.3 \times \frac{S_y}{b} \quad (1)$$

where b is the slope of the analytical curve and s is the residual standard deviation of the analytical curve.

$$\text{LOQ} = 3 \times \text{LOD} \quad (2)$$

The selectivity of the method was evaluated by the separation of the analytes while the accuracy of the method was determined as the average of three replicates and the precision of the method by recovery study. The % recovery was calculated from equation 3.

$$\%\ \text{Recovery} = \frac{X_1 - X_2}{Y} \quad (3)$$

where X_1 is the concentration of the spiked sample, X_2 is concentration of the un-spiked sample and Y is the concentration of the OC reference standard added to the spiked sample.

2.5. Health risk assessment

2.5.1 Estimation of daily pesticide residue intake

To determine the health risk associated with daily consumption of the organochlorine pesticide residues in the test samples of fluted pumpkin (*f. Telfairia occidentalis),* green amaranth (*Amaranthus Viridis)* and scotch bonnet (*capsicum chinense*), the estimated daily intake (EDI, mg/kgbw/day) of the pesticide residues in each of the sample was computed using equation 4 and compared with established acceptable daily intake (ADI, mg/kg bw/day) for each organochlorine pesticide residue in food (US EPA, 2000). .

$$\text{EDI} = \frac{C_R\ X\ IR}{BW} \quad (4)$$

where C_R is the average concentration of pesticide residue in the samples (mg/kg), and IR is the daily ingestion rate of the samples. The daily ingestion of vegetables for an adult Nigerian is 89.3 g/person/day which is 0.0893 kg/day (WHO, 2017), while the average body weight BW for an adult Nigerian used in this study is 63 kg (Kelle *et al.*,2020).

2. 5 .2 Non – carcinogenic risk

Hazard quotient and hazard index were used to estimate non carcinogenic risks of the respective organochlorine pesticide residue in each of the vegetable. Hazard quotient is the ratio of exposure to a chemical toxicant and the reference dose (Antionne *et al.*, 2017). It is expressed as shown in equation 5 (US EPA, 2014; Gerba, 2019).

$$\text{HQ} = \frac{EDI}{RfD} \quad (5)$$

Where RfD the oral reference dose (mg/kg/day) is the highest level at which no adverse health effects are expected on human population through daily exposure to a chemical toxicant. HQ < 1 implies no adverse health effects while HQ > 1 signifies possibility of non-carcinogenic risks or adverse health effects. The hazard quotient for the respective pesticide residue in each of the vegetable was estimated as the ratio of each computed estimated daily intake (EDI) of the individual pesticide residue in each sample to the RfD for each pesticide type. The hazard index expressed as shown in equation 6 (US EPA, 2014; Gerba, 2019).

$$\text{HI} = \Sigma\text{HQ} \quad (6)$$

Is the sum of hazard quotients of each chemical toxicant (organochlorine pesticide residue) in a sample. HI < 1 indicates non potential for adverse non-carcinogenic health effects or chronic risk and HI > 1 suggest potential for chronic risk or adverse non-carcinogenic health effects (USEPA, 2005; USEPA, 2014; Gerba, 2019). While hazard quotient estimates non cancer risks due to a single chemical toxicant, hazard index computes non cancer risks due to multiple chemical toxicant or cumulative effect of chemical toxicants (Gerba, 2019) that affect the same target organ or organ system (USEPA, 2005). The non-cancer risk due to cumulative effect of all the pesticide residue type was estimated using equation 6.

2.5.3 Carcinogenic risk

Incremental Lifetime Cancer Risk (ILCR) model used to evaluate the possibility of developing cancer through ingestion of carcinogenic chemical toxicants was applied to only carcinogenic organochlorine pesticide residues (only those pesticide residues with evidence of probability or possibility of causing cancer) in the vegetables to determine the possibility of developing cancer through ingestion of these pesticide residues in the vegetables. ILCR is expressed as in equation 7 (USEPA, 2014; Gerba, 2019).

$$ILCR = CDI \times CSF \quad (7)$$

CDI is chronic daily intake of chemical carcinogen, mg/kg BW/day, CSF is the cancer slope factor (CSF).

$$CDI = \frac{EDI\ X\ \ EF\ X\ ED\ X\ CF}{AT}$$

Where EF is exposure frequency (days/ year); 365 days/year, ED is exposure duration (years); 54 years (life expectancy of an adult Nigerian) (World Bank, 2018). AT is average time (days) (the period over which exposure is averaged), for carcinogens the average time is exposure frequency (days/years) multiplied by exposure duration, CF is units' conversion factor. When the computed cancer risk is between $1\ x\ 10^{-6}$ to $1 \text{ x } 10^{-4}$, it is acceptable (US EPA, 2014).

3. Results and Discussion

3.1. Mean concentration of organochlorine pesticide residues

Table 1 shows the limit of detection and quantification, and % recovery of the organochlorine pesticide residues, while, the mean concentrations of the organochlorine (OC) pesticide residues in the vegetables are presented in Table 2. Heptachlor, heptachlor epoxide, trans – chlordane and p, p'-DDE residues in all the pumpkin samples from the various sources were below detectable limit, also, alpha lindane, delta lindane, aldrin, endosulfan sulfate, endrin ketone and methoxychlor residues were below detectable limit in all the pumpkin samples except pumpkin sample from AIP. In this pumpkin sample, the mean concentration of alpha lindane was 0.01 ±0.0036 mg/kg, delta lindane 0.02 ±0.0048 mg/kg, aldrin 0.001 ±0.00045 mg/kg, endosulfan sulfate 0.001 ±0.00073 mg/kg, endrin ketone 0.001 ±0.0057 mg/kg and methoxychlor 0.0003 ±0. 00083 mg/kg. Endrin residue in pumpkin sample from OP was below detectable limit, but detected in pumpkin samples from SIP (0.019 ± 00024 mg/kg), AIP (0.011 ±0.0036 mg/kg) and IP (0.006 ±0.0081 mg/kg), while, dieldrin, endosulfan and endosulfan ether residues were detected in all the pumpkin samples from all the sources with mean concentration ranging from 0.001 ±0.0013 –0.009 ±0.0044 mg/kg dieldrin, 0.001 ±0.0092 – 0.004 ±0.0083 mg/kg endosulfan and 0.0096 ±0.00023 – 4.062 ±0.0097 mg/kg endosulfan ether. Alpha lindane, delta lindane, heptachlor, heptachlor epoxide, aldrin, trans chlordane, endrin, endosulfan sulfate and endrin ketone residues were below detectable limit in green amaranth samples from all the sources, while, dieldrin and endosulfan residues were detected in green amaranth samples from IG (0.006 ±0.0019 mg/kg dieldrin and 0.002 ±0.00068 mg/kg endosulfan) and SG (0.002 ±0.00051 mg/kg dieldrin and 0.001 ±0.0015 mg/kg endosulfan), but were below detectable limit in sample from AG. Methoxychlor and p,p'- DDE residues were below detectable limit in green amaranth samples from IG and SG, but, were detected in green amaranth sample from AG with mean concentration of 0.03 ± 0.0011 mg/kg p,p'- DDE and 0.009 ±0.00022 mg/kg methoxychlor. Endosulfan ether residue was detected in all green amaranth from all the sources with mean concentration ranging from 0.02 ±0.00038 – 0.184 ±0.0041 mg/kg. Aldrin, endrin ketone and methoxychlor residues were below detectable limit in scotch bonnet from all the sources, also, alpha and delta lindane were below detectable limit in scotch bonnet from AS and OS, but were detected in scotch bonnet from SS (0.009 ±0.000573 mg/kg alpha lindane and 0.002 ±0.0015 mg/kg delta lindane) and NS (0.005 ± 0.0019 mg/kg alpha lindane and 0.002 ±0.00087 mg/kg delta lindane). Heptachlor, Heptachlor epoxide and endosulfan sulfate residues were below detectable limit in scotch bonnet from SS and OS but detected in scotch bonnet from AS and NS. The mean concentration of heptachlor from AS was 0.37 ±0.0837 mg/kg and NS 0.34 ±0.055 mg/kg, while, heptachlor epoxide was 0.019 ± 0.0054 mg/kg in scotch bonnet from AS and 0.02 ±0.0098 mg/kg in scotch bonnet from NS, and endosulfan sulfate was 0.80 ± 0.0091 mg/kg in scotch bonnet from AS and 0.30 ±0.0079 mg/kg in scotch bonnet from NS. Trans-chlordane and P, P'-DDE residues were below detectable limit in scotch bonnet from AS, OS and NS but were detected in scotch bonnet sample from SS with mean concentrations of 0.0017 ±0.00052 mg/kg and 0.184 ±0.0018 mg/kg respectively. Dieldrin residue was below detectable limit in scotch bonnet from SS, AS and NS but was detected in scotch bonnet from OS with mean concentration of 0.001 ±0.0013 mg/kg. Endrin residue was below detectable limit in scotch bonnet from SS and AS but detected in scotch bonnet sample from OS and NS with mean concentrations of 0.02 ±0.0027 mg/kg and 0.004 ±0.00097 mg/kg respectively. Endosulfan residue was below detectable limit in scotch bonnet from NS, but was detected in scotch bonnet sample from SS (0.123 ±0.0011 m/kg), AS (0.005 ±0.0071 mg/kg) and OS (0.002 ±0.00054 mg/kg). Most of the pesticide residues were below detectable limit (BDL) (Table 2) in the vegetables, suggesting that they are not used for pest control, which implies

Table 1: Limit of detection and quantification and % Recovery of organochlorine pesticide residue in sample

Pesticide	**% Recovery**	**LOD (mg/kg)**	**LOQ (mg/kg)**
alpha lindane	84.71	0.000010	0.00003
delta lindane	89.33	0.000028	0.000084
heptachlor	91.11	0.000011	0.000033
aldrin	90.40	0.000014	0.000042
heptachlor epoxide	93.57	0.0000061	0.0000183
trans-chlordane	88.63	0.0000077	0.0000231
p,p'-DDE	95.61	0.0000055	0.0000165
dieldrin	97.34	0.000042	0.000126
endrin	98.26	0.000026	0.000078
endosulfan	89.10	0.000011	0.000033
endosulfan sulfate	94.13	0.000015	0.000045
endrin ketone	87.94	0.000025	0.000075
methoxychlor	98.71	0.0000042	0.0000126
endosulfan ether	94.30	0.000070	0.00021

LOD: Limit of detection, LOQ: Limit of quantification

Table 2: Mean concentration (mg/kg) of pesticide residue in fluted pumpkin (*f. Telfairia occidentalis*), green amaranth (*Amaranthus Viridis*) and Scotch bonnet (*Capsicum chinese*)

Pesticide	Type and source of leafy and fruiting vegetables											
	Pumpkin Mean + SE				Green Amaranth Mean + SE			Scotch bonnet Mean + SE				Maximum residue limit (MRL) mg/kg
	SIP	AIP	IP	OP	IG	SG	AG	SS	AS	OS	NS	
alpha lindane	BDL	0.01± 0.0036	BDL	BDL	BDL	BDL	BDL	0.009± 0.000573	BDL	BDL	0.005± 0.0019	0.008 (ATSDR)
delta lindane	BDL	0.02± 0.0048	BDL	BDL	BDL	BDL	BDL	0.002±0.0015	BDL	BDL	0.002±0.00087	0.005(ATSDR)
heptachlor	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.37±0.0837	BDL	0.34±0.055	0.0006 (ATSDR)
aldrin	BDL	0.001± 0.00045	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.1(fruiting vegetable) 0.5(leafy vegetable) FAO/WHO
heptachlor epoxide	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.019± 0.0054	BDL	0.02±0.0098	0.0006(ATSDR)
trans-chlordane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.0017±0.00052	BDL	BDL	BDL	0.02(FAO/WHO)
p,p'-DDE	BDL	BDL	BDL	BDL	BDL	BDL	0.03± 0.0011	0.184±0.0018	BDL	BDL	BDL	0.01(FAO/WHO
dieldrin	0.0014 ± 0.00037	0.009± 0.0044	0.0043± 0.00078	0.008± 0.0029	0.006± 0.0019	0.002± 0.00051	BDL	BDL	BDL	0.001± 0.0013	BDL	0.1(fruiting vegetable) 0.5(leafy vegetable) FAO/WHO
endrin	0.019± 00024	0.011± 0.0036	0.006±0.0081	BDL	BDL	BDL	BDL	BDL	BDL	0.02±0.0027	0.004±0.00097	0.05(FAO/WHO
endosulfan	0.002± 0.0064	0.017± 0.0055	0.004±0.0083	0.001± 0.0092	0.002± 0.00068	0.001± 0.0015	BDL	0.123±0.0011	0.005± 0.0071	0.002± 0.00054	BDL	0.007(ATSDR)
endosulfan sulfate	BDL	0.001± 0.00073	BDL	BDL	BDL	BDL	BDL	BDL	0.80± 0.0091	BDL	0.30±0.0079	0.007(ATSDR)

endrin ketone	BDL	0.001± 0.0057	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.05 FAO/WHO
methoxychlor	BDL	0.0003 ±0. 00083	BDL	BDL	BDL	BDL	0.009±0.0 0022	BDL	BDL	BDL	BDL	0.01(UNEP
endosulfan ether	0.103± 0.0018	4.062± 0.0097	0.01±0.0 057	0.0096 ±0.000 23	0.02±0. 00038	0.03±0. 00092	0.184±0.0 041	0.142±0.0 087	0.38±0. 0049	0.10±0. 0088	0.34±0.0 094	0.007(ATSDR)

n= 3, SE: Standard error of mean, SIP: Sabo – Ikorodu Pumpkin, AIP: Adamo – Ikorodu Pumpkin, IP: Ita – Oluwo Pumpkin, OP: Odungunyan Pumpkin, IG: Ilorin Green amaranth, SG: Sango – ota Green amaranth, AG: Abeokuta Green amaranth, SS: Sokoto Scotch bonnet, AS: Abeokuta Scotch bonnet, NS: Niger Scotch bonnet, Oyo Scotch bonnet, BDL: Below detectable limit, ATSDR: Agency for Toxic Substances and Disease Registry, FAO: Food and Agriculture Organisation, WHO: World Health Organisation, UNEP: United Nations Environment Programme

compliance to their non-usage as recommended by World Health Organization (WHO) and the regulatory agency which regulates the use of pesticide in Nigeria, the National Agency for Food and Drug Administration and Control (NAFDAC). Most of the pesticide residues detected in the vegetables had mean concentration below their MRL, while the remaining had mean concentration above their MRL. Maximum residue limit (MRL) is the highest level of a pesticide residue that is legally tolerated in or on food when pesticides are applied correctly in accordance with good agricultural practice (FAO/WHO, 2018). Exceedance of pesticide residue above their MRL could pose harm to consumers. The result obtained, shows that the mean concentrations of the OC pesticide residues in some of the analysed vegetables are above their MRL (Table 2). This necessitates regular monitoring of pesticide residues in food crops to ensure that food crops consumed are safe. The mean concentration mg/kg of alpha lindane in fluted pumpkin from AIP (0.01 ± 0.0036 mg/kg) and scotch bonnet from SS (0.009 ±0.000573 mg/kg) are above the MRL for alpha lindane (0.008 mg/kg) in leafy and fruiting vegetables. The MRL of delta lindane in leafy and fruiting vegetables is 0.005 mg/kg, the mean concentration of this pesticide residue in fluted pumpkin from AIP (0.02 ± 0.0048 mg/kg) was above the MRL for delta lindane. The mean concentration of heptachlor in scotch bonnet from AS (0.37 ±0.0837 mg/kg) and NS (0.34 ±0.055 mg/kg) were above the MRL of heptachlor (0.0006 mg/kg) in fruiting and leafy vegetables, likewise, the mean concentrations of heptachlor epoxide in scotch bonnet from AS (0.019 ± 0.0054 mg/kg) and NS (0.02 ±0.0098 mg/kg) were above the MRL of heptachlor epoxide (0.0006 mg/kg) in vegetables. Other pesticide residues in excess of their MRL include P, P'-DDE (MRL 0.01 mg/kg) in scotch bonnet from SS (0.184 ±0.0018 mg/kg), endosulfan (MRL 0.007 mg/kg) in fluted pumpkin from AIP (0.017 ±0.0055 mg/kg) and scotch bonnet from SS (0.123 ±0.0011 mg/kg), endosulfan sulphate (MRL 0.007 mg/kg) in scotch bonnet from AS (0.80 ± 0.0091 mg/kg) and NS (0.30 ±0.0079 mg/kg) and endosulfan ether (MRL 0.007 mg/kg) whose mean concentration in all the vegetables from specific source were above the MRL for endosulfan ether in vegetables. These high concentration values may be attributed to the persistent nature of these pesticides. p.p'- DDE has very high half-life, while heptachlor has a high half-life and endosulfan and its metabolites have moderate half-life which makes these pesticides persist in the environment (Jayaraj *et al.*, 2016; Reed and Koshlukova, 2014). Some studies conducted on pesticide residues on food crops obtained from Nigeria showed high level of pesticides residue in excess of 0.1 mg/kg, Olufade *et al.* (2014) detected aldrin in the range of 0.177 – 0.2.64 mg/kg, heptachlor 0.402 – 0.546 mg/kg, endrin 0.152 – 0.199 mg/kg, chlordane 0.154 – 0.185 mg/kg and endosulfan 0.222 – 0.264 mg/kg in cowpea from markets within Ile – Ife, South – Western, Nigeria. They also detected aldrin in the range of 0.580 – 1.050 mg/kg, heptachlor 0.354 – 0.488 mg/kg, endrin 0.158 – 0.198 mg/kg and endosulfan 0.345 – 0.427 mg/kg in dried yam chips from markets within Ile – Ife, South – Western, Nigeria. Similarly, high concentration of β lindane (1.690 – 2.340 mg/kg) and δ lindane (1.450 – 1.74 mg/kg) were detected in Capsicum annum L obtained from Ota market, Nigeria (Nsikak and Aruwajoye, 2011) and Adeleye *et al*., 2019) detected 2.987 ± 0.391 mg/kg and 3. 491 ± 0.376 mg/kg of endrin aldehyde, in amaranth and fluted pumpkin respectively from South – Western, Nigeria and endosulfan sulfate (0.661 ± 0.280 mg/kg and 2.775 ± 0.644 mg/kg in amaranth and fluted pumpkin respectively from South – Western, Nigeria. Also, Sosan *et al.*, 2020 detected heptachlor (0.108 ± 0.100 and 0.758 ± 0.02 mg/kg), endosulfan sulfate (1.030 ± 0.173 mg/kg) and aldrin (0.139 ± 0.022 – 3.219 ± 2.769 mg/kg) in maize – based complementary food samples in Nigeria. Other works which detected high levels of OC pesticides in food crops consumed in Nigeria include the work of Sosan *et al.* (2018), Dada *et al*. (2020), Ibrahim *et al*. (2018) and Unyimadu *et al*. (2018) among others.

3.2 Estimated daily intake (EDI)

The estimated daily intake EDI (mg/kgbw/day) of the organochlorine pesticide residues in the vegetables with the recommended acceptable daily intake (ADI) mg/kgbw/day for each of the organochlorine pesticide residue are presented in

Table S1 (supporting information). EDI was estimated for only those pesticide residues detected in some vegetables. The estimated daily intake (EDI) of the pesticide residues in the vegetables which contained them are; alpha lindane 7.08 x 10^{-6} to 2.76 x 10^{-5} mg/kg bw/day, delta lindane 2.40 x 10^{-6} to 3.11 x 10^{-6} mg/kg bw/day, heptachlor 4.76 x 10^{-4} to 4.81 x 10^{-4} mg/kg bw/day, aldrin 4.26 x 10^{-7} to 5.76 x 10^{-3} mg/kg bw/day aldrin 1.417 x 10^{-6} mg/kg bw/day, heptachlor epoxide 2.55 x 10^{-5} to 3.26 x 10^{-5} mg/kg bw/day, transchlordane 2.40 x 10^{-6} mg/kg bw/day, p.p'- DDE 4.00 x 10^{-5} to 2.60 x 10^{-4} mg/kg bw/day, dieldrin 5.66 x 10^{-6} to 2.70 x 10^{-5} mg/kg bw/day, endosulfan 8.50 x 10^{-7} to 1.74 x 10^{-4} mg/kg bw/day, endosulfan sulphate 1.417 x 10^{-6} to 1.10 x 10^{-3} mg/kg bw/day, endrin ketone 1.40 x 10^{-6} mg/kg bw/day, methoxychlor 4.25 x 10^{-7} to 1.275 x 10^{-5} mg/kg bw/day and endosulfan ether 1.36 x 10^{-5} to 5.76 x 10^{-3} mg/kg bw/day. Except for scotch bonnet from AS and NS (Table S1) whose EDI for heptachlor residue 4.76 x 10^{-4} mg/kgbw/day and 4.81 x 10^{-4} mg/kgbw/day respectively, were above the ADI (0.0001 mg/kgbw/day) for heptachlor, the EDI for the other pesticide residues in the vegetables were below their ADI. This implies that these vegetables may not cause health risk to its consumers. Acceptable daily intake (ADI) mg/kgbw/day is the maximum amount of a chemical that can be ingested daily over a lifetime with no appreciable health risk, and is based on the mg/kg highest intake that does not give rise to observable adverse effects (Dennis and Wilson, 2003). Ingestion of scotch bonnet from AS and NS may cause health risks to its consumers, because heptachlor has been shown to exhibit carcinogenicity in mice and it causes headache, muscle twisting, anxiety, dizziness and weakness (Pohanish, 2015).

3.3 Hazard quotient and hazard index

The hazard quotient (HQ) and hazard index (HI) of the OC pesticide residues in the vegetables are presented in **Table S2 (supporting information).** These values were calculated for only those pesticide residues detected in some vegetables. The hazard quotients of the OC pesticide residues in the vegetables are less than one, except heptachlor epoxide residue in scotch bonnet from AS (HQ = 2.0) and NS (HQ = 2.50), and endosulfan ether residue in fluted pumpkin from AIP (HQ = 1.0). This suggest that the vegetables from sources with HQ $<$ 1 are safe for consumption with no indication of non-carcinogenic risk or adverse health effect while scotch bonnet from AS and NS, and fluted pumpkin from AIP may cause adverse health effects. The hazard index which is the sum or combination of the HQ of each pesticide residue type in a specific vegetable (sample) from a specific source is less than one for all the vegetables, excluding the hazard index for fluted pumpkin from AIP (HI = 1.5), scotch bonnet from AS (HI = 3.2) and NS ((HI = 2.76). This implies that the vegetables with HI $<$ 1 may not cause chronic risks, but fluted pumpkin from AIP and scotch bonnet from AS and NS may cause chronic health risk.

3.3 Incremental Lifetime Cancer Risk (ILCR)

The result of Incremental Lifetime Cancer Risk (ILCR) for the OC pesticide residues in the vegetables is presented in **Table S3 (supporting information).** The ILCR value for alpha lindane residue in pumpkin sample from AIP is 1.3 x 10^{-5}, in green amaranth sample from SS is 7.76 x 10^{-5}, and in scotch bonnet from NS is 4.46 x 10^{-5}, while, heptachlor residue in scotch bonnet sample from AS is 2.1 x 10^{-3} and NS is 2.1 x 10^{-3} and heptachlor epoxide residue in scotch bonnet from AS is 2-3 x 10^{-4} and NS 2.9 x 10^{-4}. ILCR value for p, p'-DDE residue in green amaranth from AG is 1.34 x 10^{-5} and 8.84 x 10^{-5} in scotch bonnet from SS, while transchlordane residue in scotch bonnet from SS is 2.19 x 10^{-5}. Computed cancer risk value between 1 x 10^{-6} and to 1 x 10^{-4} is considered safe by USEPA (US EPA, 2014). Heptachlor residue in scotch bonnet sample from AS (2.1 x 10^{-3}) and NS (2.1 x 10^{-3}) and heptachlor epoxide residue in scotch bonnet from AS (2.3 x 10^{-3}) and NS (2.9 x 10^{-3}) had ILCR values greater than the safe value recommended by US EPA, hence, consumption of these scotch bonnet may cause cancer.

4. Conclusion

The results of the study show that the pesticide residues were below detectable limit in most of the vegetables. The pesticide residues were detected in few vegetables. The detected pesticide residues are likely due to the persistent nature of OC pesticides in the environment. Some of the detected pesticide residues were in mean concentration above their maximum residue limit (MRL). The EDI of the OC pesticide residues are below their ADI, except for heptachlor residue in scotch bonnet from two sources. Most of the vegetables may not cause adverse, chronic health issues and cancer. There is need for constant monitoring of the level of pesticide residues in food crops consumed in Nigeria by relevant authorities to safeguard public health, and educate the farmers to use the recommended concentrations of pesticides for pest control.

Supporting Information

The Supporting Information is available on the Journal of the Kenya Chemical Society webpage at https://kenyachemicalsociety.org/journals

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