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Organochlorine Pesticide Residues in Soil and its Potential Health Risk Assessment in Vegetables from Iyana-Iba Farm Settlement, Lagos State, Nigeria

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ABSTRACT

Organochlorine pesticides (OCP) have been banned locally and internationally as a result of its persistence in the environment and their potential health hazard. The study determined the concentrations of OCP residues in soil and assessed potential health risk in vegetables from a Nigerian farm settlement. Twenty four (24) soil samples were collected at top-soil and sub-soil depths, while vegetable samples of Jute mallow (Corchorus olitorious) and Smooth pigweed (Amaranthus hybridus) were collected at 7 and 21 days after pesticide application. Extracts from the soil and vegetable samples were cleaned up and analyzed for some OCP. The hazard risk index was calculated using guidelines recommended by the United States Environmental Protection Agency (USEPA). Data collected were analyzed using descriptive and inferential statistics. Results revealed that some OCP residues were detected from the soil and vegetable samples. Hazard risk index < 1 in the vegetables showed that their consumption poses no threat to human health. The detected Endosulfan 2 concentration was high, thus a long-term consumption of Jute mallow and Smooth pigweed from the farm settlement could present a public health concern. It was recommended that compliance with the ban of organochlorine pesticide need to be enforced through public enlightenment, campaign and compliance monitoring among farmers.

1. INTRODUCTION

Chemicals such as fertilizers and pesticides have been used in farming to tackle the problem of pests and diseases in vegetable production because the use of agrochemicals have been found to be the immediate and cheaper way to produce unblemished vegetables, and also increased farm productivity (Jipanin *et al*., 2001).

Thus, there has been a global increase in the use of fertilizers and pesticides in order to improve yields and quality of farm produce in general (Ranu, 2015). However, this practice has created numerous problems such as environmental pollution, harm to human health, and also compromising the sustainability of agricultural system (IAEA, 2001; Arjmandi *et al*., 2009; Chauhan *et al.,* 2013). It has been estimated that about 2 million tons of pesticides are used worldwide on an annual basis, which has resulted in the poisoning of about 3 million people and 200,000 deaths yearly (Al-Hatim *et al.*, 2015). Organochlorine pesticides comprises of a class of non-polar toxic chemicals containing carbon, hydrogen and chlorine. They are composed of five broad groups, namely: the dichlorodiphenyl-trichloro ethane (DDT) and analogues (e.g., diclofor, methoxychlor), the hexachlorocyclohexane or benzene hexachloride and their isomers (e.g., lindane), the cyclodienes (e.g., chlordane, heptachlor, aldrin, dieldrin, endrin, endosulfan, isobenzene), the chlordecones (e.g., kelevan, mirex), and the toxaphenes (Ibigbami *et al.*, 2015). They are known to resist biodegradation; therefore, they can be recycled through food chains and produce significant magnification of their original concentration at the end of the chain. Organochlorine use in Nigeria has been on the increase after it was introduced in the early 1950s and particularly in 1957 when Lindane was introduced and recommended for use due to the incidence of diseases and pest of cocoa in the country (Adegbola *et al.*, 2012).

Vegetables are one of the most common daily food intakes in Nigeria; they are also one of the agricultural products exported to other countries, and therefore serve as an important source of revenue for the country (The Nation Newspaper, 2017). Vegetables play important roles in human nutrition and health. They contain substances that regulate or stimulate digestion, act as laxatives or diuretics, and phenolic compounds which play a part in regulating the pH of intestines (Hossain *et al.*,2015). In general, consumers demand quality vegetables that are succulent, fresh-looking, and with no visible rashes or holes caused by pests or diseases. Pesticides are therefore used in vegetable production to prevent, destroy or control pests (El-Wakeil *et al.*, 2013).

About 15,000 metric tons of pesticides are imported into Nigeria annually; this exposes the environment and consequently the individuals to about 135 pesticide active ingredients, which can be implicated in several health challenges including neuro-cardiovascular diseases, cancer, birth defects, morbidity, etcetera. Pesticide residues cause direct health risks when present in food substances, while those in soils can be subjected to erosion and leaching, leading to the contamination of water in wells, rivers and ponds (Chauhan *et al.*, 2013). The occurrence of agrochemical residues in foodstuffs, crops, vegetables, milk and milk products have been reported in many countries. The presence of organophosphorus pesticide residues (diclorvos, diazinon, chlorpiryfos and fenithrothion) have been observed in the leaf of tomato in very high concentrations and root of spinach in low concentrations in Borno state, Nigeria (Akan *et al.*, 2013). Williams and Unyimadu (2013) also reported organochlorine residues in aquatic animals and rivers in Lagos state, Nigeria. Research works conducted in Ghana and internationally also point to the presence of pesticide residues in a number of food items such as strawberries, onions, cucumber, lettuce, cabbage, okra, pepper, tomatoes, beans, oranges and lemons (Hanson *et al.*, 2007).

Iyana-Iba farm settlement, locally known as 'oko-efo' (vegetable farm) has been in existence for over 15 years. It is situated in Ojo local government, Lagos state, Nigeria (Adesalu and Olugbemi, 2015). Some of the vegetables grown on this farm include plumed cockscomb (*Celosia argentea)*, potatoes *(Solanum tuberosum)*, African basil (*Occimum gratissimum)*, lettuce (*Lactuca sativa)*, waterleaf (*Talinum triangulare*), jute mallow (*Corchorus olitorius*), smooth pigweed (*Amaranthus hybridus*) and fluted pumpkin (*Telfaria occidentalis*). Aside local consumption, many of these vegetables are exported to other countries, thereby serving as means of income to many rural and urban dwellers in the country (Ibeawuchi *et al.*, 2015). The problem of pesticide residue in some Nigerian food products was brought to the fore when the European Union banned the imports of several agricultural produce from Nigeria in 2015 due to the intolerable level of pesticide residue in the products. Therefore, this study determined the concentrations of OCP residues in soil and assessed potential health risk in vegetables from a Nigerian farm settlement.

2. MATERIALS AND METHODS

2.1. Study Area

This study was conducted at a farm settlement in Iyana-Iba, Ojo local government, Lagos state, Southwest Nigeria.

2.2. Collection of Vegetables and Soil Samples

The farmland was divided into three portions (uphill, hill wash and valley bottom). At each sampling sites, vegetable samples (jute mallow and smooth pigweed) were collected at each portion of the farmland. Composite soil samples were obtained from bulking soil samples at 5 random points from each portion (uphill, hillwash and valley bottom) at depths of 0-15 cm (top soil) and 15-30 cm (sub soil). The soil and vegetable samples were collected at 7 and 21 days after pesticide application (at two stages of harvesting), labeled and transported to a laboratory.

2.3. Extraction of Vegetable Samples

Five grams of each vegetable sample was homogenized using mortar and pestle. The homogenates were then mixed with dichloromethane: hexane (1:1 v/v, 25 ml) in a Soxhlet extractor for 16 hours. The homogenates were wrapped in a filter paper before putting them in the Soxhlet extractor. The extracts were then air dried to reduce volume to concentrated form as reported by Nsikak and Aruwajoye, (2011).

2.4. Extraction of Soil Samples

The cold solvent extraction method was used for extraction of soil samples as described by William *et al.* (2013) with slight modifications. Five grams of each soil sample was weighed and 5 g of sodium sulphate was added to remove any traces of water while 25 ml of dichoromethane:hexane (1:1 v/v) was added and shaken rigorously at 270 rpm for 50 minutes. The mixture was then allowed to stand for 30 minutes and then filtered using a thick cotton wool and funnel. The extracts were then concentrated to dryness by air drying (William *et al.*, 2013).

2.5. Extracts Clean-up

The soil and vegetable extracts were cleaned up as reported by Akan *et al.* (2014) with modifications by packing the 10 mm chromatographic column with cotton wool and dried silica gel. Sodium sulphate was added to the top of the chromatographic column to trap any moisture. The evaporated samples were dissolved in iso-octane and poured into the chromatographic column. Twenty millilitres of n-hexane and DCM (ratio of 4:1) was used to elute twice and then evaporated to dryness.

2.6. Gas Chromatographic Analysis

One micro litre each of the cleaned- up samples was injected in turns into gas chromatography model Agilent 7890A equipped with electron capture detector (ECD). The carrier gas helium flow rate was 2.0 ml/min and nitrogen was used as the makeup gas. The temperature of injector was held at 250 °C, oven temperature was at 150 °C and electron capture detector was set at 290 ^oC respectively. The oven temperature was programmed at 150 ^oC and increased to 280 ^oC at 6 ^oC /min. The total run time was 21.667 minutes. The column type was DB 17 (30 m X 250 um and 0.25 um). The GC result was in ng/ul. The result in ng is the amount of analyte(s) or congener(s) of interest present in the quantity of sample taken for extraction. Organochlorine pesticide (OCP) residue was then calculated using Equations 1 and 2.

$$\text{OCP residue } \left(\text{ng / g or } \mu\text{g / kg}\right) = \frac{\text{GC result of analyte from chromatogram} \times \text{dilution factor}}{\text{Weight of sample taken for extraction}} \quad (1)$$

$$\text{Dilution factor} = \frac{\text{Final volume of cleaned up}}{\text{Initial volume of extract}} \quad (2)$$

2.7. Data Analysis

Data were analyzed using descriptive (mean and standard deviation) and inferential (ANOVA) statistics. Means were separated using Duncan multiple range test (DMRT).

2.8. Health Risk Assessment

Health risk estimations were done based on the pesticide residues detected in vegetables. Hazard risk indices of the pesticide residues via dietary intake of vegetables were assessed according to the guidelines recommended by the United State Environmental Protection Agency, where the estimated daily intake (EDI) was divided by the acceptable daily intake (ADI) (Hart *et al.,* 2005).

$$Hazard\ Risk\ Index\ (HRI) = \frac{EDI}{ADI} \quad (3)$$

The estimated daily intake was calculated as reported by Raisa *et al.* (2015).

$$EDI = C \times \frac{F}{W} \quad (4)$$

Where C is the sum of the concentration of pesticide residues in each vegetable (µg/kg), F is the mean daily intake of food per person.

The average daily vegetable intake was considered to be 0.345 kg/person/day according to the previous reports (Hart *et al.*, 2005; WHO, 2012; Hossain *et al.*, 2015; Ogunkunle *et al.*, 2017). W is the mean body weight for adults (60 kg) and 30 kg for children between 1-11 years according to previous report (Ibigbami *et al.*, 2015).

3. RESULTS AND DISCUSSION

Tables 1 and 2 show the concentration (mean ± standard deviation) of the OCP residue analyzed in jute mallow and smooth pigweed samples collected 7 and 21 days after pesticide application (DAPA) respectively. A total of 17 OCPs were analyzed. The residues in jute mallow ranged from below detection limit (BDL) to 3.18±2.94 µg/kg and a total of 3.46±3.15 µg/kg and 0.44±0.57 µg/kg were detected 7 and 21 days after pesticide application respectively. The result for the organochlorine residues in smooth pigweed ranged from BDL to 1.75±1.59 µg/kg and a total of 2.02±1.87 µg/kg and 0.73±0.94 µg/kg were detected 7 and 21 days after pesticide application respectively. Some of the pesticides detected in the vegetables such as heptachlor, DDT, DDE, and endosulfan are known to have endocrine and estrogenic disrupting properties (Okoya *et al.*, 2013). Cyclodiene subgroup to which Endosulfan I and II belong had the highest value of 1.94 µg/kg and 0.67 µg/kg in smooth pigweed after 7 and 21 days of pesticide application respectively when compared to other groups. Hexachlorohexane (HCH) to which lindane belongs had the lowest value of 0.029 µg/kg and 0.026 µg/kg both 7 and 21 days respectively among all the three groups. Dichlorodiphenyltrichloroethane (DDT) subgroup had a total of 0.048 µg/kg and 0.038 µg/kg 7 and 21 days respectively among all the three subgroups. There was a decrease in the organochlorine pesticide residue in both jute mallow and smooth pigweed samples after 21 days of organochlorine pesticide application when compared to the 7 days after OCP application. This is because pesticide residues diminish measurably with time, though the speed of reduction may differ in different compounds (Frehse and Walter, 2006). A decrease in the organochlorine pesticide residue was also observed in smooth pigweed when compared to jute mallow.

Table 1: Organochlorine pesticides residues in jute mallow collected at 7 and 21 days after pesticide application

Organochlorine	7 DAPA (µg/kg)	21 DAPA (µg/kg)
α-HCH	BDL	BDL
β-HCH	0.014±0.015[a]	BDL
γ-HCH	0.005±0.0042[a]	0.0057±0.0049[a]
δ-HCH	0.005±0.0042[a]	0.0037±0.0058[a]
Aldrin	0.0087±0.010[a]	0.009±0.0030[a]
Heptachlor	0.012±0.0017[a]	0.015±0.00058[a]
Heptachlor epoxide	0.014±0.0065[a]	0.008±0[a]
Dieldrin	0.022±0.020[a]	BDL
Endrin	0.0005±0.00049[a]	BDL
Endrin aldehyde	0.08±0.072[ab]	0.15±0.26[a]
Endosulfan I	0.0077±0.0068[a]	0.0093±0.0081[a]
Endosulfan II	3.18±2.94[a]	0.48±0.42[a]
Endosulfan sulfate	0.05±0.087[a]	BDL
DDT	0.017±0.015[a]	0.O12±0.0093 [a]
DDE	0.005±0.0010[a]	0.004±0[a]
DDD	0.002±0.0035[a]	BDL
Methoxychlor	BDL	BDL
Total	3.46±3.15	0.44±0.57

Values are means ± Standard deviation; Values in the same column followed by superscript 'a' are not significantly ($p > 0.05$) different.

Table 2: Organochlorine pesticide residues in smooth pigweed collected at 7 and 21 days after organochlorine pesticide application

Organochlorine	7 DAPA (µg/kg)	21 DAPA (µg/kg)
α-HCH	BDL	BDL
β-HCH	0.0067±0.012[a]	0.010±0.017[a]
γ-HCH	0.0047±0.0040[a]	0.007±0.003[a]
δ-HCH	0.018±0.014[a]	0.0093±0.0093[a]
Aldrin	0.012±0.0029[a]	0.009±0.0017[a]
Heptachlor	0.086±0.12[a]	0.01±0.002[a]
Heptachlor epoxide	0.0093±0.010[a]	0.008±0.0036[a]
Dieldrin	0.02±0.035[a]	BDL
Endrin	BDL	0.03±0.0052[a]
Endrin aldehyde	0.09±0.10[ab]	0.017±0.029[b]
Endosulfan I	0.0043±0.0075[a]	0.01±0.0057[a]
Endosulfan II	1.75±1.59[a]	0.27±0.47[ab]
Endosulfan sulfate	BDL	0.14±0.23[ab]
DDT	0.018±0.0029[a]	0.017±0.021[a]
DDE	0.03±0.0026[a]	0.0037±0.0035[a]
DDD	BDL	BDL
Methoxychlor	BDL	0.017±0.029[a]
Total	2.02±1.87	0.73±0.9

The high organochlorine content in jute mallow could be attributed to the foliage system and high fat content nature of the plant since organochlorine compounds are lipophilic in nature, hence, vegetables having less water and greater lipid contents have greater potential to accumulate organochlorine pesticides (Akinloye *et al.*, 2011; Akan *et al.*, 2014). The means of the organochlorine pesticide residue in this study are lower than those reported by Akan *et al.* (2014) in the roots, stem and leaves of spinach, lettuce, cabbage, tomato, and onion harvested from the Alau Dam and Gongulong agricultural sites, Borno state, Nigeria. The values of the organochlorine pesticide residues were also lower than those detected by Ozcan, (2016) in cherry tomato, banana pepper, lettuce, purslane, green beans, cucumber and onions from Kirklareli, Turkey.

The OCP residues in soil ranged from BDL to 3.46±3.38 µg/kg and a total of 3.66±3.47 µg/kg organochlorine residues were detected in the top soil and 1.83±1.56 µg/kg in the sub soil 7 DAPA as shown in Tables 3 and 4. The OCP residues ranged from BDL to 3.46±3.38 µg/kg and a total of 3.66±3.47 µg/kg organochlorine residues were detected in the top soil and 1.83±1.56 µg/kg in the sub soil 7 DAPA. OCP residue analyzed in the soil samples collected 21 DAPA ranged from BDL to 3.41±0.45 µg/kg and a total of 3.61±0.55 µg/kg OCP in the top soil and 2.04±0.54 µg/kg in the sub soil. There was a decrease in the lower depth of the soil (sub soil) when compared to the upper depth (top soil) of the soil samples 7 and 21 days after OCP application. The values of the organochlorine residue were higher in the top soil when compared to the sub soil. However, there was no difference in the values of the organochlorine residue between the soil samples collected 7 days after and those collected 21 days after OCP application. The values of the organochlorine residue in soil were lower when compared to those detected by Akan *et al.* (2014) in Borno state, Nigeria. However, the values of the organochlorine residue in soil were higher than those detected by Owusu-Boateng and Amuzu, (2013) in farms along river Oyansia, Accra, Ghana. This shows that though organochlorine pesticides are still in use, the quantities applied is not much when compared to that of Borno state and also the persistent nature of organochlorine pesticides could also be responsible for the higher values detected in Borno and Turkey.

The results in Table 5 showed that the organochlorine pesticide (OCP) residues detected in jute mallow and smooth pigweed posed no threat to the consumer's health if consumed, all the 17 organochlorine pesticides detected were lower than available daily intake when compared on the table, none has a value equal to or higher than the acceptable daily intake limit stipulated by World Health Organization and Food and Agriculture Organization of the United Nations (WHO, 2012). All hazard risk indices (HRI) of each of the organochlorine was less than 1 making it safe for human health because an index more than 1 is considered as not safe for human health as reported by Hossain *et al.* (2015) and Raisa *et al.* (2015).

Table 3: Organochlorine pesticide residues in soil samples collected 7 days after organochlorine pesticide application

Organochlorine	Top soil (µg/kg)	Sub soil (µg/kg)
α-HCH	BDL	BDL
β-HCH	BDL	BDL
γ-HCH	0.0072±0.00029[a]	0.0055±0.0047[a]
δ-HCH	0.022±0.022[a]	0.013±0.012[ab]
Aldrin	0.008±0[a]	0.0069±0.00012[a]
Heptachlor	0.13±0.036[b]	0.01±0[b]
Heptachlor epoxide	0.0054±0.00035[a]	0.006±0[a]
Dieldrin	BDL	BDL
Endrin	BDL	BDL
Endrin aldehyde	0.39±0.52[ab]	0.046±0.04[a]
Endosulfan I	0.008±0.007[a]	BDL
Endosulfan II	3.46±3.38[ab]	1.73±1.50[ab]
Endosulfan sulfate	BDL	BDL
DDT	0.026±0.0091[a]	0.007±0.00006[a]
DDE	0.023±0.024[a]	0.004±0[a]
DDD	BDL	BDL
Methoxychlor	BDL	BDL
Total	3.66±3.47	1.83±1.56

Table 4: Organochlorine pesticide residues in soil samples collected 21 days after organochlorine pesticide application

Organochlorine pesticide	Top soil (µg/kg)	Sub soil (µg/kg)
α-HCH	BDL	BDL
β-HCH	0.0067±0.012[a]	BDL
γ-HCH	0.012±0.002[ab]	0.0047±0.0040[a]
δ-HCH	0.014±0.0015[ab]	0.003±0.0026[a]
Aldrin	0.012±0.0085[ab]	0.011±0.0015[a]
Heptachlor	0.028±0.0029[b]	0.018±0.0064[b]
Heptachlor epoxide	0.0023±0.0040[a]	0.0064±0.0026[a]
Dieldrin	BDL	BDL
Endrin	BDL	BDL
Endrin aldehyde	0.06±0.05[ab]	0.015±0.022[ab]
Endosulfan I	0.011±0.01[ab]	0.013±0.011[ab]
Endosulfan II	3.41±0.45[ab]	1.92±0.55[ab]
Endosulfan sulfate	BDL	BDL
DDT	0.17±0.12[ab]	0.013±0.0058[ab]
DDE	0.0017±0.0029[a]	0.0023±0.0021[a]
DDD	BDL	BDL
Methoxychlor	BDL	BDL
Total	3.61±0.55	2.04±0.54

Table 5: Estimated hazard indices of organochlorine pesticide exposure in vegetable samples

OCPs	EDI (µg/kg/day)		ADI (µg/kg/day)	Hazard indices	
	1-11 yrs	Adult		1-11 yrs	Adult
α-HCH	BDL	BDL	0.50	BDL	BDL
β-HCH	0.0002	0.00008	0.50	0.0004	0.0002
γ-HCH	0.00007	0.00003	0.50	0.0001	0.00006
δ-HCH	0.0001	0.00005	0.50	0.0002	0.0001
Aldrin	0.0001	0.00007	0.10	0.001	0.0007
Heptachlor	0.0002	0.0001	0.10	0.002	0.001
Heptachlor	0.002	0.00008	0.10	0.02	0.0008
Dieldrin	0.0003	0.0001	0.10	0.003	0.01
Endrin	0.00007	0.00003	0.20	0.0004	0.0002
Endrin aldehyde	0.0009	0.0005	0.20	0.005	0.003
Endosulfan I	0.00009	0.00005	0.05	0.002	0.001
Endosulfan II	0.04	0.02	0.05	0.8	0.4
Endosulfan	0.0006	0.003	0.05	0.01	0.006
DDT	0.0003	0.0001	0.50	0.0006	0.0002
DDE	0.00006	0.00003	0.50	0.0001	0.0001
DDD	0.00002	0.00001	0.50	0.00004	0.00002
Methoxychlor	BDL	BDL	0.01	BDL	BDL

4. CONCLUSION

The results of this work revealed the following: The detection of the residual levels of the organochlorine pesticide shows that organochlorine pesticides are still being used in the country even though some of it has been banned. Consumption of the vegetables however pose no threat to human health, but its consumption over a long period of time and in other crops could not be ascertained as not posing health threat to human since organochlorine is a persistent organic pollutant and DDT and its metabolite have been classified as human carcinogens. Also, OCP residues were discovered to decrease with time in the vegetables, so the longer the harvesting time after pesticide application, the lower the organochlorine pesticide residue found in the vegetables. Organochlorine pesticides are more persistent and stay longer in soil and decompose slowly. The soil act as a pathway for the transport of pesticides to contaminate the ground/surface water, plants and river through leaching and erosion. This can further contaminate fishes and humans exposed to the river. Based on the results of this study, government should enforce laws and strict compliance to the eradication of organochlorine pesticides and also enforce routine monitoring of pesticide residue in vegetables to prevent environmental pollution and consumption of pesticide residues that could pose threat to human health.

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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