ASSESSMENT OF ORGANOCHLORINE PESTICIDE RESIDUE LEVELS IN VEGETABLES SOLD IN FEDERAL CAPITAL TERRITORY (FCT), NIGERIA

ABSTRACT

The study evaluated the organochlorine pesticide residue levels in vegetables sold in Federal Capital Territory, Abuja. Six samples were bought from eleven major markets across the six Area Councils of FCT, Abuja. The samples were mixed to form composite groups of the fruits and vegetables, and prepared using QuEChERS method. It was analysed using Agilent 7890 Gas Chromatography equipped with a micro-cell Electron Capture Detector (μECD). The analysis revealed that the hazard index (HI) for all the fruits and vegetables studied were well below the levels of adverse health effect for chronic exposure except for Onion in children which was 1.2287. From the hazard index and cancer risk analysis, Aldrin and Dieldrin have been implicated as the pesticides of concern since they were present in reasonable concentrations for almost all the samples and age groups studied. The highest potential of cancer risk was found in Onions for children. Also, the total cancer risk implicated Onions and Pepper as the most contaminated foodstuffs with pesticide residues in the studied area. The order of the total cancer risk from the study was Onions > Pepper > Tomato > Green Amaranth Leaves > Fluted Pumpkin Leaves > Okra for both adult and children. Therefore, periodic monitoring of pesticides residues in these fruits and vegetables cannot be over emphasized, but will go a long way to prevent, control and reduce environmental pollution and health risks. Also, taking precautionary measures before consumption of these foodstuffs is advised.

Keywords: Pesticide, Pesticide residue, Organochlorine, QuEChERS method, Maximum residue limit/level, Hazard quotient/index/indices, Cancer risk

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INTRODUCTION

Fruits and vegetables are commonly grown with pesticides to prevent and control pest infestation. Preventing and controlling pest infestation in the farm leads to higher yield [1]. Pesticides are also used to preserve or transport harvested crops during storage and handling. Therefore, contamination with pesticides may occur at any point from farm to fork [2,14]. The remains or residues of these pesticides in food become an analytical concern and an important threat to life because some are highly persistent to environmental degradation. Pesticide exposure may also lead to wide array of health problems such as cancer, birth defect, neural and kidney damage, immune suppression, diminished intelligence, hormone disruption, reproductive abnormalities, congenital disabilities, etc. [3]. Therefore, this necessitates the identification and quantification of pesticide residues in agricultural products such as fruits and vegetables. It is a very important concern to both developed and developing countries. For example, in 2015 European Union (EU) issued a ban on the importation of beans from Nigeria into Europe due to their high residue level (0.03mg/kg to 4.6mg/kg) of Dichlorvos [4]. This study will provide experimental data, dietary survey and statistical analysis on some pesticides residues in selected fruits and vegetables which will help enhance better regulation of pesticide use in Nigeria.

METERIALS AND METHODS

Study Area

The experimental samples were bought from eleven major markets in FCT, *viz.* Abaji, Bwari, Dutse-Alhaji, Garki, Gwagwalada, Kuje, Kwali, Utako, Nyanya, Wuse, and Zuba, while the control samples which were grown without pesticides spray were collected from a personal farm in Garki. The various markets were drawn from all the six area councils of the FCT. Each market was divided into four quadrants and the same cost of samples were bought from each quadrant. The samples were sorted and mixed to form composite samples of each type i.e tomatoes from one market were mixed with those from another market. Thus, six composite samples were formed representing the markets in FCT, *viz.* Tomato (Solanum lycopersicum), Pepper (Capsicum spp), Okra (Abelmoschus esculentus), Onion (Allium cepa), Fluted Pumpkin Leaves (Telfairia occidentalis) and Green Amaranth Leaves (Amaranthus spp).

FCT is the capital city of Nigeria, which is located between latitude of 8.25^{o} N – 9.20^{o} N, and longitude of 6.45^{o}E – 7.39^{o} E. It is located north of the confluence of the Niger River and Benue River. It was created in 1976 and made the Federal Capital Territory in 1991. FCT is bordered by Niger, Kaduna, Nasarawa and Kogi states.

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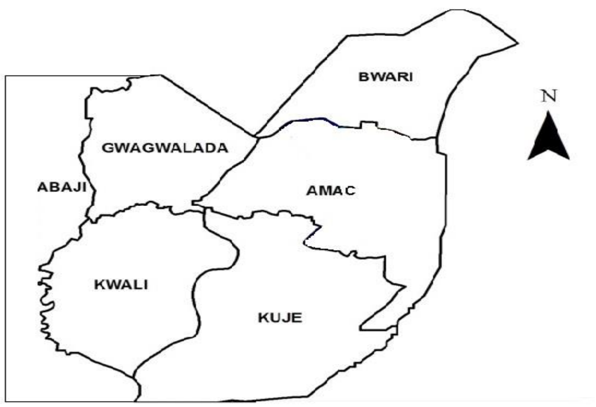

Figure 1: Map of FCT Showing the Area Councils

Sample Pre-Treatment and Preparation

Dirt and particles were removed, and they were washed and rinsed with running water. The composite samples were grinded separately using dry ice in Stephan Electronic 2010 Blender to control temperature and spill of the liquid content (cryogenic milling). This process is used to increase homogeneity of the samples or to reduce the sub-sampling variation and to enhance the extraction of analytes. The grinded samples were packed separately in a clean disposable closed plastic plates and stored in the fridge at about 4°C until required for extraction.

Sample Extraction

10 g of each sample was weighed into different 50mL centrifuge tubes and labelled accordingly. 10 mL of acetonitrile was added to each, followed by 0.1 mL of 50 ppm PF-38 and the mixture homogenized with a voltex mixer. Extraction salt were added to each and the mixture homogenized again with a voltex mixer. They were then centrifuged at 3000 rpm at 0°C for 5 minutes and 6 mL of the upper layers were decanted into different 14 mL centrifuge tubes and labelled accordingly. Clean up salt were added to each and the mixture homogenized with a voltex mixer. They were then centrifuged again at 3000 rpm at 0°C for 5 minutes and the supernatants decanted into different test tubes and labelled accordingly.

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50% Formic Acid was added for each as buffer solution at the rate of 10 µL per mL followed by 0.125 mL of 20 ppm Triphenylphosphate (TPP) as an internal standard and the mixture homogenized. 1 mL of each solution was transferred into different test tubes and labelled accordingly. It was then placed into the Turbo Vap LV for drying. After drying, each test tube was reconstituted or made up to the volume with n-hexane:acetone (4:1, v/v). The mixture was then homogenized and transferred to vials and to the GCMS for instrumentation.

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Health Risk

This involves non-carcinogenic risk and carcinogenic risk assessment. The non-carcinogenic risk assessment involved the calculation of the estimated daily dose, hazard quotient and hazard indices of the different vegetables for adult and children. The estimated daily doses for the vegetables for adult and children were calculated using the formula in Equation 3 while the hazard quotients (HQ) was calculated using Equation 4. The hazard index (HI) of the vegetables were calculated as sum of the hazard quotients for the various pesticides identified and quantified for each foodstuff and cancer risks were calculated using Equation 5.

$$EDI = \frac{C_i \, . FCR \, . F \, . ED}{B_W \, . AT} \quad (1)$$

For daily exposure to these pesticides the Exposure Factor (EF) is equal to 1

$$EF = \frac{(F \, . \; ED)}{AT} = 1 \quad (2)$$

Thus,

$$Adjusted\ EDI = \frac{C_p . FCR}{B_W} \quad (3)$$

Where;

EDI = Estimated Daily Intake (in mg/kg-day)

C_i = Concentration of Pesticide Residues (in mg/kg, from the experimental result)

FCR = Food Consumption Rate (in kg/day) (see Appendix 1)

F = Exposure Frequency (in days/year)

ED = Exposure Duration (in years)

B_w = Body Weight (60 kg for adult and 15 kg for children)

AT = Average Time (in days)

$$HQ = \frac{EDI_i}{RfD_i} \quad (4)$$

Where;

RfD_i = Reference Dose (in mg/kg-day)

$$Cancer\ Risk = C_i . \frac{FCR_i . F_i . ED_i . CF}{B_W . AT} . SF_i \quad (5)$$

Where;

CF = Conversion Factor as 1.0 x 10^{-6} (in kg/mg)

SF_i = Oral Slope Factor (in mg^{-1}kg-day)

Food Consumption Rate (FCR) is calculated from a survey data recovered from the study area while Estimated Daily Intake (EDI), Hazard Quotient (HQ) and Cancer Risk were calculated with reference to the standard assumption of adults and children having an average body weights of 60 kg and 15 kg respectively. Cancer risk of 1.0 x 10^{-6} implies that 1 in 1,000,000 persons has the lifetime risk of developing cancer through the pathway (of ingestion only). The maximum risk value or threshold is 1.0 x 10^{-6} but values greater than that is unacceptable.

RESULTS AND DISCUSSION

Results obtained from the study are shown on Tables 1-5. The percentage recovery (R%) values of the analytes in the spiked samples were in the range of 83-118% for Tomato and 89-117% for Fluted Pumpkin Leaves. The blank determination recorded no peak at all.

Table 1: Analytical Result of samples under review

Commented [IA10]: Organochlorine pesticide residue found in 6 different vegetable and fruit collected from FCT

S/N	PESTICIDES	Tomato (mg/kg)	Pepper (mg/kg)	Okra (mg/kg)	Onion (mg/kg)	Fluted Pumpkin (mg/kg)	Green Amaranthus (mg/kg)
1	Aldrin	0.0140	0.0310	0.0095	0.0439	0.0019	0.0140
2	a-BHC	0.0064	0.0161	0.0025	0.0368	0.0802	0.0227
3	b-BHC	0.0164	0.0475	0.0000	0.0147	0.0003	0.0017
4	d-BHC	0.0157	0.0084	0.0070	0.0660	-	0.0075
5	Chlorothalonil	0.0097	0.0000	0.0000	0.0334	0.0000	0.0000
6	o, p' - DDE	-	0.0105	0.0091	0.0276	-	0.0504
7	p, p' - DDE	0.0014	0.0440	-	0.0096	0.0087	-
8	Dieldrin	0.0043	0.0401	-	0.0292	0.0000	0.0025
9	Endosulfan I	0.0002	0.0799	0.0002	0.0292	0.0043	0.0026
10	Endosulfan II	-	0.0332	-	0.0129	-	0.0082
11	Endrin	-	0.0072	-	0.0858	-	-
12	Heptachlor	0.0164	0.0842	0.0254	0.0596	0.0221	0.0430
13	Heptachlor Epoxide	0.0009	0.0058	-	0.0346	-	0.0311
14	Lambda Cyhalothrin	-	0.0823	-	0.0287	-	-
15	Lindane	0.0018	0.0066	0.0037	0.0256	0.0008	0.0037
16	PCB-153	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Table 2: CODEX MRLs of Pesticide Residues under review

S/N	PESTICIDES	Codex MRL (mg/kg)	Codex MRL (mg/kg)	Codex MRL (mg/kg)	Codex MRL (mg/kg)	Codex MRL (mg/kg)	Codex MRL (mg/kg)
1	Aldrin	0.10	0.10	0.10	0.05	0.05	0.05
2	a-BHC	2.00	1.00	1.00	1.00	2.00	2.00
3	b-BHC	2.00	1.00	1.00	1.00	2.00	2.00
4	d-BHC	2.00	1.00	1.00	1.00	2.00	2.00
5	Chlorothalonil	5.00	7.00	-	1.50	-	-
6	o, p' - DDE	0.05	0.05	0.05	0.01	0.05	0.05
7	p, p' - DDE	0.05	0.05	0.05	0.01	0.05	0.05
8	Dieldrin	0.10	0.10	0.10	0.05	0.05	0.05
9	Endosulfan I	0.50	0.50	0.50	0.20	2.00	2.00
10	Endosulfan II	0.50	0.50	0.50	0.20	2.00	2.00
11	Endrin	0.05	0.05	0.05	0.01	0.01	0.01
12	Heptachlor	0.05	0.05	0.05	0.05	0.05	0.05

13	Heptachlor Epoxide	0.05	0.05	0.05	0.05	0.05	0.05
14	Lambda Cyhalothrin	0.30	0.30	0.20	0.20	0.30	0.30
15	Lindane	2.00	1.00	1.00	1.00	2.00	2.00
16	PCB-153	0.05	0.05	-	-	-	-

From Table 1 and 2, a total of twelve organochlorine pesticides were detected in tomatoes while o, p' – DDE, Endosulfan II, Endrin, and Lambda Cyhalothrin were below detection limit and none of them exceeded the Codex MRL. These represents the organochlorine pesticides the farmers used in cultivation of these crops. For pepper, almost all the sixteen organochlorine pesticides studied were detected and all the analytes were below the Codex MRL except for Heptachlor (168.4%). For Okra, seven organochlorine pesticides were detected and all were below the Codex MRL. For Onions, all the sixteen organochlorine pesticides were detected and three were above the Codex MRL, *viz.* o, p'–DDE (276.0%), Endrin (858.0%) and Heptachlor (119.2%) and required urgent attention and control. For Fruited Pumpkin Leaves, a total of seven organochlorine pesticides were detected while eleven were detected in Green Amaranth Leaves. None of the analytes were above the Codex MRL except for o, p'-DDE in Green Amaranth Leaves which had 100.8% and calls for concern and further monitoring.

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The result of this study revealed that of all the fruits and vegetables investigated, only Pepper, Onions, and Green Amaranth Leaves reported the presence of pesticide residues above Codex MRL. It is pertinent to note that this result agrees with Njoku *et al.*, 2017 [5] that reported the presence of Aldrin in all the vegetables purchased from six markets in Lagos state. He also reported that some of the pesticide residues were above the maximum residue levels and can pose carcinogenic and non-carcinogenic health effects.

Table 3: Reference Dose (RfD) (mg/kg/d) and Oral Slope Factor (SF) (mg-1kgd)

S/N	PESTICIDES	RfD	SOURCE	SF	SOURCE
1	Aldrin	3.00E-05	USEPA, 1986 [6]	1.70E+01	USEPA, 2006 [9]
2	a-BHC	5.00E-04	USEPA, 1986 [6]	6.30E+00	USEPA, 2006 [9]
3	b-BHC	2.00E-04	USEPA, 1986 [6]	1.80E+00	USEPA, 2006 [9]
4	d-BHC	2.00E-04	USEPA, 1986 [6]	1.10E+00	USEPA, 2006 [9]
5	Chlorothalonil	1.50E-02	USEPA, 1986 [6]	1.70E-02	USEPA, 1986 [6]
6	o, p' - DDE	3.00E-03	USEPA, 2006 [9]	3.40E-01	USEPA, 2011 [10]
7	p, p' - DDE	3.00E-03	USEPA, 2006 [9]	3.40E-01	USEPA, 2011 [10]
8	Dieldrin	5.00E-05	USEPA, 2011 [10]	1.60E+01	USEPA, 2011 [10]
9	Endosulfan I	6.00E-03	USEPA, 1986 [6]	-	-
10	Endosulfan II	6.00E-03	USEPA, 1986 [6]	-	-
11	Endrin	3.00E-04	USEPA, 2002 [8]	-	-
12	Heptachlor	5.00E-04	USEPA, 1986 [6]	4.50E+00	USEPA, 1986 [6]
13	Heptachlor Epoxide	1.30E-05	USEPA, 1986 [6]	9.10E+00	USEPA, 1986 [6]
14	Lambda Cyhalothrin	5.00E-03	USEPA, 2002 [8]	-	-
15	Lindane	3.00E-04	USEPA, 1986 [6]	1.30E+00	USEPA, 1986 [6]
16	PCB-153	2.00E-05	USEPA, 1996 [7]	2.00E+00	USEPA, 1996 [7]

Table 4: Hazard Quotients (HQ) and Hazard Indices (HI) for samples under review

S/N	PESTICIDES	TOMATO		PEPPER		OKRA		ONION		F. PUMPKIN LEAVES		G. AMARANTH LEAVES	
		Adult	Children	Adult	Children	Adult	Children	Adult	Children	Adult	Children	Adult	Children
1	Aldrin	0.0226	0.0902	0.0310	0.1239	0.0087	0.0350	0.0788	0.3151	0.0016	0.0066	0.0085	0.0338
2	a-BHC	0.0006	0.0025	0.0010	0.0039	0.0001	0.0005	0.0040	0.0159	0.0041	0.0165	0.0008	0.0033
3	b-BHC	0.0040	0.0159	0.0071	0.0285	0.0000	0.0000	0.0040	0.0158	0.0000	0.0002	0.0002	0.0006
4	d-BHC	0.0038	0.0152	0.0013	0.0051	0.0010	0.0039	0.0178	0.0711	-	-	0.0007	0.0027
5	Chlorothalonil	0.0000	0.0001	0.0000	0.0000	0.0000	0.0000	0.0001	0.0005	0.0000	0.0000	0.0000	0.0000
6	o, p' – DDE	-	-	0.0001	0.0004	0.0001	0.0003	0.0005	0.0020	-	-	0.0003	0.0012
7	p, p' – DDE	0.0000	0.0001	0.0004	0.0018	-	-	0.0002	0.0007	0.0001	0.0003	-	-
8	Dieldrin	0.0042	0.0167	0.0241	0.0964	-	-	0.0314	0.1257	0.0000	0.0000	0.0009	0.0037
9	Endosulfan I	0.0000	0.0000	0.0004	0.0016	0.0000	0.0000	0.0003	0.0010	0.0000	0.0001	0.0000	0.0000
10	Endosulfan II	-	-	0.0002	0.0007	-	-	0.0001	0.0005	-	-	0.0000	0.0001
11	Endrin	-	-	0.0007	0.0029	-	-	0.0154	0.0616	-	-	-	-
12	Heptachlor	0.0016	0.0064	0.0051	0.0202	0.0014	0.0056	0.0064	0.0257	0.0011	0.0045	0.0016	0.0063
13	Heptachlor Epoxide	0.0035	0.0138	0.0134	0.0537	-	-	0.1434	0.5735	-	-	0.0434	0.1736
14	Lambda Cyhalothrin	-	-	0.0005	0.0020	-	-	0.0003	0.0012	-	-	-	-
15	Lindane	0.0003	0.0011	0.0007	0.0026	0.0003	0.0014	0.0046	0.0184	0.0001	0.0003	0.0002	0.0009
16	PCB-153	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
HAZARD INDICES		0.1121	0.4485	0.0995	0.3981	0.0120	0.0480	0.3085	1.2341	0.0135	0.0539	0.1103	0.4411

Table 5: Cancer Risk for samples under review

S/N	PESTICIDES	TOMATO		PEPPER		OKRA		ONION		F. PUMPKIN LEAVES		G. AMARANTH LEAVES	
		Adult	Child	Adult	Child	Adult	Child	Adult	Child	Adult	Child	Adult	Child
1	Aldrin	1.2E-05	4.6E-05	1.6E-05	6.3E-05	4.5E-06	1.8E-05	4.0E-05	1.6E-04	8.4E-07	3.4E-06	4.3E-06	1.7E-05
2	a-BHC	2.0E-06	7.8E-06	3.0E-06	1.2E-05	4.3E-07	1.7E-06	1.2E-05	5.0E-05	1.3E-05	5.2E-05	2.6E-06	1.0E-05
3	b-BHC	1.4E-06	5.7E-06	2.6E-06	1.0E-05	0.E+00	0.E+00	1.4E-06	5.7E-06	1.4E-08	5.6E-08	5.6E-08	2.2E-07
4	d-BHC	8.4E-07	3.3E-06	2.8E-07	1.1E-06	2.1E-07	8.5E-07	3.9E-06	1.6E-05	-	-	1.5E-07	6.0E-07
5	Chlorothalonil	8.0E-09	3.2E-08	0.E+00	0.E+00	0.E+00	0.E+00	3.1E-08	1.2E-07	0.E+00	0.E+00	0.E+00	0.E+00
6	o, p' – DDE	-	-	1.1E-07	4.3E-07	8.6E-08	3.4E-07	5.0E-07	2.0E-06	-	-	3.1E-07	1.2E-06
7	p, p' – DDE	2.3E-08	9.2E-08	4.5E-07	1.8E-06	-	-	1.8E-07	7.0E-07	7.6E-08	3.0E-07	-	-
8	Dieldrin	3.3E-06	1.3E-05	1.9E-05	7.7E-05	-	-	2.5E-05	1.0E-04	5.6E-09	2.3E-08	7.3E-07	2.9E-06
9	Endosulfan I	-	-	-	-	-	-	-	-	-	-	-	-
10	Endosulfan II	-	-	-	-	-	-	-	-	-	-	-	-
11	Endrin	-	-	-	-	-	-	-	-	-	-	-	-
12	Heptachlor	3.6E-06	1.4E-05	1.1E-05	4.5E-05	3.2E-06	1.3E-05	1.4E-05	5.8E-05	2.6E-06	1.0E-05	3.5E-06	1.4E-05
13	Heptachlor Epoxide	4.1E-07	1.6E-06	1.6E-06	6.4E-06	-	-	1.7E-05	6.8E-05	-	-	5.1E-06	2.1E-05
14	Lambda Cyhalothrin	-	-	-	-	-	-	-	-	-	-	-	-
15	Lindane	1.1E-07	4.4E-07	2.6E-07	1.0E-06	1.3E-07	5.3E-07	1.8E-06	7.2E-06	2.5E-08	1.0E-07	8.7E-08	3.5E-07
16	PCB-153	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
TOTAL CANCER RISK		2.3E-05	9.3E-05	5.5E-05	2.2E-04	8.5E-06	3.4E-05	1.2E-04	4.7E-04	1.6E-05	6.6E-05	1.7E-05	6.8E-05

From Table 4, it is observed that the Hazard Quotients for all pesticides residues detected were all satisfactory and the Hazard Index for all the vegetables studied were well below the level at which adverse health effect for chronic exposure was observed except for Onion in children, which was 1.2287. This calls for concern over prolonged use of these Onion from the area by children. The percentages of chronic reference dose for Heptachlor Epoxide and Aldrin in Onions for children were 57.35 and 31.51 respectively. It was observed that the percentage of chronic reference dose were higher for children than for adults. The result is consistent with that of Adeleye *et al.,* 2019 [11] that reported HI > 1 for Aldrin, Dieldrin and Heptachlor Epoxide in Fruited Pumpkin Leaves and Green Amaranth Leaves for adult and children. This result showed that Heptachlor Epoxide, Aldrin and Dieldrin occurred more in the fruits and vegetables studied and as such, their use required strict monitoring.

Table 5 shows that eleven organochlorine pesticides have cancer effect, namely; Aldrin, a-BHC, b-BHC, d-BHC, Chlorothalonil, o,p'–DDE, p,p'–DDE, Dieldrin, Heptachlor, Heptachlor Epoxide and Lindane. It is worthy of note that only twelve out of the sixteen organochlorine pesticides studied had oral slope factors (Table 3). The calculated cancer risk associated with consumption of these fruits and vegetables showed values well below the upper bound of 1.0×10^{-6} except for few pesticides. The highest potential of cancer risk was found in Onions for children. Also, the total cancer risk implicated Onions and Pepper as the most contaminated foodstuffs with pesticide residues in the studied area. The result is consistent with that of Adeleye *et al.,* 2019 [11] that reported Aldrin and Dieldrin to pose carcinogenic health risks to adult, while Aldrin, Dieldrin, Heptachlor and Heptachlor Epoxide to pose carcinogenic health risks to children. It is pertinent to note that Aldrin and Dieldrin are already banned due to concern about their impact on human health such as increased risk of breast cancer. Aldrin is readily converted to Dieldrin, which is one of the most persistent of all organochlorine pesticides [12]. Also, Heptachlor is readily converted to Heptachlor Epoxide and other products in the environment. Heptachlor Epoxide degrades more slowly and, as a result, it is more persistent than Heptachlor [13].

CONCLUSION

From the hazard index and cancer risk analysis, Aldrin and Dieldrin have been implicated as the pesticides of concern since they were present in reasonable concentrations for almost all the samples and groups studied. Other pesticides of interest is Heptachlor and Heptachlor Epoxide detected in Onions and Pepper for adult and children.

Generally, the study showed that children in the studied area has more cancer risk than adult. The highest potential of cancer risk was found in Onions for children. Also, the total cancer risk implicated Onions and Pepper as the most contaminated foodstuffs with pesticide residues in the studied area. The order of the total cancer risk from the study was Onions > Pepper > Tomato > Green Amaranth Leaves > Fluted Pumpkin Leaves > Okra for both adult and children. Therefore, periodic monitoring of pesticides residues in these fruits and vegetables cannot be over emphasized, but will go a long way to prevent, control and reduce environmental pollution and health risks. Also, taking precautionary measures before consumption of these foodstuffs is advised.

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