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Quantitative and Qualitative Screening of Organochlorine Pesticide Residues in Some Available Fruits Sold in Kaura Namoda Zamfara State

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Abstract - **Fruits and vegetables are generally considered essential dietary components because they provide vitamins, minerals, and fiber. Fruits are often threatened by pests during planting; therefore, farmers resort to the indiscriminate use of organochlorine pesticides. This research aims to determine the organochlorine pesticide residues in selected fruit samples (banana, guava, pineapple, lettuce, spinach, cucumber, and cabbage) obtained from Kaura Namoda, Zamfara State. QuEChERS and GC-MS were used for extraction and analysis, respectively. Twelve organochlorine standards were used, and the results show that only Endosulfan I, with an average concentration of 0.0023 mg/kg, was detected in all the samples, while alpha lindane was found in pineapple and watermelon with an average concentration of 0.0045 mg/kg. Heptachlor, aldrin, and DDE were not detected in the fruit samples. All the pesticide residues detected in the fruit samples were below the recommended maximum residue limit (MRL) of the WHO, except for Endosulfan I, which was higher than the WHO-recommended MRL. In conclusion, there is a need for continued monitoring of pesticide residues to provide consumers with information on the level of contamination in fruits and vegetables.**
Keywords: **Organochlorine, Residues, Fruit, Vegetables, GC-MS, Kaura Namoda**

I. INTRODUCTION

Organochlorine pesticides are one of the pesticides widely used by farmers because they are considered readily available and affordable. The residues of this compound continue to persist in our food and environment [1].

Fruit and vegetables are considered vulnerable to these compounds because of the indiscriminate direct application of this organochlorine pesticide by the farmers in order to protect their crops from pest and more also, to obtain a good yield. This has led to direct human exposure to the residues since vegetables and fruits are consumed by humans [2].

The World Health Organization (WHO) has published the toxicological data on the organochlorine pesticide for use by other regulatory bodies in various countries. The evidence presented at the international conference supports the public health justification [3]. This results in a gradual facing out and outright ban of some organochlorine pesticides because of the associated health issues, such as neurotoxic, endocrine-disrupting, reproductive effects, and carcinogenic effects. As a result of this recommendation, countries such as Nigeria are using the WHO AND FAO guidelines for pesticide residues also placed a ban on the use of organochlorine pesticides [3].

Despite the ban and restrictions on the use of these compounds, evidence has shown from different researchers across Nigeria the presence of organochlorine pesticide residues in the farm land soil, water, food, and vegetables [4]. Kaura Namoda is one of the Zamfara state local government, where most of their vegetables and fruits are transported from other states of Nigeria. Most of the fruit and vegetables were purchased with minimal or non-processing procedures take before the intake of the fruits and vegetable. There is no comprehensive data in respect of the level of organochlorine pesticide residues in the fruit and vegetables consumed in Kaura Namoda and Zamfara state at large. Therefore, this research will provide baseline data on the level of organochlorine pesticide residues in the fruit and vegetables to guide the consumers and the regulatory agency [8].

II. MATERIALS AND METHODS

A. Reagents

All the chemicals, organochlorine pesticides, and other laboratory solvents used were analytical grade, used as purchased. The laboratory apparatus used were cleaned with distilled water.

B. Sample Collection

The fruit and vegetables were collected randomly from Kaura Namoda central market from the different fruit and vegetable vendors. (Banana, watermelon, guava, pineapple, and vegetables such as lettuce, spinach, cucumber, and cabbage).

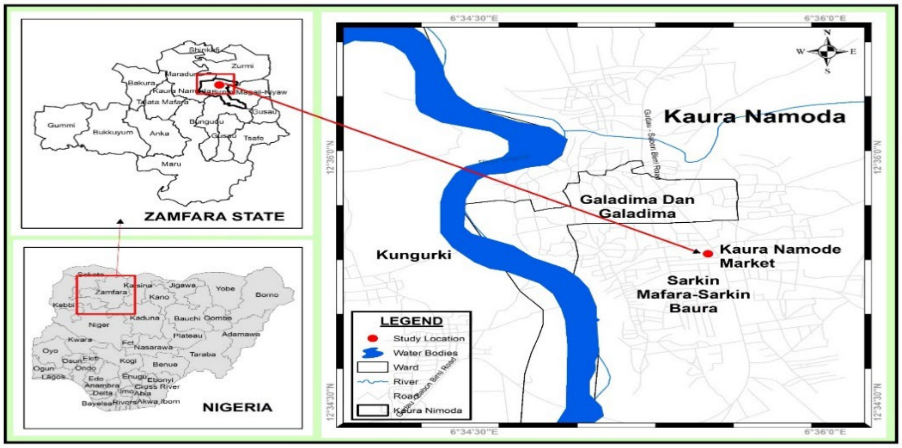

Fig.1 Map of Zamfara Showing Kaura Namoda Central Market

C. Samples Extraction

The samples were cleaned with distilled water, and inedible parts were peeled to obtain the edible parts in the case of bananas and pineapples. The samples were ground using a cleaned blending machine to obtain homogeneous samples before extraction.

Quick Essay, Cheap, Effective, Rugged, and Safe. (QuEChERS) A method was used to extract the pesticide residues from the fruit and vegetable matrix. This method was globally accepted as the best method of pesticide residue extraction [8]. Acetonitrile solvent was used because it does not mix with water in the samples [9]. Magnesium sulfate and sodium chloride were used to remove the water and increase the polarity of the samples, respectively.

D. Clean up

The vegetable and fruit extract were purified with the aid of primary and secondary amin PSA; thereafter, the solid phase was used and load pesticide cartridges with the extract. The cartridge was eluted with acetonitrile and evaporated to dryness using an evaporator; thereafter, the residue was diluted with acetonitrile and saved for GC-MS analysis [5].

III. INSTRUMENTATION, MEASUREMENT, AND CONDITION OF GC-MS

Gas chromatography coupled with a mass spectrometer is one of the analytical instruments used for pesticide residues for identification and quantification. It is used to separate a complex mixture into different components, while the MS detector will provide the quantitative and qualitative information. The GC-MS used a model 7890 Agilent Technologist with autosampler and capillary column (HP5ms). The column has 0.320mm internal diameter, 30 mm length, 30 mm length and 0.25 micrometre. The temperature was programmed from 60 °C and held for 5 minutes with the final temperature of 300 °C. The samples were injected with a spitless injector of 1 microliter with purge flow of 3.0 ml per minute while the pressure was held at 150 °C. The standard residues and the samples extract were analysed under the same conditions in mg/kg

IV. RESULTS AND DISCUSSION

Twelve organochlorine pesticide residue standards were screened from the selected fruit and vegetable samples obtained from Kaura Namoda, Zamfara. pentachloro cyclohexane was detected in watermelon, cucumber, and lettuce with concentrations of 0.0150 mg/kg, 0.0121 mg/kg, and 0.0112 mg/kg, respectively. However, it was not detected in the samples of banana, pineapple, spinach, cabbage, and guava. The concentration detected in the samples was below the WHO maximum residual limit (MRL).

These delta Pentachloro cyclohexane (HCH) residues, identified as an isomer of hexachlorocyclohexane, occurred during the degradation of the pesticide in soil. It is mostly used for insects as an insecticide. These residues persist in the soil, water and are absorbed by plants and end up in our food [4]. This residue is dangerous because of its lipophilic nature and can bioaccumulate in the body to cause toxic effects. It has been banned by the WHO.

Alpha lindane (α-HCH), also known as alpha hexachlorocyclohexane, is an organochlorine pesticide residue that is considered one of the persistent organic pollutants. Both the alpha and beta lindane standards were screened on the fruit and vegetable samples; alpha lindane was detected in Banana, watermelon, lettuce, spinach, with the concentration 0.0010 mg/kg, 0.0021 mg/kg, 0.0012 mg/kg, and 0.0031 mg/kg, respectively.

Beta-lindane was only detected in watermelon, pineapple, cucumber, spinach, and cabbage. Both alpha and beta lindane were reportedly banned by the WHO. Therefore, the concentration of the residues detected is lower than the maximum residual limit MRL set by WHO. The presence of these residues indicates that farmers are still using these pesticides for pest control.

TABLE I ORGANOCHLORINE PESTICIDES RESIDUES (MG/KG) IN FRUITS AND VEGETABLES

OCP	Banana	Watermelon	Pineapple	Cucumber	Lettuce	Spinach	Cabbage	Guava	WHO/FAO
Delta pent	NR	0.0150	NR	0.0121	0.0112	NR	NR	NR	Banned
Alpha-lindane	0.0010	0.0021	NR	NR	0.0012	0.0031	NR	NR	Banned
Beta-lindane	NR	0.0011	0.0041	0.0023	NR	0.0051	0.0012	NR	Banned
Endosulfan I	NR	0.0061	0.0030	0.0015	0.0022	0.0011	0.0033	NR	-
Endosulfan II	0.0010	0.0016	0.0030	0.0051	0.0020	0.0010	0.0030	0.0010	Banned
Heptachlor	NR	NR	NR	0.0010	0.0011	NR	NR	NR	Banned
Aldrin	NR	NR	00021	NR	NR	NR	NR	NR	-
Isodrin	NR	NR	NR	0.0031	0.0016	NR	NR	NR	Banned
Heptoclor II	NR	0.0011	NR	NR	NR	0.0021	NR	NR	Banned
DDE	0.0011	0.0021	0.0032	0.0012	0.0017	0.0014	0.0018	NR	Banned
Dieldrine	NR	NR	NR	NR	NR	NR	NR	NR	Banned
Endrin ketone	NR	0.0011	0.0021	0.0014	0.0012	NR	NR	NR	Banned

Key: NR = No detected, OCPR = Organochlorine Pesticide Residue

The reasons for the continued usage of these pesticides may not be far from, that the pesticide is cheap and readily available to the farmers; moreover, they also consider these pesticides as more efficacy and give them results of protecting their crops without being mindful of the adverse effects [6],[8].

Endosulfan I and II were also detected in the fruit and vegetable samples, namely, watermelon, pineapple, cucumber, lettuce, spinach, and cabbage. It was not detected in the guava and banana samples. The concentrations were 0.0061 mg/kg, 0.003 mg/kg, 0.0015 mg/kg, 0.0011 mg/kg, and 0.0003 mg/kg, respectively. On the other hand, all the fruit and vegetable screens were found to be contaminated with endosulfan II. This Endosulfan exists as stereoisomers called alpha and beta Endosulfan, and they are hydrophobic and highly adsorbed to the particles of the soil. It was reported [8,10] that Endosulfan has a half-life of 60 – 800 days depending on the soil pH and organic matter. It was also reported that this pesticide is mainly used for fruit and vegetables. Also undergoes degradation by hydrolysis, oxidation, and with the aid of microbial activities. When these residues are absorbed into the body, they bioaccumulate in plant, animal, and human tissues. They cause endocrine disruption, reproductive effects, and nervous system [11]

Heptachlor was only detected in cucumber and lettuce samples. This shows that the farmers are still using the banned pesticides, but the concentrations are 0.0010 mg/kg and 0.0011 mg/kg, respectively, are below the maximum residual limit MRL.

Aldrin was detected only in pineapple samples with a concentration of 0.0021mg/kg below the maximum residual limit. While isodrin was only detected in cucumber and lettuce, with concentrations of 0.0031 mg/kg and 0.0016 mg/kg, respectively. Both Aldrin and Isodrin are degradation products of DDT, dieldrin, and endrin, they termed as one of the highly toxic persistent compounds in our environment. They are accumulated in the soil, water, and human tissues. Both the residues were also named as persistent organic pollutants when exposed. They cause convulsions, dizziness, and even with a chronic condition, it cause cancer and developmental effects [12].

Heptachlor II was only detected in spinach with a concentration of 0.0021 mg/kg, and it was banned from use as a pesticide. DDE was found in all the vegetables and fruit except Guava, where it was not detected. The presence of DDE may be a result of long-term use of DDT. On the other hand, dieldrin was not detected in all the vegetable and fruit samples. Edrin ketone was detected in watermelon, pineapple, cucumber, and lettuce, but it was not detected in Banana, Spinach, Cabbage, and Guava samples. Meanwhile, endrin ketone was banned by the WHO because of its persistence in the environment [12], [13].

A. Comparison of the Present Study with the Previous Work

O. Adeoluwa *et al.*, [1] assessed the organochlorine pesticide residues OCP from the leafy vegetable's samples obtained from southwest Nigeria, among the residues standard screen, heptachlor, Adrin, dieldrien, endrin, endosulfan I & II, DDE were detected with concentration 0.323 mg/kg, 0.026 mg/kg, 0.391 mg/kg, 1.456 mg/kg, 0.079 mg/kg and 3.491 mg/kg respectively. The results obtained were all higher than the present research, as shown in the table1 above. The variation of the concentration may result higher demand for the vegetables, which in turn puts pressure to the farmers and results to indiscriminate use of the organochlorine pesticide.

In another study carried out in Mina, Niger state of Nigeria, to determine the OCP residues in pepper samples from the market [5], reported the presence of heptachlor (0.129 mg/kg), Adrien (1.55 mg/kg), and dieldrin (0.22 mg/kg). although pepper samples were not used in the present study, the research also proved the use of banned organochlorine pesticides by farmers in Nigeria. The same trend occurred for the research carried out by [6].

V. CONCLUSION

The results of the present study on organochlorine pesticide residues in some commonly available fruits in Kaura Namoda, Zamfara State, show the levels of these residues in fruits, namely banana, guava, pineapple, lettuce, spinach, cucumber, and cabbage, after using twelve organochlorine pesticide residue standards. Endosulfan I, with an average concentration of 0.0023 mg/kg, was detected in all the samples, while alpha-lindane was found in pineapple and watermelon with an average concentration of 0.0045 mg/kg. Heptachlor, aldrin, and DDE were not detected in any of the fruit samples. All the pesticide residues detected were below the recommended maximum residue limits (MRLs) set by the WHO, except for Endosulfan I, which exceeded the WHO-recommended MRL. There is a need for continued seasonal monitoring of pesticide residues and for ensuring consumer awareness regarding contamination levels in fruits and vegetables.

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Declaration of Conflicting

The author declares no potential conflicts of interest with respect to the research, authorship and /or publication of this article.

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Use of Artificial Intelligence (AI) - Assisted Technology for Manuscript Preparation

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