



Assessment of Dichlorvos and Endosulfan Pesticide Residue Levels in Selected Fruits and Vegetables Sold in Some Major Markets in Ibadan, Oyo State, Nigeria

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Authors' contributions

This work was carried out in collaboration between all authors. Author AYB designed the study, performed the statistical analysis, wrote the protocol and wrote the first and final draft of the manuscript. Author OTA carried out the technical aspect and literature review. Author CBO managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Pesticides are used to increase the yield of agricultural products. They are very hazardous and toxic for organisms as well as for humans but when used properly, they constitute an important input in fruits and vegetable production. The application of different pesticides results in the contamination of soil, water and food. These pesticide residues are dangerous for environment and human health too. Different classes of pesticides are used on the basis of their active ingredients and thereby necessitated the need to monitor its accumulation on the fruits and vegetables. Thus, the aim of the study was to determine the residues of some pesticides in fruits and vegetables

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purchased from various markets in Ibadan, Oyo State. A selection of oranges, cucumber, jute leaf, bitter leaf, banana, African spinach and fluted pumpkin leaf was purchased from four major markets in Ibadan. Extraction of the samples was carried out using a mixture of chloroform; methanol; orthophosphoric acid to determine the residual concentration of pesticides. Ultraviolet-Visible Spectrophotometer was used for quantitative analysis. The results showed that Cucumber had the highest concentration of dichlorvos (4.98 ± 0.02) $\mu\text{g}/\text{kg}$ while banana had lowest level of endosulfan 0.77 ± 0.02 $\mu\text{g}/\text{kg}$. All values were all below the maximum residual limit (MRLs) and European Union (EU) set for vegetables and soil by the Codex 2009 (WHO and FAO). These results showed that the fruits and vegetables from these major markets in Ibadan are safe for human consumption. However, there is a need for continuous monitoring so as to guide the usage of pesticides.

Keywords: Pesticides; dichlorvos; endosulfan; maximum residual limit; fruits; vegetables.

1. INTRODUCTION

Use of pesticides has become an integral part of modern day agricultural practice in food production worldwide. Based on the target organisms, they may be classified as insecticides, rodenticides, fungicides, herbicides and weedicides [1], and are not only used as agricultural commodity but also used in non-agricultural sectors. Based on the chemical nature of the pesticide, it can be classified as organophosphate, organochlorides and carbamates [2]. Their very nature of killing organisms renders them harmful to humans and other living beings [3,4].

Pesticides contain different toxic elements and compounds that can kill pests selectively, and may be toxic for humans if food contaminated by them is ingested [5]. These compounds have a wide range of both acute and chronic health effects such as cancer, neurological damage, reproductive effects, immune suppression, birth defects; and are suspected endocrine disruptors [6,7]. Being persistent and toxic, they pose serious environmental and health hazards, not only in the areas of applications, but up to the food chain [5]. Pesticides applied to food crops in the field can leave potentially harmful residues [8], and may interact with the plant surfaces, be exposed to environmental factors such as wind and sun, and may be washed off during rainfall [9]; thereby exposing consumers of such pesticide affected foods to intoxication [10].

Fruits and vegetables are important components in human diet as they provide essential nutrients for maintenance of good health and prevention of diseases in humans [11,12]. Consumption of conventionally grown fruits and vegetables is a major source of non-occupational pesticide exposure [13]. Vegetables as part of human diet are usually infested by various insects, resulting

in severe loss of quality and quantity. In order to combat the insect pest problem, a lot of pesticides are used by the farmers. However, their indiscriminate use particularly at fruiting stage and non-adoption of re-entry interval can lead to bioaccumulation of pesticide residues in consumable vegetables. Contamination of vegetables with pesticide residues has been reported by several researchers [14-16]. The maximum permissible residue levels, types and amounts have been set by regulatory bodies worldwide. The maximum residual level (MRLs) is the maximum concentration of pesticide residues resulting from the use of pesticides according to good agricultural practices. Consequently, there is no table for maximum residue limit in Nigeria hence the rely on European Union (EU) standard as the guide. Determining the level of residual pesticides in vegetables and fruits being consumed by populations is an important process in establishing the level of their safety for consumption [17]. This study was undertaken to assess the residual pesticide levels in leafy vegetables and fruits being sold in some major markets in Ibadan to determine their level of contamination and safety as there is paucity of information on the pesticide residue in the vegetables and fruits consumed in Ibadan.

2. MATERIALS AND METHODS

2.1 Sample Collection and Preparation

Fresh samples of fruits (orange, banana, cucumber) and vegetables, (jute leaf, bitter leaf, African spinach and fluted pumpkin leaf) were collected from four major markets (Bodija, Apete, Sango and Mokola) in Ibadan once a week consecutively for four weeks on the market day as farmers bring these items in large quantity to the markets for sale. This study was conducted during the rainy season around July-August as

the samples are sufficient around this period. Each sample was kept inside polythene bag and taken immediately to Institute of Agricultural Research and Training, Obafemi Awolowo University, Moor Plantation, Ibadan, Nigeria for further analysis.

2.2 Extraction

The vegetables were washed with distilled water to remove the dirt and cut, while fruits were washed and peeled as consumed. All the samples were extracted fresh. Samples were milled and 2g of sample was weighed into a 250ml conical flask. One hundred and fifty millilitre (150ml) of mixture of Chloroform: Methanol: Orthophosphoric acid in the ratio 20 : 79.9 : 0.01) was added and extracted for 30 minutes by shaking on an orbital shaker and subsequently filtered through doubled layer Whatman No 42 filter paper. This extraction was repeated twice to ensure complete extraction of dichlorvos and endosulfan from the sample [18], this is necessary because dichlorvos and endosulfan pesticides are widely used to control various pests mainly in developing countries.

2.3 Determination of Dichlorvos and Endosulfan Pesticide Residues

About 5 ml of the extract was pipetted into a 100 ml beaker, 2 ml of methyl sulphonyl chloride was added and mixed thoroughly for 1 minute to develop a yellowish colour. Working standards solutions of range 10-50 ug/ml Dichlorvos and Endosulfan were prepared from 100 ug/ml Dichlorvos and Endosulfan stock solutions respectively and treated same way like the sample, while pentane was used as blank. The absorbance of the samples as well as standard solutions were read on a Cecil 2843 spectrophotometer at a wavelength 420nm. The level of the pesticide residues (Dichlorvos and Endosulfan) in $\mu\text{g}/\text{kg}$ was calculated using the formula:

$$\left\{ \frac{\text{Absorbance of sample extract} \times \text{Gradient Factor} \times 1000}{\text{Weight of sample taken}} \right\}$$

2.4 Clean Up

Residues of organophosphate OP and organochlorine OC were dissolved in ethyl acetate, these were cleaned by passing the extract through Whatman No 42 filter paper to exclude the solid part. The extraction was in diluted form and rotatory evaporator was used for concentrating the extracted sample [19].

The data were analyzed using SPSS version 15.0, data presented (means \pm standard deviation), analysis of variance i.e. ANOVA was used to interpret the data obtained at $p < 0.05$.

3. RESULTS

Table 1 shows the classification of analyzed pesticides while Tables 2 and 3 show the levels of pesticide residues in the selected vegetables and fruits from the four major markets in Ibadan respectively. The analysed vegetables had relatively low concentrations of dichlorvos and endosulfan (Table 2). The levels of dichlorvos were higher in the vegetables than the endosulfan ($p < 0.05$). Bitter leaf was highest in values of both pesticides followed by pumpkin leaf, while jute leaf was lowest in value for dichlorvos and spinach lowest in endosulfan content.

Table 1. Classification of studied pesticides

Pesticides name	Group	Molecular formula
Dichlorvos	Organophosphate	$\text{C}_4\text{H}_7\text{Cl}_2\text{O}_4\text{P}$
Endosulfan	Organochlorine	$\text{C}_9\text{H}_6\text{Cl}_6\text{O}_3\text{S}$

Table 2. Concentration of dichlorvos and endosulfan residues ($\mu\text{g}/\text{kg}$) in fresh vegetables

Samples	Dichlorvos	Endosulfan
A-Spinach	1.94 \pm 0.02 ^a	1.05 \pm 0.02 ^b
B-Spinach	3.10 \pm 0.02 ^a	1.60 \pm 0.04 ^b
M-Spinach	3.17 \pm 0.02 ^a	1.71 \pm 0.04 ^b
S-Spinach	3.25 \pm 0.01 ^a	1.91 \pm 0.02 ^b
A-Bitter leaf	3.26 \pm 0.01 ^a	2.21 \pm 0.02 ^b
B-Bitter leaf	4.54 \pm 0.01 ^d	3.10 \pm 0.02 ^e
M-Bitter leaf	4.42 \pm 0.02 ^a	2.86 \pm 0.02 ^b
S-Bitter leaf	4.84 \pm 0.01 ^a	2.92 \pm 0.01 ^b
A-Pumpkin leaf	2.92 \pm 0.02 ^a	2.02 \pm 0.06 ^b
B-Pumpkin leaf	3.40 \pm 0.01 ^c	2.64 \pm 0.01 ^d
M-Pumpkin leaf	3.25 \pm 0.02 ^c	2.74 \pm 0.01 ^d
S-Pumpkin leaf	3.18 \pm 0.01 ^a	2.80 \pm 0.01 ^b
S-Jute leaf	2.07 \pm 0.01 ^c	2.27 \pm 0.01 ^d
S-Jute leaf	2.42 \pm 0.01 ^c	2.68 \pm 0.02 ^d
S-Jute leaf	2.44 \pm 0.01 ^c	2.32 \pm 0.01 ^d
S-Jute leaf	2.62 \pm 0.01 ^c	2.82 \pm 0.01 ^d

Values with different superscripts on the same row are significantly different ($p < 0.05$)

A=Apete market; B=Bodija market; M=Mokola market; S=Sango market

Sango market spinach, bitter leaf and jute vegetable samples were highest in dichlorvos, and the spinach and pumpkin leaves highest in

endosulfan ($p < 0.05$). Bodija pumpkin leaf was highest in dichlorvos, while its bitter leaf and jute were highest in endosulfan content. Apete market vegetable samples were significantly lower ($p < 0.05$) in pesticide residues content in the studied vegetables compared with other market vegetable samples.

The levels of the pesticide residues were much lower in the selected fruits compared with the vegetables (Table 3). The levels of dichlorvos were higher than that of endosulfan in all the fruits. There were significant differences ($p < 0.05$) in residual pesticides content of the fruits from the four major markets. Mokola market orange sample was highest in the pesticides, Bodija and Sango banana samples were highest in dichlorvos, while Sango sample was highest in endosulfan content. Sango cucumber sample was highest in the residual pesticide content.

Table 3. Concentration of dichlorvos and endosulfan residues ($\mu\text{g}/\text{kg}$) in fresh fruits

Samples	Dichlorvos	Endosulfan
A-Orange	1.28 \pm 0.02 ^a	0.87 \pm 0.03 ^b
B-Orange	1.30 \pm 0.02 ^a	1.22 \pm 0.01 ^b
M-Orange	1.41 \pm 0.01 ^a	1.38 \pm 0.02 ^b
S-Orange	1.38 \pm 0.01 ^a	1.34 \pm 0.01 ^b
A-Banana	1.20 \pm 0.02 ^a	0.77 \pm 0.02 ^b
B-Banana	1.54 \pm 0.01 ^a	1.12 \pm 0.01 ^b
M-Banana	1.48 \pm 0.02 ^a	1.20 \pm 0.02 ^b
S-Banana	1.54 \pm 0.01 ^a	1.24 \pm 0.01 ^b
A-Cucumber	4.33 \pm 0.02 ^b	3.69 \pm 0.02 ^c
B-Cucumber	4.86 \pm 0.01 ^b	4.02 \pm 0.01 ^c
M-Cucumber	4.68 \pm 0.02 ^b	4.46 \pm 0.01 ^b
S-Cucumber	4.98 \pm 0.02 ^b	4.65 \pm 0.02 ^b

Values with different superscripts on the same row are significantly different ($p < 0.05$)

A=Apete market; B=Bodija market; M=Mokola market; S=Sango market

4. DISCUSSION

The detection of residual pesticides in the vegetables and fruits samples studies confirms the their presence in fruits and vegetables as reported by [20] who determined organochlorine pesticides in raw fruits, vegetables and tubers from some markets in Nigeria. The residual pesticide levels in the vegetables and fruits were low and below the concentration level set for maximum residue limit of $< 0.01 \mu\text{g}/\text{g}$ by Federal Environmental Protection Agency [21].

The highest level of dichlorvos was found in Cucumber (Table 3). This high level of dichlorvos

residue can be attributed to the rate at which the cucumber farmers make use of the pesticide. The results obtained from this study are consistent with the findings of [22], who reported that fruit and vegetable samples contained trace levels of pesticide residues which are below the recommended maximum residue limits when compared with maximum limit set by FEPA. Although all the vegetable and fruit samples were found contaminated with pesticide residues, none of their values was above MRLs specified by [23].

The detection of the different pesticides formulations in the tissues of the vegetables shows that the vegetables were exposed to pesticides at one stage of their production or another. The exposure could be in the field or at storage sites. The variation in the levels of the pesticide residues detected in the samples from the different markets could be attributed to difference in the level and type of pesticide used at the various locations that the fruits and vegetables originated from. This is in agreement with earlier studies by [8,24-25] that reported differences in pesticide residues in food items from different locations. The lower level of residual pesticides in the oranges and banana compared with the vegetables is believed to be due to the peeling of their outer covering or pericarp which may have prevented the penetration of the pesticides into the endocarp of the fruits, whereas cucumber fruit is consumed without removal of its pericarp, hence its higher value of the pesticides.

The level of endosulfan was relatively low in all the samples, with the lowest concentration found in banana (Table 3), signifying that banana is not a good accumulator of pesticides compared with other samples analyzed. Higher concentrations of dichlorvos in all the fruits and vegetables samples than endosulfan is in agreement with the findings of [26] that detected higher values of dichlorvos in all the vegetable samples analyzed. Serious exposure to dichlorvos might have damaging effect on the peripheral nervous system, chronic fatigue syndrome and effects on the heart.

Overall, the pesticide residues were higher in vegetables than in fruits except cucumber that is grown inside the soil, pesticides may reach the soil through direct application to the soil surface, incorporation in the top few inches of soil or during application to crops [26]. This can be attributed partly to the fact that vegetables are

often grown in polluted and degraded environmental conditions in the peri-urban zone as agreed to by [27] that small scale farmers in rural areas appear to respect safety requirements than those in urban areas while subsequent contamination are subjected to further pollution from vehicles while the fruits are plucked from trees. The vegetables are often irrigated with contaminated water, as well as the addition of fertilizers and metal-based pesticides to boost production [28]. Consequently, consumers prefer vegetables with no blemish unlike fruits hence more use of pesticides on vegetables than it is required by farmers.

5. CONCLUSION

The concentrations of the two types of pesticides residues detected in the fruits and vegetables from the four markets within Ibadan metropolis were found to be much lower than the European Union (EU) set maximum residue limits. This implies that the vegetables and fruits are safe for human consumption and can help in local and export markets like Europe thereby increasing farmers income. However, the high prevalence of contamination is worrisome considering their bio-accumulative nature and regularity of consumption of these food supplements. Hence there is need for regulation and monitoring of indiscriminate use of pesticides by vegetable and fruit farmers to reduce level of health risk caused by taking contaminated fruits and vegetables.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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