P-ISSN: 2305-6622; E-ISSN: 2306-3599

International Journal of

Agriculture and Biosciences

www.ijagbio.com; editor@ijagbio.com

Research Article

Constant Pesticide Residue Monitoring in Soil and Vegetables Grown under Pesticide in Abakaliki Southeastern Nigeria Required to Allay Consumers Concern on Food Safety

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Article History: 20-1298 Received: 21-Nov-21 Revised: 24-Dec-21 Accepted: 12-Jan-22

ABSTRACT

The residue of pesticide in soil and vegetables grown under pesticide was evaluated at the teaching and research farm, Department of Soil Science and Environmental Management, Ebonyi State University, Abakaliki, Southeastern Nigeria. The experiment was laid out in a randomized complete block design (RCBD) with four treatments replicated four times. The treatments were 0; 0.5; 1 and 1.5 l ha^{-1} of DD-Force pesticide applied fortnightly till harvest. The test vegetable crops were Amaranth, Garden Egg, and Okra The data generated was subjected to standard deviation and coefficient of variation. The results showed that Amaranth, Garden Egg and Okra gave chloride residue of 0.547; 0.624 and 0.585 mg kg^{-1} respectively. The untreated plots recorded 0.325; 0.331 and 0.345 mg kg^{-1} respectively. Similarly, the phosphate level in Amaranth, Garden Egg and Okra were 1.211; 1.380 and 1.350 mg kg^{-1}; while untreated plots gave 0.588; 0.835 and 0.600 mg kg^{-1} respectively. The chloride and phosphate residue passed the Maximum Residue Limit (MLR) in the vegetable crops, while the soil samples failed the phosphate MLR, but passed the chloride MLR. It was recommended that more vegetables, other crops treated and not treated with the current pesticide and other types of pesticides be investigated to ascertain the tolerable levels of these residues in our food and soil.

Key words: Chloride residue, Tolerable level, Pesticide, MLR.

INTRODUCTION

Pesticides, especially insecticides, herbicides and fungicides represent the great bulk of over one billion kilogramme of chemicals that are used annually in most countries of the world (Igboji 2015; Pretty 2002; RSC 2001; Baird 2001). Almost half of the usage of pesticides in the US involves agriculture. Indeed, the current ability in developed countries to produce and harvest large amounts of food on relatively small amounts of land with a relatively small input of human labour has been made possible by the use of pesticides (Igboji 2015; Pretty 2002; RSC 2001; Baird 2001).

Currently, the greatest use of insecticides occurs in the growing of cotton, whereas the majority of herbicides use comes in the growing of maize and soybeans (Pretty 2002; Baird 2001; RSC 2001). Most domestic households contain at least one synthetic pesticide in developed countries. Typical examples are weed killers for the lawn and garden, algae controls for the swimming pool, flea powders for use on pets and sprays to kill insects (Baird 2001; RSC 2001).

Synthetic pesticides have been of concern because of the potential impact on human health of eating food contaminated with these chemicals. About half the foods eaten in the US contain measurable levels of at least one pesticide (Baird 2001; RSC 2001). For that reason, many have been banned or restricted in their use. In most developing countries like Nigeria these pesticides are still used in checking pests of vegetables and other crops like ants, weevils, flies, downy mildew to enhance yield of leaves and fruits (Denton and Olufolaji 2000).

In Abakaliki vegetable forcing and production is part of the agrarian culture of the people during rainy and dry seasons, with more pests infestation during the rainy season. The farmers resort to use of traditional and chemical sprays to control pests including ash; DD-Force, aldrin dust (Igboji 2015; Igboji 2000). Most of the chemical pesticides are known for their persistence in the soil and residue in food products (RSC 2001; Baird 2001; Pretty 2002). There are also incidences of food poisoning arising from consumption of raw vegetables or fruits or improperly prepared ones; attributed to pesticide residues. Hence, the

Cite This Article as: Igboji and Ola P, 2021. Constant pesticide residue monitoring in soil and vegetables grown under pesticide in Abakaliki Southeastern Nigeria required to allay consumers concern on food safety. Int J Agri Biosci, 10(4): 219-223. www.ijagbio.com

motive of present study that assessed pesticide residue in vegetable crops in Abakaliki, Southeastern Nigeria.

MATERIALS AND METHODS

Geographical and Climatic Information

Abakaliki lies within Longitude 08° 06¹ E and Latitude 06° 19¹ N at an altitude of 128 meters above sea level. It lies within the derived savannah belt of South eastern Nigeria. The mean annual rainfall for 25 years (1977 – 2012) was 154.75 mm spread across April – November; while the mean annual minimum and maximum temperatures for same period were 23.58 and 32.40°C respectively; with higher and lower temperatures during the dry and rainy seasons respectively. On the other hand, the average annual sunshine hours for same period was 5.13, while the mean annual relative humidity@09/15 hrs was 80.2 and 59.93% respectively; with higher and lower relative humidity during rainy and dry season respectively. The rainfall, temperature, and relative humidity of the area are presented in Fig. 1, 2 and 3 (NMI, 2013). The soil is shallow with consolidated parent materials within 1m of the soil surface classified as *Dystric Leptosol* (Anikwe, *et al*; 1999).

Materials

The pesticide common in vegetable production in the area was DD-Force. This is formulated as Dichlorvos (DDVP) 1000 g EC (Emulsifiable Concentrate) with major active ingredients namely: methyl, chloride and phosphate (Jubaili Aggrotech, 2015). It belongs to organophosphate family described as stomach poison insecticide for pesticide group known as acaricide. The Jubaili Agrotech has patent right for its production and marketing. It is exclusively formulated for agricultural uses only. They are packaged in 100 ml; 250 ml and 1 litre. The seeds of garden egg, amaranth and okra were procured from Ebonyi State Agricultural Development Programme.

Experimental Design and Layout

The experiment was laid out in a randomized complete block design (RCBD) with four treatments, replicated four times. The treatments were: 0; 0.5; 1 and 1.5 litres ha^{-1} DD-Force equivalent to 0; 0.2; 0.4 and 0.6 ml per plot. This was diluted with 360 mls of water before application. The plot size was 3 m x 3 m ($9m^2$) with 1 m between blocks and 0.5 m within plots. The seedlings from garden egg, amaranth nursery and in-situ seeds of okra were planted at a spacing of 25 cm x 25 cm to give plant population of 64 per plot or 160,000 stands per hectare.

Soil and Tissue Sampling

Before seedbed preparation auger soil samples at depth of 0 – 20 cm was collected for routine analysis of soil physico-chemical properties. Then after seedbed preparation, planting and pesticide application, another soil samples were collected at same depth for pesticide residue analysis. Similarly, vegetable parts were picked for residue analysis after harvest.

Laboratory Procedure/Methods

The soil particle size was determined using Boyoucous method as described by Boyocous (1962). The bulk density of the soil was determined using the core method as described by Stolt (1997); while total porosity was calculated from the bulk density using an assumed particle density of 2.65 g cm^{-3}. Thus: TP = 100 [1 – BD/PD). The soil pH in water was determined using electrode glass pH meter in the ratio of 1:2.5 (soil:water) ratio as described by Mclean (1982). The soil organic carbon was determined by the modified method described by Nelson and Somners (1982); while organic matter was determined by the Kjeldahl method as described by Bremner and Mulvaney (1982). The soil available phosphorus was determined using the Bray II method as described by Bray and Kurtz (1945); while the exchangeable bases namely: calcium, magnesium, potassium and sodium was extracted using ammonium acetate, followed by Atomic Absorption Spectrophotometer as detailed by Tell and Hagarty (1984). The base saturation was got by dividing TEB by ECEC and multiplying by 100.

The chloride level was determined titrimetrically according to AOAC (2012). This method employs silver nitrate as titrant and potassium chromate as the end point indicator and the ion present in the sample is precipitated as white silver chloride:

$$Ag^{+} + Cl^{-} \rightarrow AgCl \quad (1)$$

The procedure involves the weighing of 0.50 g of the slurry sample into a washed and dried crucible and then ashed in a furnace at 500 °C for 3h. This is allowed to cool at room temperature and then dissolved in 10 mL of nitric acid (HNO_3). About 5 mL of ash (aliquot) was pipetted into conical flask and was titrated with 0.I N silver nitrate ($AgNO_3$) using potassium chromate as indicator (K_2CrO_4). The appearance of brown reddish precipitate marked the end point or titre value which is noted. The chloride concentration (mg L^{-1}) is thus calculated as:

$$C_c = T_V \times 35.5 \quad (2)$$

Where C_C is the chloride concentration and T_V is the titre value.

The phosphate status was determined colorimetrically (AOAC, 2012). 0.5 g of the slurry sample was weighed into a well washed and dried crucible. The sample was ashed in muffle furnace at 500 °C for 3 h and then allowed to cool. It was digested with 5 mL of HNO_3 and the volume made up to 50 mL with distilled water. Approximately 5 mL of the diluted sample was pipetted into a test tube and 2 mL ammonium molybdate and 10N sulphuric acid solution was added. The mixture was swirled and allowed for 10 min. The absorbance was read at 420 nm using distilled water as blank. The standard solutions were treated as test sample. The calibration graph for the phosphate standard was used to read off the concentration of the phosphate:

$$C_p = A_b \times G_{spg} \quad (3)$$

where C_p is the concentration of the phosphate, Ab is the absorbance and G_{spg} is the gradient from the standard plot of the phosphate concentration.

Statistical and Data Analysis

The data were subjected to statistical variations using coefficient of variation, standard deviation as described by steel and Torrie (1980). The values were compared with Maximum Residue Limit as described by Obida *et al* (2012) for tissue samples and Edeogu (2007) for soil samples.

RESULTS

Soil Physico-Chemical Properties Before Pesticide Application

The results of soil analysis before pesticide application is given in Table 1. The levels of sand, silt and clay were 552.0; 304.0 and 144.0 g kg^{-1} respectively. The soil texture was sandy. The pH was 5.90, while available phosphorus was 5.90 mg kg^{-1}. The total nitrogen was 22.10 g kg^{-1}, total carbon and organic matter were 1.56 and 15.5 g kg^{-1} respectively. The exchangeable calcium, magnesium, potassium and sodium were 2.40; 1.20; 0.070 and 0.18 cmol kg^{-1}. The exchange acidity (EA) was 1.12 cmol kg^{-1}; effective cation exchange capacity (ECEC) was 4.97 cmol kg^{-1}; while base saturation was 77%.

Chloride Residue in Soil after Pesticide Application

The chloride residue in soil after the application of the Dichlorvos (DDForce) pesticide was 0.401 mg kg^{-1} in Amaranth plot; 0.447 mg kg^{-1} in garden egg plot; 0.378 mg kg^{-1} in Okra. The control plot gave 0.278 mg kg^{-1} across the vegetables tested. The standard deviation between pesticide treated and untreated plots were 0.65, 0.64, and 0.66 mg kg^{-1} in Amaranth, Garden Egg and Okra respectively. Their corresponding coefficient of variation were 189.43; 175.73; and 200.02 percent respectively. When this is compared to the Maximum Permissible levels in soil set by WHO as reported by Edeogu (2007) it was found that the chloride level passed the standard – Table 2.

Phosphate Residue in Soil after Pesticide Application

On the other hand the phosphate residue in the soil after cropping were 1.205, 1.311 and 1.050 mg kg^{-1} in Amaranth, Garden Egg and Okra respectively. The control in respective plots were 1.165 mg kg^{-1}. The standard deviation across the treated and untreated plots were 0.66, 0.67 and 0.64 mg kg^{-1} for Amaranth, Garden Egg and Okra; with cvs of 57.38; 51.91 and 47.06% respectively. The MLR permissible limits in the soil as reported by Edeogu (2007) was 0.4 mg kg^{-1}. Based on this, the tested soil exceeded the maximum permissible limit set for phosphate in the soil – Table 2.

Chloride and Phosphate Residue in Vegetable Biomass

In Table 2, the chloride residue in Amaranth, Garden Egg and Okra were 0.547, 0.624, and 0.585 mg kg^{-1} in treated plots; while in control the residue levels were 0.325, 0.331 and 0.345 mg kg^{-1} for the respective crops. The standard deviation were 0.625; 0.641 and 0.623 mg kg^{-1} across the treated and control plots; with cvs of 143.51; 128.44 and 133.90% respectively.

With regards to phosphate residue, the values recorded for Amaranth, Garden Egg and Okra were 1.211, 1.380 and 1.350 mg kg^{-1} respectively. In control plots, the values were 0.588; 0.835 and 0.600 mg kg^{-1} respectively. The SD between treated and untreated plots were 0.589; 0.592 and 0.588 mg kg-1 respectively; with cvs of 65.55; 53.46 and 60.34% respectively. The MLR as cited by Obida *et al* (2012) was 0.02 – 2 mg kg^{-1} for all vegetables tested. The tested vegetables passed the phosphate limit tests.

Table 1: Soil physic-chemical properties before pesticide application

Soil parameter	Unit	Value
Sand	g kg-1	552.0
Silt	g kg-1	304.0
Clay	g kg-1	144.0
Texture	Dimensionless	Sandy
pH	Dimensionless	5.90
Available phosphorus	mg kg-1	22.10
Total Nitrogen	g kg-1	1.56
Total organic carbon	g kg-1	15.5
Total organic matter	g kg-1	26.7
Exchangeable Ca	cmol kg-1	2.40
Exchangeable Mg	cmol kg-1	1.20
Exchangeable K	cmol kg-1	0.070
Exchangeable Na	cmol kg-1	0.18
Exchange Acidity	cmol kg-1	1.12
ECEC	cmol kg-1	4.97
Base saturation	%	77

Fig 1: Garden egg and amaranth nursery

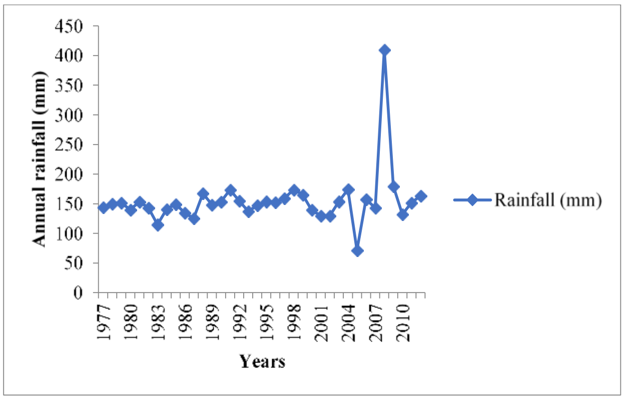

Fig 1: Annual rainfall for Abakaliki (1977 – 2012) – mm.

DISCUSSION

The fact that soil phosphate was above permissible levels may be due to parent soil phosphate. That of chloride that passed the test may be due to the fact that chloride are trace minerals in the soil that can only combine with sodium and other cations to produce soil salinity. In most cases of normal aerated and moistened soil; cases of soil salinity and sodicity do not occur. In the test soil there have never been reports of soil salinity or sodicity. Hence, the values got for the chloride may have come mainly from

Table 2: The chloride and phosphate residue from DDForce (Dichlorvos) pesticide as contained in the tissue analysis of amaranth, okra, and garden egg and that of soil samples (mg kg^{-1})

Plant Tissue Samples	Amaranth	Garden Egg	Okra	Amaranth	Garden Egg	Okra
Pesticide Residue	Chloride	Chloride	Chloride	Phosphate	Phosphate	Phosphate
Pesticide application	0.547	0.624	0.585	1.211	1.380	1.350
No pesticide application (control)	0.325	0.331	0.345	0.588	0.835	0.600
Mean	0.436	0.478	0.465	0.8995	1.108	0.975
SD	0.625	0.614	0.623	0.589	0.592	0.588
CV(%)	143.51	128.44	133.90	65.55	53.46	60.34
MLR (Maximum Residue Limit) (Source:Obida *et al*, 2012).	0.02 – 2	0.02 – 2	0.02 – 2	0.02 - 2	0.02 - 2	0.02 - 2
Soil samples						
Pesticide application	0.401	0.447	0.378	1.205	1.311	1.450
No Pesticide Application (Control)	0.278	0.278	0.278	1.165	1.165	1.165
Mean	0.344	0.3625	0.328	1.185	1.238	1.308
SD	0.65	0.64	0.66	0.67	0.64	0.62
CV(%)	189.43	175.73	200.02	57.38	51.91	47.06
MLR (Source Edeogu, 2011).	250	250	250	0.4	0.4	0.4

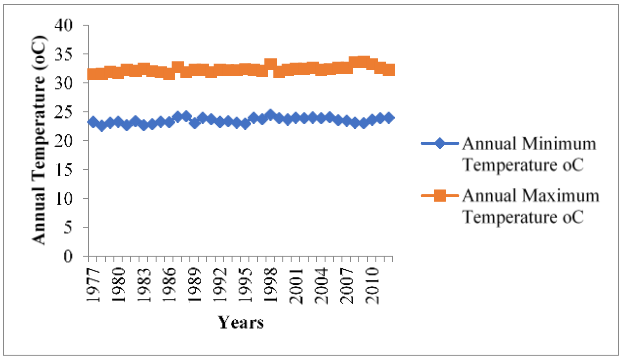

Fig 2: Annual minimum and maximum temperature for Abakaliki (1977 – 2012) - °C

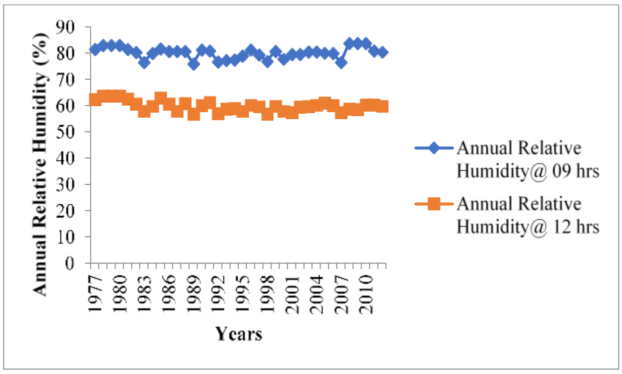

Fig. 3: Annual relative humidity@09/12 hrs at Abakaliki (1977 – 2012) - %s

Dichlorvos pesticide which has methyl, chloride and phosphate as major active ingredients.

The fact that the Amaranth, Garden Egg and Okra treated with Dichlorvos pesticide passed the maximum residue limits set for chloride and phosphate may have been due to the utilization of the anions by soil microbial communities as part of their nutrition; while the little uptake by the vegetables was still within tolerable limits. The control plots that received no pesticide, but grown with corresponding Amaranth, Garden Egg and Okra also recorded some elements of chloride and phosphate which shows that they are part of the mineral elements in the soil. Other factors like pesticide drift via rain water or air can also contribute to the chloride and phosphate recorded in control plots.

The work of Edeogu (2007) and Obida *et al.* (2012) explored various levels of anions in Abakaliki soil and crops and effects of pesticides on crops respectively as cited in Table 2. Worldwide, it has been established that pesticides residues are not desirable in our food including vegetables (Ogah, *et al.*, 2011).

Conclusions

Based on these findings it can be concluded that pesticides such as Dichlorvos (DDForce) can contribute to food poisoning if the residue concentrations exceed the maximum residue limit. Likewise, the soils can exceed the maximum permissible limits set for these anions that come from the use of pesticides in crop production. The fact that vegetables are grown and used occasssionally with pesticides to control insect pests calls for caution. There should be limits and controls their use and production. This current study is relevant in Abakaliki in view of the fact that dry and rainy season vegetable production makes use of pesticides to check pests. The vegetables being supplied to customers need regular monitoring and checks to minimize food poisoning. It was recommended that other vegetables and crops be tested using the same pesticide and other ones that dominate the market in Abakaliki in particular. Similarly, constant monitoring of food and food products in circulation in Ebonyi State and Nigeria.

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