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PESTICIDES RESIDUE LEVEL IN VEGETABLES; AN ASSESSMENT OF VEGETABLE SAFETY IN MAKURDI LOCAL GOVERNMENT AREA, BENUE STATE.

EMMANUEL O OGWUCHE [1,] **ESHIEMOKHAI COSMAS ODION**[2]
[1]Department of Environmental Sciences, University of Agriculture, Makurdi
[2]Department of Chemistry, Center for Food Technology and Research (CEFTER), Benue State University Makurdi

Abstract
An assessment of pesticide residues in vegetables was necessitated based on the fact that pesticides are very dangerous to humans and the environment, hence, their use on the increase posed a serious residual challenge. Fresh samples of Spinach (*Amaranthus hybridus*), fluted pumpkin (*Telferiaoccidentalis*) and Pepper (*Capsicum spp*) were randomly selected, and 36 composite samples of identical vegetables were drawn from commercial farmers (North bank, Wurukum, and Wadata) and Non-commercial farmers. Pesticide residue levels were determined using High Performance Liquid Chromatography (HPLC) at Chemistry Advanced research Centre, Sheda Science and Tech, Kwali. Data obtained from structured questionnaire and HPLC were statistically analyzed using Minitab 16.0, descriptive statistics operations and compared with WHO/FAO limits. The pesticide residues in the vegetables were within the WHO/FAO permissive level except the commercial vegetables; Cypermethrin in pumpkin, mean value of (21.030 ± 0.182 µg /L - 0.000±0.000µg /L), Emaectin benzoate in pepper, (1.8643 ± 0.0117 mg / ml - 0.000 ± 0.000 mg / ml) and Emaectin benzoate in pumpkin, (2.201±0.002mg/ml - 0.000±0.000mg/ml). These values were all above the WHO/FAO standards and MRLs of 0.02, 0.02, 0.01 and 0.03 mg/kg for Cypermethrin and Emaectin benzoate respectively. The presence of pesticides in these Vegetables calls for strict regulation regarding the application of pesticides in farms.

Keywords: Pesticides, Residue, Vegetables, Assessment, Safety.

INTRODUCTION
"Vegetables are edible plants or plant parts intended for cooking or eating raw" (Enenche, 2012). This definition can be extended to include some growing plants

and trees. Some vegetables are consumed raw, they only needed to be washed clean. Many others must be cooked to destroy certain natural toxins or microbes before they become edible. Yet some have to passed through some processes other than cooking before they can be eaten. Vegetables are mainly used as integral ingredients for preparing soups, stews and serve as important detail in major source of a meal (Akan *et al.,* 2013).

Major vegetables include onion, tomato, okra, pepper, Amaranthus, Carrot, Melon, *Corchorusolitorus* (ewedu), *Hibiscus sabdariffa* (sobo), *Adansoniadigtata* (baobab leaves). In Nigeria, enormous quantities of fruits and vegetables are produced and staggering figures are sometimes given as estimated annual production (Ibeawuchi*et al.,* 2015).

The production of vegetable is a tool for improving livelihood and alleviating the nutritional status of the people. The hunger and malnutrition challenge in the country can be remedied with vegetable cultivation and consumption. Vitamin "A" deficiency, a nutritional problem in Nigeria can be reduced by regularly eating fruit and green leafy vegetables (Olaniyi *et al.,* 2010). African leafy vegetables are increasingly recognized as possible contributors of both micronutrients and bioactive compounds to the diets of populations in Africa. In tropical countries of Africa where the daily diet is dominated by starchy staple foods, vegetables are the cheapest and most readily available sources of important protein, vitamins, minerals and essential amino acids (Oladiran*et al.,* 2016).

The control of pesticides residues is very important to protect consumers from effects of pesticide residue in pesticide grown vegetables. Safe food should have above all an appropriate nutritious value and contain the least possible amounts of substances that could be hazardous to health. It is therefore crucial to monitor pesticide residues in fruit and vegetables (Jolanta *et al.*, 2011).

Pesticides are group of chemicals that are deliberately used in the environment to inhibit or kill plant and animal pests and to protect agricultural and industrial products. Most commonly, pesticides are used in health sector and agricultural crops (Yadav *et al.* 2015). The continuous use of pesticides can lead to loss of biodiversity and because they cannot be easily broken down, they remain in soil, leach to ground water and surface water and contaminate the environment (Pesticide Action Network – Europe, 2010). Despite beneficial results of using pesticides in agriculture and public health sector, their use also invites deleterious environmental and public health effects. Pesticides hold a unique position among environmental contaminants due to their high biological activity and toxicity. Most pesticides do not distinguish between pests and other similar incidental life forms. They are potentially harmful to humans, animals, other

living organisms, and the environment if used incorrectly. It is estimated that about 5000 – 20,000 people died and about 500,000 to 1 million people get poisoned every year by pesticides (FAO/WHO, 2000, Yadav *et al.,* 2015). At least 50% of the intoxicated and 75% of those who die due to pesticide is agricultural workers. The rest is being poisoned due to eating of contaminated food. Pesticide residue poisoning is suspected to be a major cause of many human diseases such as cancer, cardiovascular disease, dermatitis, birth defects, morbidity, impaired immune function, neurobehavioral disorder and allergy sensitization reaction (WHO, 2002).

Maximum Residue Limits is the highest level of a pesticide residue that is legally tolerated in food or feed when pesticides are applied correctly (European Food Safety Authority-EFSA, 2016). They are intended primarily as a check that pesticides are being used in compliance with the approved label and to assist international trade in treated produce (Crop Protection Association, 2007).

A survey of literature has shown that data on pesticide residues of vegetables in Makurdi metropolis are limited. This calls for concern as the toxicological effects of the chemicals which humans and animals are exposed to daily are ever increasing (Cserhati and Szogyi, 2012, Etonihu*et al.,* 2011). Hence, this study was done to assess the concentration of pesticide residues and ascertain the safety of pesticide grown vegetables on humans, animals and environmental health.

With interest in Cypermethrin and Emaectin Benzoate pesticide in three vegetables; Spinach *(Amaranthus hybridus),* Pumpkin (*Telfairiaoccidentalis*), Pepper **(*Capsicum spp*)** grown along the banks of River Benue, Makurdi Local Government Area. Samples were drawn from three farming locations: Wurukum, Wadata, and Northbank. Fresh samples of Spinach (*Amaranthus hybridus*), fluted pumpkin (*Telferiaoccidentalis*) and Pepper (*Capsicum spp*) were randomly selected, and 36 composite samples of identical vegetables were drawn from commercial farmers (North bank, Wurukum, and Wadata) and Non-commercial farmers. Pesticide residue levels were determined using High Performance Liquid Chromatography (HPLC) at Chemistry Advanced research Centre, Sheda Science and Tech, Kwali. Data obtained from structured questionnaire and HPLC were statistically analyzed using Minitab 16.0, descriptive statistics operations and compared with WHO/FAO limits.

MATERIALS AND METHODS

Study Area

The study was done in Makurdi metropolis, Benue State along the banks of the Benue River. Makurdi is located on latitude 7°44'1.50" North and longitude

8°31'17.00" East. The daily temperature ranges between a maximum of 25.8^{0}C to 26.3^{0}C within the months of March and August recording the highest temperature. Crops grown include cassava, rice, yam, maize, groundnut, and vegetables. This research was carried out between mid April to May, 2019 along the banks of River Benue at Wurukum, Northbank, Wadata and a Control site situated at Agber village.

Field Survey

A field survey to determine the nature of Pesticides used in the study area was done using structured questionnaires through direct interview/contact. Sixty farmers were interviewed which provided demographic information, types of vegetable grown, types of pesticide used, quantity/volume of pesticide, methods of application, knowledge and perception on the hazardous effects of the pesticide and effectiveness of the pesticide used and application of the pesticides in the study areas (Wurukum, Wadata and Northbank) and control site. The farmers were assisted in filling the questionnaires using English, Tiv, Idoma, Hausa, Jukun (Native Languages). The commonly cultivated vegetables such as Spinach (*Amaranthus hybridus*), fluted pumpkin (*Telfairiaoccidentalis*) and Pepper (*Capsicum spp*) were selected for the research

Samples Collection

Fresh samples of Spinach (*Amaranthus hybridus*), fluted pumpkin (*Telfairiaoccidentalis*), and Pepper (*Capsicum spp*) were collected from three vegetable farms (Wurukum, Wadata and Northbank) located along the bank of River Benue farms and control site. The vegetable samples were randomly selected and composite samples of identical vegetable items (each containing 3 Spinach (*Amaranthus hybridus*), 3 fluted pumpkin (*Telfairiaoccidentalis*), and 3 Pepper (*Capsicum spp*)) were drawn from each study sites. The samples were wrapped in aluminium foil according to type, properly labelled, packed in polythene bags and transported to Chemistry Advanced Research Centre, Sheda Science and Technical Complex (Complex) km 10 Kwali -Abuja Road Gwagwalada for analysis.

Reagents and Materials

Analytical-grade (BEST and ATTAKE) standard pesticides from certified seller were used for preparation of standard solution in liquid form. Deionized water, dimethyl formamide; anhydrous magnesium sulfate and Primary Secondary Amine (PSA)-bonded silica were used for the sample preparation procedure.

Ultrapure water and Acetonitrile of HPLC grade were used as mobile phase, 500 mL Erlenmeyer flask was selected and 0.45 mm polyvinylidene difluoride (PVDF) filter was used for filtration before injection.

Sample Preparation

For the detection of Cypermethrin concentrations in the samples, the acetate-buffered sample preparation method for pesticides was applied to all the samples. The vegetable samples were homogenized with a house-hold homogenizer, (equipped with stainless steel knives). Ten grams of the well-chopped, homogenized sample was weighed into a 40 mL polypropylene (PP) centrifuge tube followed by addition of 10 mL of acetonitrile. The mixture was properly homogenized by shaking vigorously for approximately 1 minute. Next, an addition of 4g anhydrous MgSO4 and 1g NaCl, followed by intense agitation. Afterwards, a 1 mL aliquot of the upper acetonitrile layer was transferred into a centrifuge vial containing 25 mg of PSA sorbent and 150 mg of anhydrous MgSO4. The obtained supernatant was taken from the centrifuge vial and as a final extract to be analyzed directly using LC-techniques coupled with mass spectrometry detectors. To determine the concentration of Emaectin benzoate in the vegetable samples, approximately 10g of each sample was also weighed and macerated and 10 mL of water-methanol (50/50) was added. It was soaked overnight. The filtrate was partitioned with 10 mL acetone- hexane and the hexane layer was collected into a sample bottle. Renata et al. (2017)

High Performance Liquid Chromatography (HPLC) Analysis

Extracted samples of vegetables were analyzed by High Performance Liquid Chromatography (HPLC) following the method of Khan *et al.* (2011). The High-Performance Liquid Chromatography was performed in isocratic system using a Perkin Elmer Chromatograph including Series 200 pump, Series 200 UV/VIS detector, and a Supelco C18 analytical column (25 cm x 4.6 mm (i.d). Acetonitrile/water (20/80 v/v) was used as mobile phase. 20 µl samples were injected through auto sampler. The column temperature was kept 30 ºC with a flow rate of 1mL min^{-1}.

Statistical Analysis

Data obtained from structured questionnaires were collated and entered into Microsoft Excel Workbook (2010 version). Descriptive statistical operations were applied using frequency, percentages, tabulations pie chart and bar chart. Data obtained from quantification of pesticides in plant samples were analyzed

using Minitab 16.0. The following tests were applied: Chi square test of association, Mann Whitney U-test of comparing two non-parametric systems, Kruskal-Wallice H-test as a non-parametric One-way ANOVA analog and the Independent t-test of significance. All hypotheses were tested at 95% level of significance.

RESULTS AND DISCUSSION

Results

The percentages of pesticides types and methods of pesticides application are shown fig. 1 and 2 bellow

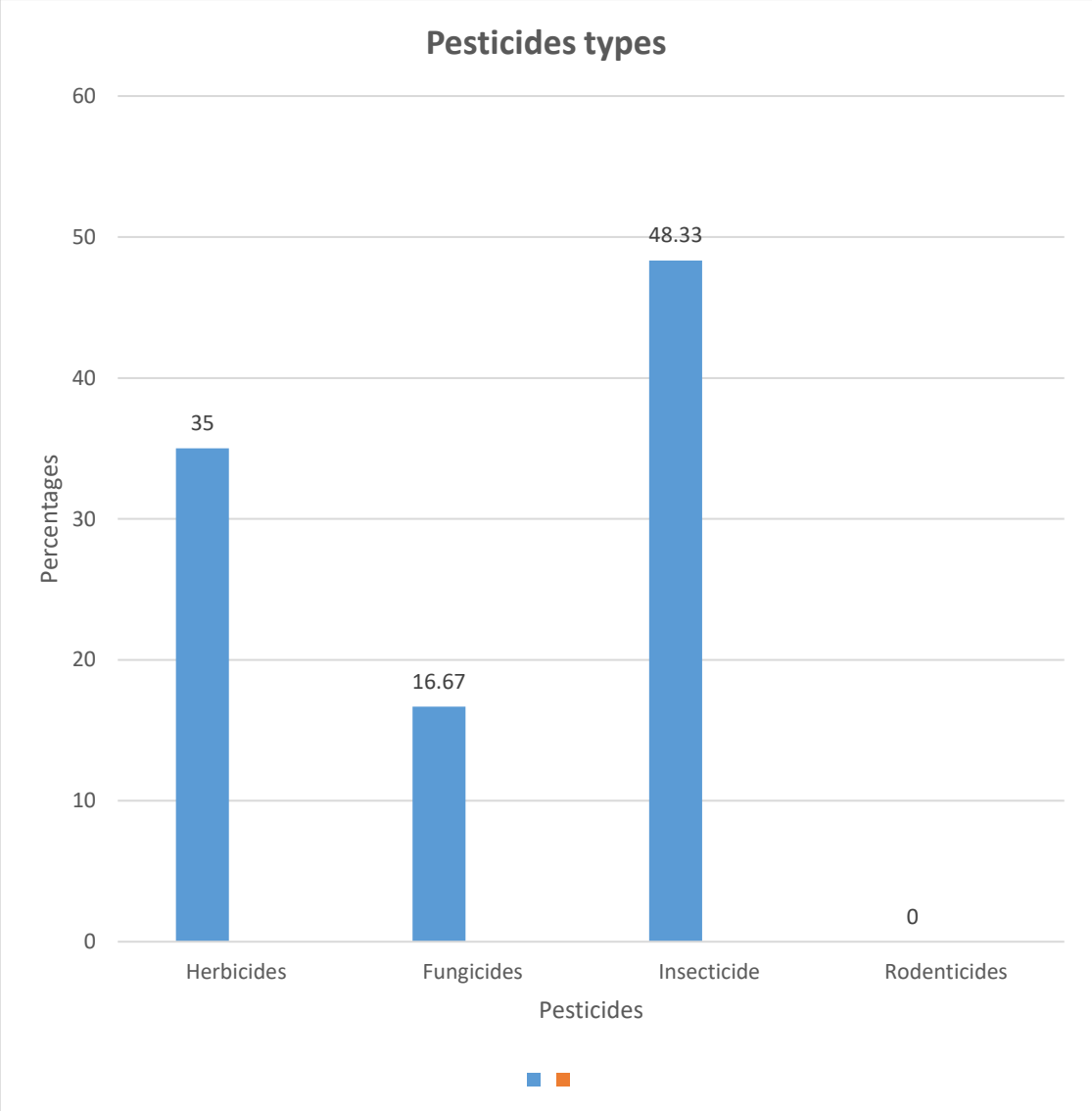

Figure 1: Types of pesticide used on vegetable farms in Makurdi.

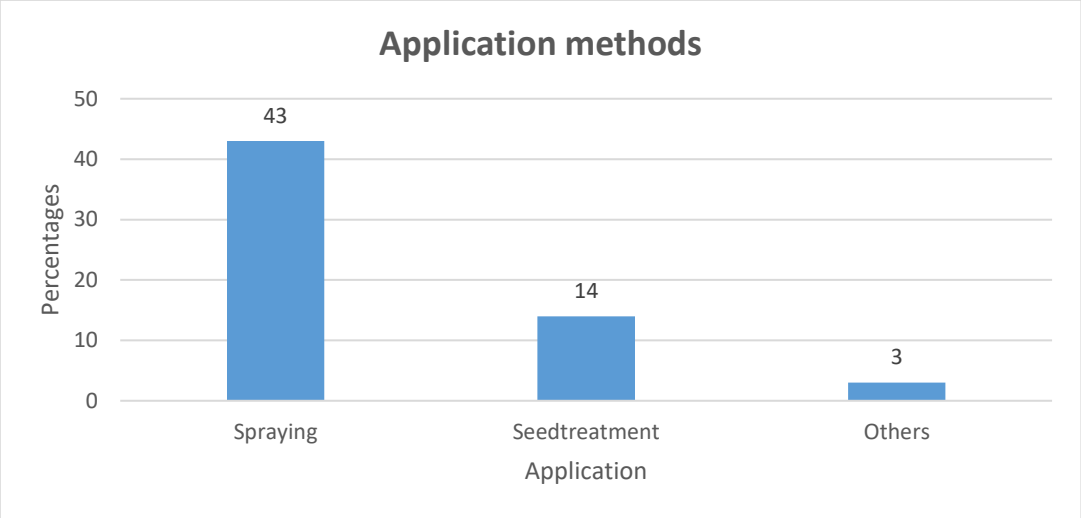

Figure 2: Application methods of pesticides used on vegetable farms in Makurdi

Mean Concentrations of pesticides residue are shown in figures 3, 4, 5 and 6. Note that cypermethrin was not found in Pepper *(Capsicum spp)* and Spinach (*Amaranthus hybridus*)

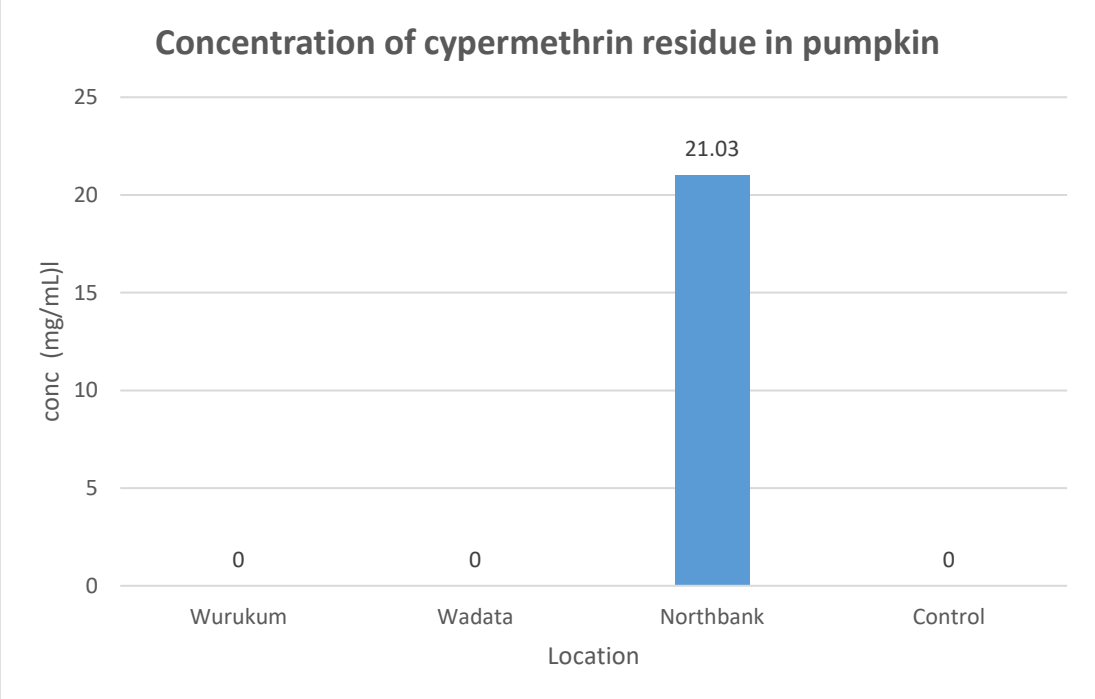

Fig. 3: Mean concentration of cypermethrin residue in pumpkin (*Telferiaoccidentalis*)

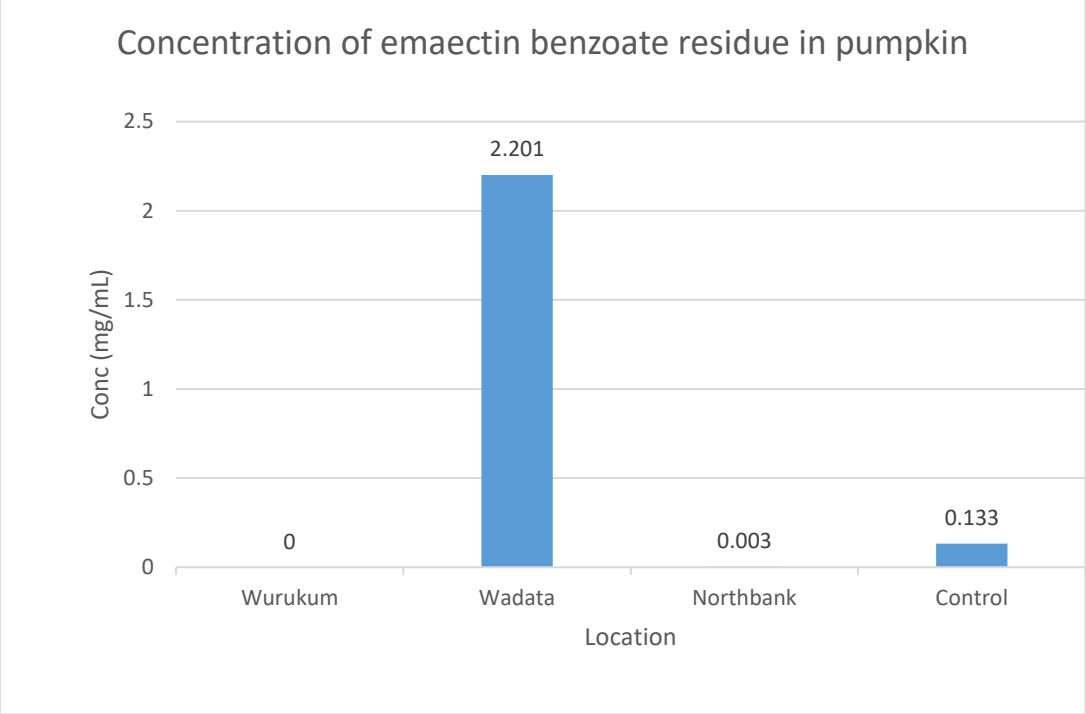

Fig. 4: Mean concentration of emaectin benzoate residue in pumpkin (*Telferiaoccidentalis*)

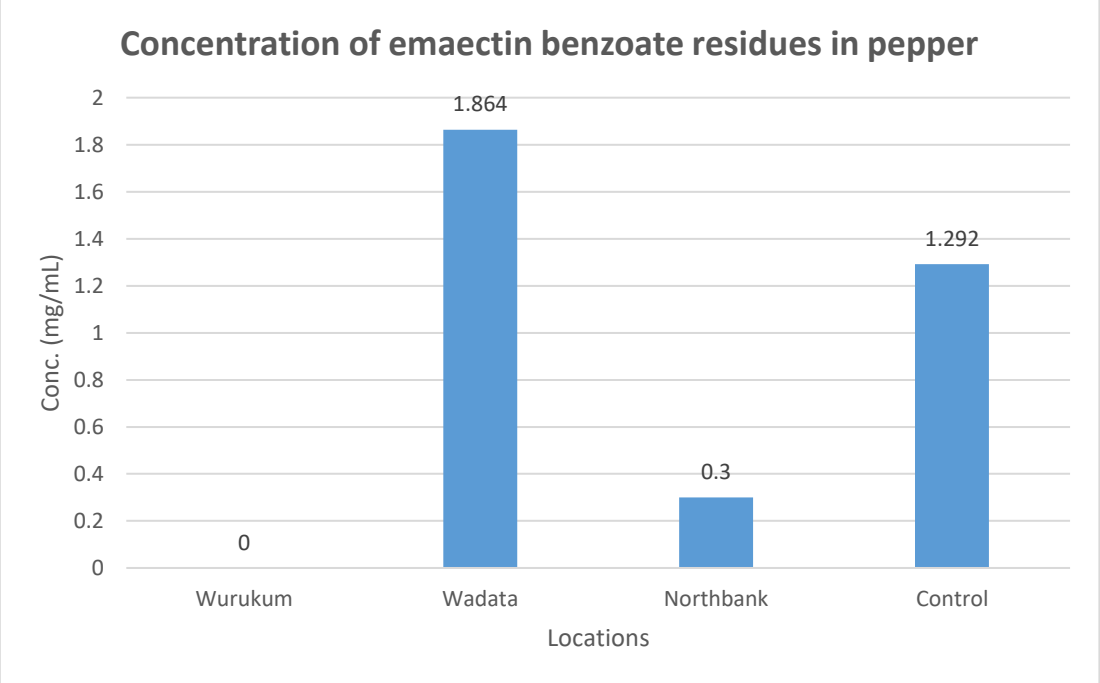

Fig. 5: Mean concentration of emaectin benzoate residues in pepper *(Capsicum spp)*

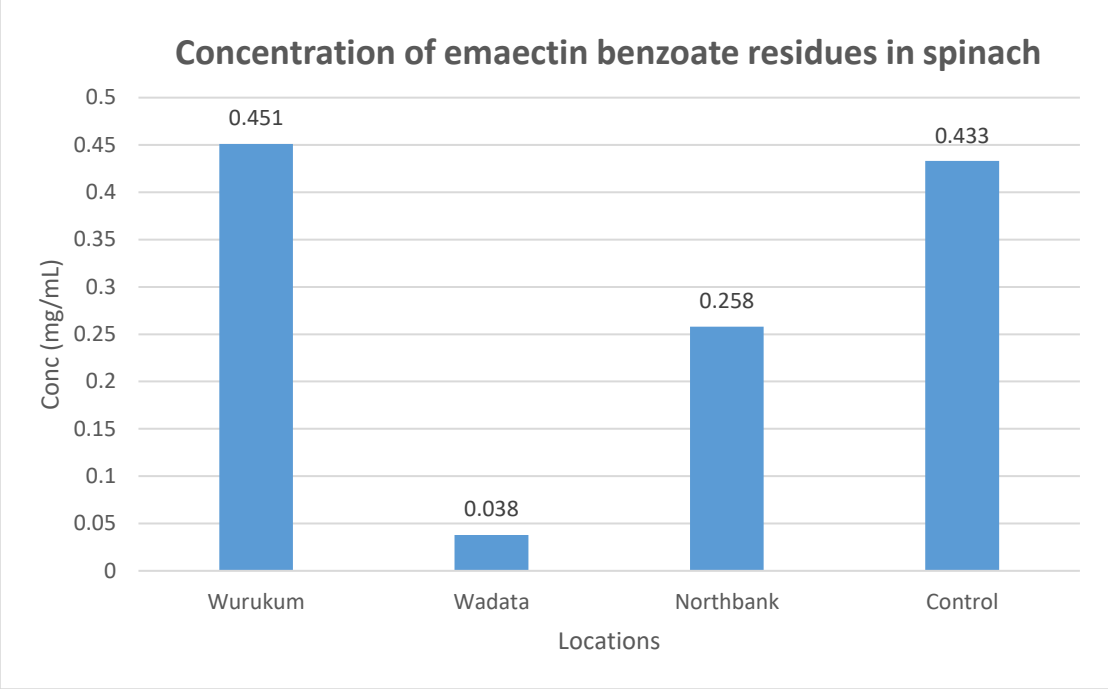

Fig. 6: Mean concentration of emaectin benzoate residues in spinach (*Amaranthus hybridus*)

Discussion

All respondents admitted using pesticides on their vegetable farms, specifying that insecticides and herbicides were the most commonly used type of pesticides. Fig. 1. Insecticides have the highest application of 48.33% while herbicide has 35.00%. 43% of the pesticide were applied by spraying and 14% by seed treatment methods. Fig. 2. Bamidele *et al.*, (2019) had reported that one of the cross cutting constraints in vegetable production are pests and diseases which often call for the use of pesticide. Hence, the level of residual pesticides in vegetables should be assessed and kept within limit.

Cypermethrin is rapidly degraded in the environment; its rate of decomposition is facilitated by its exposure to sunlight, water and oxygen (Anggoro*et al.,* 2017). The environmental persistence of many of these compounds are in the range of 1–2 days. Cypermethrin was not detected in pepper samples from the three study sites (Wurukum, Wadata, Northbank) and control site in Makurdi. The fact that Cypermethrin was not detected indicated that the pepper samples do not have cypermethrin residue or in very low concentrations and safe with no harm to consumers or the farms were not treated with cypermethrin. The residue concentration level of cypermethrin in spinach (*Amaranthus hybridus*) from the

three locations of the study and control were within the permissible limit of 0.7µg/mL FAO/WHO standards, fig. 3. There was an observed significant difference of cypermethrin residue in pumpkin which implies that the vegetable farms had presence of cypermethrin more than the control but they are within the permissive level of FAO/WHO standard of 0.7µg/mL except North bank sample that exceeded the permissive level which suggested that the pumpkin was not safe for human consumption as bioaccumulation of these residues was likely to pose health risks to consumers (Onuwa*et al.,* 2017).

The detection of Emaectin benzoate residue in all the vegetable samples reflects the presence of Emaectin benzoate residue on vegetable farms in Makurdi irrespective of the location, fig. 6. Pepper samples had residual concentrations above FAO/WHO standards (0.7µg/mL) except North bank and Wurukum, (fig. 4 and 5) were below the MRLs or showed no trace. Spinach (*Amaranthus hybridus*) and Pumpkin (*Telfairiaoccidentalis*) residue concentration exceeded FAO/WHO standards, but Wadata and North bank were below the MRLs or showed no trace. Fig. 6.

The concentration of most of these pesticides were well below the established tolerances level, but continuous consumption of such vegetables even with moderate contamination level can accumulate in the receptors body and may prove dangerous to human population in the long term (Ibrahim *et al.*; 2018). The cause of relatively low contamination may be attributed to change in the usage pattern of different groups of insecticides in Makurdi. In the past decade, there has been a shift in the use of pesticides which favor the use of organophosphorus instead of the earlier used of organochlorine which is more persistent to environmental degradation. Mostly the shift has occurred in favor of the organophosphorus insecticides because of their wide spectrum of activity and less persistence (Kumari *et al.*, 2003).

The detection of the different pesticides in the tissues of the vegetables shows that the vegetables were exposed to pesticides in one stage of their production or another. The variation in the levels of the pesticide residues detected in the tissues of the vegetables from the different vegetable farms could be attributed to difference in the level and type of pesticide used at the various locations that the vegetables grown. This is in consonance with earlier studies by Omoyajowo, *et al*., (2018) which reported differences in pesticide residues of food items from different locations.

According to locations, most of the vegetables from Wadata showed presence of Emaectin benzoate residue above the FAO/WHO standards of 0.2 mg/kg which poses a source of concern; while Wurukum and North bank vegetables were

below the MRLs. Cypermethrin was only detected in vegetables from North bank while others had no trace of Cypermethrin residue. This calls for a synergy between researchers and extension workers in agricultural sector in developing IPM strategies that will reduce their heavy reliance on pesticide usage. Maximum Residue Limits (MRLs) is defined as the highest concentrations of pesticide multi-residues in (mg/kg). However, MRLs are not safety limits and exposure to residues in excess of an MRL does not automatically imply a hazard to health (Sadło*et al.,* 2007)

Conclusion and recommendation

The analyses of the three vegetables; Spinach (*Amaranthus hybridus*), fluted pumpkin (*Telfairiaoccidentalis*), and Pepper (*Capsicum spp*) revealed the presence of pesticide residues in the vegetable. Herbicides and insecticides constituted the main pesticides that farmers use to control weeds and insects in the study sites. Pepper, spinach and pumpkin samples in Makurdi did not show Cypermethrin residual concentration or they were negligible and within the permissive level and safe with no harm to consumers in Makurdi. All samples showed presence of Emaectin benzoate residue, pepper and pumpkin concentrations were above MRLs. This is an indication of a serious threat to health, particularly on a long-term basis due to bio- accumulation. This in turn indicates that uncontrolled pesticide grown vegetable are not safe for human health. The detection of Cypermethrin and Emaectin benzoate in the tissues of the vegetables should trigger a quick check to avoid environmental harm. The variation in the levels of the pesticide residues detected in the tissues of the vegetables from the different vegetable farms could be attributed to difference in the level and type of pesticide use at the various locations that the vegetables were grown.

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