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DETERMINATION OF SOME PESTICIDE RESIDUES IN VEGETABLES (OKRO, SORREL AND SPINACH) SAMPLES FROM FORI, JERE LOCAL GOVERNMENT AREA, BORNO STATE, NIGERIA

***Aliyu S. Auwalu, Baba Abdullateef and Mohammed I. Abdullahi**

Department of Pure and Applied Chemistry, Faculty of Physical Sciences, University of Maiduguri, Borno State, Nigeria

*Corresponding authors' email: auwalunass@gmail.com

ABSTRACT

This study is aimed at determining the levels of some pesticide residues in vegetable samples (Sorrel, African spinach and Okro) from the Fori area of Jere. The vegetable samples were collected at three different sampling points designated F1 to F3. Extraction and clean-up of vegetable samples were carried out using standard procedures. The levels of the pesticide residues in the vegetable samples were determined using GC-MS. Dichlorvos was observed (178 – 2814.5 mg/kg) in all the samples from the three locations. In location, F3, 16 mg/kg and 11 mg/kg of cypermethrin were observed in okro and spinach respectively. In location, F2, 3979 mg/kg and 3220 mg/kg of cyhalothrin was observed in Okro and spinach respectively. Diazinone (3380 mg/kg) was observed only in F2 (sorrel). The concentrations of the detected pesticides in all the three vegetable samples from the three locations were greatly higher than the Minimum Regulatory Limit (MRL) values. When comparing our result with that agricultural standard (TAS 9002-2013) we found out that our MRL value is far higher than the institute value. As such we recommended that the farmers in Fori should be properly trained and directed on how to use pesticides and another related chemical to avoid health risks and toxic effects on the farmers, the plants and generally to the consumers.

Keywords: Pesticide residues, Vegetables, Samples, Jere

INTRODUCTION

In Thailand, vegetables Okra, spinach, and sourdough are important cash crops. It is grown when the pods are still green and the seeds have grown to be between 80 and 90 % of the pod width, which is after the R6 but before the R7 growth stage. Due to its distinct flavour and great nutritional value—as a source of protein, minerals including phosphorus, calcium, and iron, as well as vitamins A, B1, B2, and C—vegetable okra, spinach, and cilantro are popular throughout Nigeria and all of Africa (Cycon and piotrowska, 2016). In Asia, the crop's on-farm output falls well short of its potential. Insect infection from germination to harvest is one of the main causes of low yield. Since these pests have the potential to completely destroy crops, control is essential. Field crops will inevitably include pesticide residues as a result of their use (Cycon et al., 2016). A digestive and contact insecticide, cypermethrin, also goes by the name (RS)- -cyano- 3-phenoxybenzyl(1RS,3RS;1RS,3SR)-3-(2,2-dichloro-vinyl)2,2dimethylcyclopropanecarboxylate. It works well against a variety of insect pests, especially those that feed on leaves and fruit in cotton, fruit, vegetables, vines, tobacco, and other crops (Akbar et al., 2014). Farmers frequently use cyclomethrin to manage insect pests on their vegetables. Consumer concern and awareness about the risks posed by pesticides have grown recently. Farmers continue to support the use of pesticides for pest control despite the implementation of integrated pest management programmes, citing the latter's quick results. However, the use of pesticides either before or after harvest may leave residues on food products that could endanger consumers' health. Therefore, insecticide residues should be checked after using them to manage the Okro, Spinash, and Sorrel insect pest complex. To ensure safe consumption, an attempt has been undertaken to determine the amount of cypermethrin residue that remains in Okro, Spinash, and Sorrel days of preharvest intervals (Akbar et al., 2014).

Sorrel scientifically known as rumex acetosa is a leafy vegetable belonging to the family of polygonaceal species of R. acetosa genus. Rumex kingdom plantae of which is used by many people in Maiduguri metropolitan council, it is sour is highly consumable, sorrel is rich in sodium carbohydrate, fiber, protein and Vitamin A. Sorrel is used to treat anemia, improve eyesight, help in weight loss, detoxify the body, prevent cancer and tumor and lower blood pressure. (Blamey et al., 2003). Okro: It is referred to by its scientific name, Abelmoschus esculentus. belongs to the family of mallow flowers. Its tasty green seed pods are what make it valuable. It is a good source of fiber, vitamins, minerals, and antioxidants. It was categorized as belonging to the Okra family of marvales (Ndunguru and colleagues, 2004). Okro commonly is found in Africa as an edible plant, leaf and fruit are used for making soup and herbs, it is commonly called in Hausa *kubewa* and known as *Goltokiri* in the Kanuri language.

African spinach, sometimes known as green spinach, is a plant with more than 60–70 different varieties that is commonly grown throughout Africa. Its scientific name is Amaranthus Cruentus. Smooth or indistinct punctate, 1.2–1.6 mm in diameter, lenticular to elliptic, test delicious. A healthy heart keeps blood pressure levels stable, enhances digestion and vision, is beneficial for stomach ulcers, strengthens bones, prevents anaemia, boosts metabolism, supports brain health, is good for skin, aids in weight loss, and guards against cancer (Roberts et al., 2016). Rich in several nutrients, green leafy vegetables like spinach belong to a significant group of vegetables that have been dubbed "nature's vegetables." marvels of anti-aging and therapeutic efficacy (Gupta and Prakash, 2009). Produced on almost 921000 acres worldwide, spinach is a vital crop for both food and medicine. Over 26 million tonnes are grown there. Asia produced roughly 25 million tonnes, with China leading the way with 24 million tonnes and Iran Approximately 117,000 tonnes of spinach were produced in 2016, placing it sixth in the world (FAO, 2018). According to Roberts et al. (2016), phytochemicals and bioactives derived from spinach have the potential to reduce food intake by preventing macromolecular oxidative damage, scavenging reactive oxygen species, and stimulating the production of satiety hormones. They can also alter the expression and function of genes related to inflammation, metabolism, proliferative processes, and antioxidant defense.

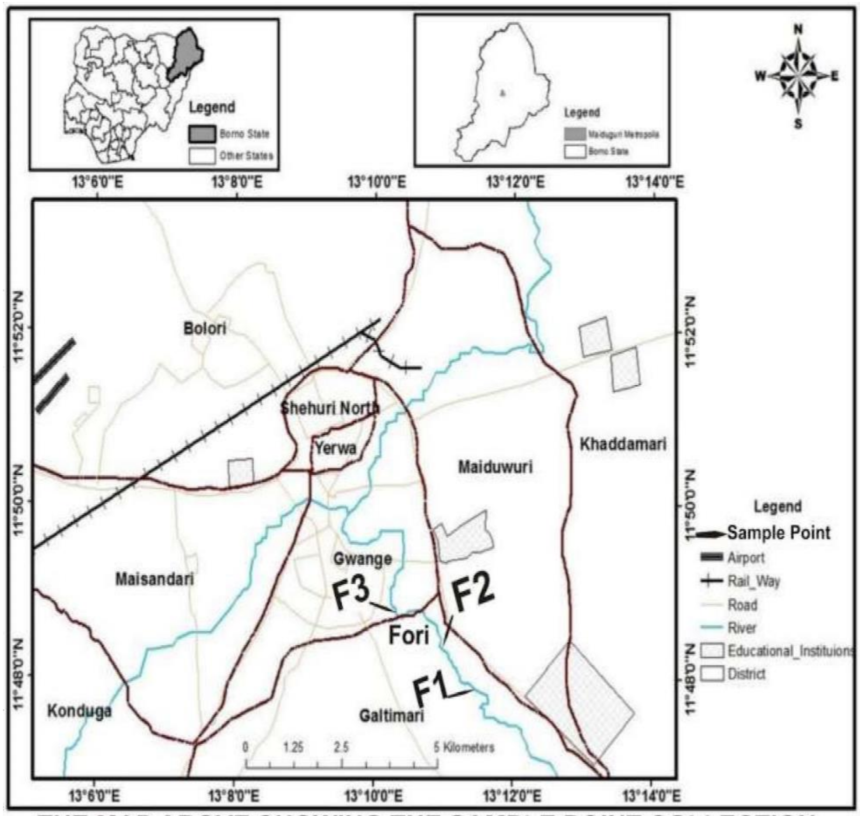
According to Akbar et al. (2014), pyrethroids are relatively harmless to mammals and avian species, but they are very toxic to aquatic life. It comes in wettable powder or emulsifiable concentrate form and is used to manage a wide range of pests, including lepidopterous pests of cotton, fruit, and vegetable crops. Approximately 98% of insecticides used in vegetable cultivation end up in non-target species, the air, water, and soil, causing pollution. Farmers respond to this issue by using a variety of chemical treatments. Synthetic Pyrethroid insecticide cypermethrin[(±)-α-Cyano-(3-phenoxyphenyl)methyl(±)-cis/trans-3-(2,2dichlorovinyl)-2,2dimethylcyclopropane carboxylate] has a comparatively low toxicity to mammals, selective insecticidal activity, and a reduced environmental persistence. Pyrethroids are quite poisonous to aquatic organisms, but they pose little hazard to mammals and bird species (Rahati, et al., 2016). It comes in emulsifiable concentrate or wettable powder form and is used to manage a wide range of pests, including lepidopterous pests of cotton, fruit, and vegetable crops. Approximately 98% of insecticides that are administered end up polluting the air, water, soil, and non-target species (Rahati et al., 2014). Pesticide usage without consideration for consequences destroys natural enemies, such as predators and parasitoids (Ngowi et al., 2007), contaminates soil and ground water, and, depending on its toxicity, may have negative effects on human health (Blamey et al., 2003). Edible vegetables nonetheless contain residual pesticides, and eating these vegetables puts human health at risk because of the food chain (Nafees & Jan, 2009). For safe use, several industrialized nations have established Maximum Residue Limits (MRLs) based on Potential Daily Intake (PDI) and Acceptable Daily Intake (ADI) (Rahman, 2007). There is evidence that using pesticides on edible vegetables increases the chance of users developing a variety of acute and chronic illnesses (Yao et al. (2018). According to research, hazardous insecticidal residue is present in between 50 and 70 percent of edible vegetables (Yao et al., 2018). Within the retention period of three to five days, no insecticide was prescribed (Yao et al., 2018). Due to farmers' ignorance of pesticide residue, a careless application of insecticides and the marketing of vegetables one to two days after spraying are thought to be standard practices (Yao et al., 2018). The breakdown mechanisms of pesticides in plants have been examined in lettuce and cabbage plants. Ester cleavage is the main metabolic process that yields α-cyano-3phenoxybenzyl alcohol. Although it's a very small pathway, hydroxylation also happens in a variety of places.

The Fori area of Jere, which is situated between latitudes 1142°N and 1200°N and longitudes 12.54 and 13.4°E, was the study's location (Haruna, 2010). Jere L.G.A. borders the city on the north, Konduga L.G.A. borders the city on the west, south, and west, and Mafa L.G.A. borders the city on the north west. The research focuses on the determination of the level of pesticide residues in vegetable samples such as Okro, green vegetables and spinach which are commonly found in the Fori area of Jere Local Government, Borno State.

MATERIALS AND METHODS

Sample Collection

Fresh Okro, spinach, and sorrel leaf samples were collected from the fori river bank of Jere, in September 2022. The samples were collected from three locations F1, F2 and F3 as shown in the map. The samples were air dried under shade, rendered free of foreign materials through manual picking and ground with a wooden mortar and pestle to a powdered form.



THE MAP ABOVE SHOWING THE SAMPLE POINT COLLECTION

Figure 1: Map of the study area

Sample Preparation

The Samples were washed with distilled water to eliminate the pollutants; the leaves were separated from the stem and dried using a drying cabinet. The dried samples were smashed into fine powder using a porcelain pestle and mortar. 5 g of each sample was weighed in different vessels before analysis. 10 ml of acetone was added and ultrasonically extracted for 20 mins. The sample was centrifuged for 20 mins at 8,000 rpm and allowed to stand overnight and then the supernatant was collected in different vessels. A mixture of 10 ml of petroleum ether and 10 mL of dichloromethane was added and votex were mixed for 1 min. The sample mixture was filtered with Whatman No 1 filter paper through a funnel containing Anhydrous Sodium Sulphate and Activated Charcoal. The filtrate was kept in an incubator for 24 hours to evaporate; finally, the extract was collected in vials and 1 mL of methanol was added for analysis.

Sample Analysis

Using the ultrasonic extraction method, 1.0 g of the material was extracted using a 1:1 ratio of methylene chloride to acetone. After the samples were weighed and put into a 50 mL flask, 10 mL aliquots of the extraction solvent combination were added, and the samples were ultrasonically extracted for 40 minutes at room temperature. Following the extraction, each combination was centrifuged for 110 minutes at 3000 rpm. After doing the extraction process twice, the extracts from each were mixed into a pear-shaped bottle and dried at a low temperature and light vacuum in a rotary evaporator. After dissolving the residue twice with 20 millilitres of n-hexane, sulfuric acid and potassium permanganate were used in succession for cleaning. The residue was eluted with the cleaning solutions after being passed through a column made of deactivated glass. Many pesticides with single components that are organochlorine or organophosphorus will be eliminated by using this cleanup method. Following cleanup, a measured aliquot of the extract was injected into a gas chromatograph fitted with a mass spectrometer detector for analysis.

Analytical Methods

Adsorptive column chromatography and solvent-solvent partition are used in conjunction with gas chromatography using an electron capture detector (GC/ECD) to determine residue. Thin layer chromatography (TLC) followed by GC/ECD or GC with mass selective detection (~C-MSD) can both be used to determine the identity of residues. Cypermethrin residues in the presence of other synthetic pyrethroids or other pesticide classes, such as organochlorine pesticides, have been measured using techniques based on these techniques.

Although they have been described, alternative techniques based on calorimetric endpoint TLC and high-performance liquid chromatography with UV detection (HPLC/UV) have not gained much traction because of the ease of use and sensitivity of GC/ECD approaches. This also holds for more intricate techniques that rely on derivatization and hydrolysis. The most significant metabolites of amide, cypermethrin, 3-phenoxybenzoic acid (PBA), and cyclopropanecarboxylic acid (CPA) were also identified using these new methods. These compounds are identified by HPLC/UV or GC following derivatization for two acids, following extraction and purification. Recommended techniques for determining cypermethrin residues are listed by the Codex Commission on Pesticide Residues (FAO/WHO 1986).

RESULTS AND DISCUSSION

Table 1: Concentration of pesticide Residues in Okro

Pesticide	RT (MIN)	Concentration (mg/kg)			MRL
		F1	F2	F3	
Dichlorvos	2.409	259	3055	2814.5	1.0
Paraquat dichloride	2.58	ND	ND	ND	
Cyphermetrin	3.801	ND	9582	6747.9	0.5
Cyhalotrin	4.282	ND	ND	16	0.3
Diazinone	5.556	ND	3979	ND	0.5
Chlorpyrifos	6.105	ND	ND	ND	
Dichlorodiphenyltoluene	6.534	ND	ND	ND	
Dieldrin	7.522	ND	ND	ND	
Aldrin	8.075	ND	ND	ND	
Heptachlor	9.431	ND	ND	ND	
Pyrazophos	11.051	ND	ND	ND	
Endrin	12.221	ND	ND	ND	
Endosulfan	13.232	ND	ND	ND	
TOTAL	93.843	259	16616	9562.4	2.3

Note: F1=Fori1, F2=Fori2, and F3=Fori3, ND = Not detected

Table 2: Concentration of Pesticide Residue in sorrel

Pesticide	RT (MIN)	Concentration (mg/kg)			MRL
		F1	F2	F3	
Dichlorvos	2.409	210	2700	22.5.2	1.0
Paraquat dichloride	2.58	ND	ND	ND	
Cyphermetrin	3.801	ND	7560	5580.7	0.5
Cyhalotrin	4.282	ND	ND	5221	0.3
Diazinone	5.556	ND	3380	13	0.5
Chlorpyrifos	6.105	ND	ND	ND	
Dichlorodiphenyltoluene	6.534	ND	ND	ND	

Dieldrin	7.522	ND	ND	ND	
Aldrin	8.075	ND	ND	ND	
Heptachlor	9.431	ND	ND	ND	
Pyrazophos	11.051	ND	ND	ND	
Endrin	12.221	ND	ND	ND	
Endosulfan	13.232	ND	ND	ND	
TOTAL	92.799	210	13640	10814.7	2.3

Note: F1=Fori1, F2=Fori2 and F3=Fori3, ND = Not detected

Table 3: Concentration of Pesticide Residues in Spinach

Pesticide	RT (MIN)	Concentration (mg/kg)			MRL
		F1	F2	F3	
Dichlorvos	2.409	178	2520	2100.5	1.0
Paraquat dichloride	2.58	ND	ND	ND	
Cyphermetrin	3.801	ND	6880	6747.9	0.5
Cyhalotrin	4.282	ND	ND	5221	0.3
Diazinone	5.556	ND	3220	11	0.5
Chlorpyrifos	6.105	ND	ND	ND	
Dichlorodiphenyltoluene	6.534	ND	ND	ND	
Dieldrin	7.522	ND	ND	ND	
Aldrin	8.075	ND	ND	ND	
Heptachlor	9.431	ND	ND	ND	
Pyrazophos	11.051	ND	ND	ND	
Endrin	12.221	ND	ND	ND	
Endosulfan	13.232	ND	ND	ND	
TOTAL	92.799	178	12620	14080.4	2.3

Note: F1=Fori1, F2=Fori2 and F3=Fori3, ND = Not detected

Table 1 shows the concentrations of pesticide residue in Okro which indicate that the concentration of dichlorvos in F1, F2 and F3 are 259 mg/kg, 3055 mg/kg and 28414.5 mg/kg respectively. While the concentration of cypermetrin in F2 and F3 is 9582 mg/kg, 6747.9 mg/kg respectively. Also the concentration of cyhalotrin in F3 is 16 mg/kg and the concentration of diazinone in F2 is 3979 mg/kg respectively. The concentration of dichlovos in Okro, from the three locations is higher than the (MRL) value (0.1-1.0 mg/kg)

Table 2 Shows the concentrations of pesticide residues in sorrel which indicate that the concentration of dichlorvos in F1, F2 and F3 is 210 mg/kg, 2700 mg/kg and 2215.2 mg/kg respectively. While the concentration of cypermetrin in F2 and F3 is 7560 mg/kg, 5580.7 mg/kg respectively. Also, the Concentration of cyhalotrin in F3 is 13mg/kg and the concentration of diazinone in F2 is 3380 mg/kg respectively.

Dichlovos in sorrel, from the three location are higher than the (MRL) value (0.1 -1.0 mg/kg)

Table 3 shows the concentrations of pesticide residue in spinach which indicate that the concentration of dichlorvos in F1, F2 and F3 are 178 mg/kg, 2520 mg/kg and 2100.5 mg/kg While the concentration of cypermetrin in F2 and F3 is 6880 mg/kg, 5221mg/kg respectively. Also, the concentration of F3 for cyhalotrin is 13mg/kg and the concentration of diazinone in F2 is 3220 mg/kg respectively.

The concentration dichlovos in spinach, from the three locations are higher than the (MRL) value (0.1-1.0 mg/kg). However, when comparing our result with another journal we found out that the concentration of the four vegetable results is far higher than the conventional result (TAS 9002-2013). The presence or absence of the pesticide in other parameters is because it was sprayed on the plant or it was sprayed on the plant/vegetable and the absence is because it was not sprayed. The current values are higher than that of the Thai agricultural standard (TAS 9002-2013). This might be because the spray in excess and large deposit were traced to the poor experience of the farmer.

CONCLUSION

The vegetables (Okro, Sorrel and Spinach) contain various levels of the pesticide (Dichloorvos, cypermethrine, cyahlothrin and diazinone) which are higher than the minimum regulatory limit (MRL). Therefore, the vegetables were not consumable based on the levels of detected pesticides.

RECOMMENDATIONS

To safeguard farmers and customers as well as to maintain the agricultural industry's sustainability, a few of the following recommendations must be taken into account.

i. To teach farmers about the repercussions of improper pesticide use, training courses should be structured in an understandable manner. This calls for a strategy that facilitates communication and collaboration amongst stakeholders, including farmers, retailers, and product manufacturers.
ii. Safe pesticide use should be encouraged by enacting regulations for crop protection and increasing public knowledge of pesticide residues in different agricultural products.
iii. Monitoring efforts ought to be broadened to include a variety of sources, such as markets and farms, as well as imported and organic food stores.

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