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Determination of Organochlorine Pesticides Residue Levels in some Selected Vegetables Cultivated in Mubi-north and Mubi-south LGA of Adamawa state, Nigeria

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ABSTRACT

Organochlorine pesticide residue level in some selected vegetables cultivated in Mubi-North and Mubi-South LGA of Adamawa state were evaluated in order to determine their contamination levels using QuEChERS method of extraction and GC-MS analyzer. A total of 72 samples (12 of each of the selected vegetables) were analyzed for10 organochlorine compounds. The result obtained showed that pesticide residue levels on the majority of the vegetable samples analyzed were in conformity with the maximum residue limits (MRL) stipulated by Codex Alimentarius Commission. However, levels higher than the MRLs were also detected.Residues of organochlorine pesticide higher than the MRLs were found most in tomato 30% followed by lettuce 20% and then spinach 10%. Despite the fact that majority of the vegetables indicated pesticide residue levels below the detectable limits, detection of contamination levels higher than the MRls are strongly indicative of potential health risk to consumers. It is therefore recommended that public awareness programs on pesticide residue should be organized by the authorities in agricultural production communities to protect the consumer from indiscriminate exposure to pesticides.

Keywords:- Contamination levels, Organochlorines, Maximum Residue Limits, Vegetables.

INTRODUCTION

Nutrition experts in Nigeria encourages everyone to consume more vegetables as a least means of providing the human body with adequate vitamins, minerals and fiber,however, the most alarming concern is the safety on the consumption of these vegetables due to wide spread of environmental pollutants such as pesticide, pathogens and toxic heavy metals. Vegetable famers all over the world use different types of pesticides including organochlorine pesticides (OCP)(which have been banned) against the possibility of a devastating crop loss from pests and diseases, as well as to increase productivity to meet the desired need of consumers [1], however, these same chemicals have had unforeseen effects on human health and the environment [2].Such persistent chemicals may remain on the vegetables as pesticide residues, [3].

Hazards associated with theconsumption of residuesof OCPs in food substance aretheirbioaccumulation potentialinthe adiposetissues of animals and humans through the food chain,and theirtoxiceffectsonhumansand wildlife[4].

The impacts of pesticide residue contamination of foodstuffs have been well documented in many parts of the developed countries as well as some developing countries[5],[6], [7],[8][9], [10]and[11]. On the contrary, there is very little or no data on the levels of pesticide residues on foodstuff in most of the developing countries [12]. As [11] opined, monitoring data from the developing countries is an important source of information portraying the state of environment as well as reflecting the effectiveness of environmental policies.

The studied area comprises of four selected vegetable farms in Mubi north and Mubi south local government areas of Adamawa state, these farms accumulates varying amount of pollution load from atmospheric deposits, anthropogenic inputs and agrochemical practices. The pollution load may contain different organochlorine pesticide chemicals which are gradually taken up by vegetables and finally passed to man via the food chain. With time, the levels of these pesticide chemicals become higher than the acceptable limits which may be responsible for some of the chronic diseases afflicting the human population.

A critical perusal of available literature revealed a growing interest in the investigation of pesticide residues in both human and environmental samples

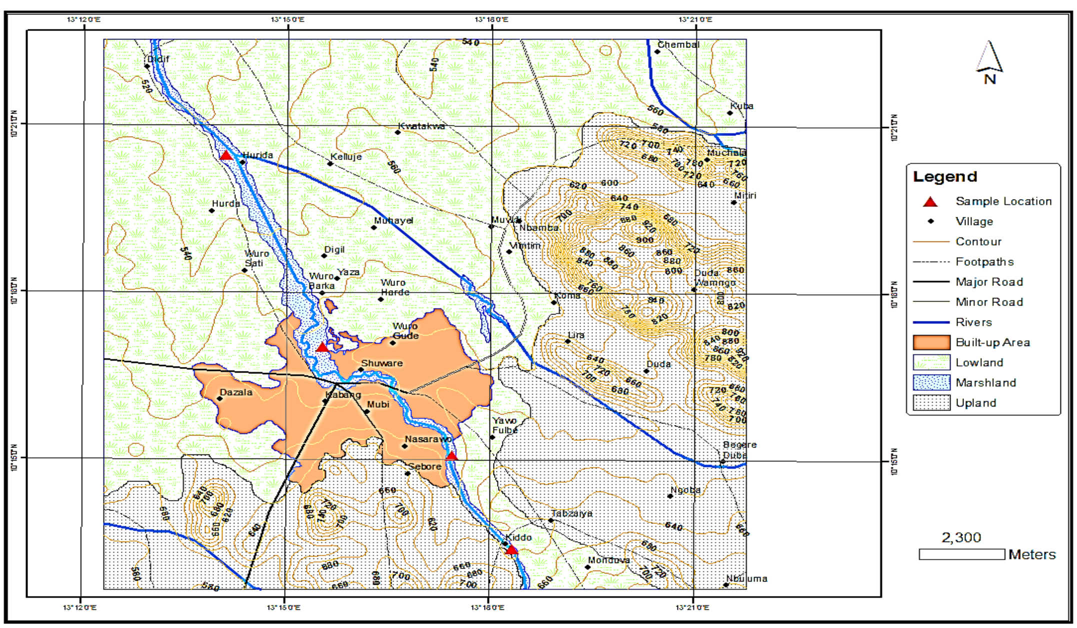
[5],[6],[13],[14] and [15]. Nevertheless, to the best of our knowledge, no investigations of regular surveys, monitoring and assessment have been reported on the concentration of pesticides residues levels of vegetables cultivated in Mubi north and Mubi South local government areas of Adamawa state Nigeria.

This work is therefore aimed at assessing the contamination level of some selected vegetables cultivated in the study area with respect to organochlorine pesticide chemicals; this will in no doubt provide us with the opportunity to proffer rational advice to the community on the issues of pesticide residues in food crops.

MATERIALS AND METHODS

Study area

The study area comprised of some selected marshlands in Mubi North and Mubi South Local Government Areas of Adamawa State where vegetables are cultivated in commercial quantity



Map of the Study Area.

Sampling of Vegetables

Sampling was performed by randomcollection of the vegetable samples from various sampling points identified in the study areas according to the Food and Agricultural Organization/World Health Organization[FAO/WHO] recommendation [3]. Three replicate samples about 1kg of the vegetable was collected from each sampling point. All the samples were packed in a well-labeled plastic bag and transported to the laboratory in an ice box immediately after collection. Samples were stored between 0^{o}C to 4^{o}C in the laboratory for analysis within 24hrs.

Samples were taken among commodities considered of high consumption rate and commonly cultivated at the study areas. E.g, Lettuce *(Lectuca sativa L.)*, Spinach *(Spinaciaoleracea L.)*, Moringa *(Moringa Oleifera L.)*, Okra *(Abelmoschusesculentus L.)*, Pepper *(Capsicum L.)*, and Tomato*(Lycopersiconesculentus L)*.

Extraction and Clean-up from Vegetable Samples

QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) extraction, Association of Analytical Chemists[AOAC] method [26] with slight modification was used for the extraction of pesticide residues from the vegetable samples. Fresh vegetable sample was thoroughly shredded and homogenized. 15g of the homogenized sample was macerated with 15ml acetonitrile (1.0% acetic acid) in 50ml centrifuge tube the screw cap was closed, and the tube was then vigorously shaken for 1 min using a vortex mixer at a maximum speed, afterwards, 6 g anhydrous magnesium sulphate and 1.5 g sodium chloride were added then extraction was performed by shaking vigorously by hand up to down for 10 min and centrifuged for 10 min at 6000 rpm.

An aliquot of 4 ml was transferred from the supernatant to a new clean 15 ml centrifuge tube and cleaned by dispersive solid-phase extraction (SPE) with 100 mg primary-secondary amine (PSA), 20 mg activated charcoal and 600 mg magnesium sulphate $MgSO_4$. Afterwards, centrifugation was carried out as mentioned above. Then, 3 ml aliquot of the supernatant was taken and evaporated to dryness using rotary test tube evaporator. The residue was then reconstituted in 2 ml acetonitrile (i.e. 1.5 g matrix per ml extract). The sample was then analyzed in Gas Chromatograph[GC] system.

Analysis

Gas chromatography–mass spectrometer (GC-MS) model GCMS-7890A Agilent technologist inert MSD 5975CM Equipped with ^{63}Ni electron capture detector was used for the analysis.

Pure nitrogen was used as the carrier gas, a low polar Hp-5MS was used as the separation column the column dimension was 30cm x 0.34mm, 0.25αm and the operating conditions were: column oven temperature: 60^{0}C, detector temperature: 300^{0}C, injection temperature 250^{0}C, flow rate of the carrier gas was 1.61ml/min, pressure of 100.2kPa and a linear velocity of 46.3cm/sec. Splint injection mode was used; a volume of 1.0μl of the sample extract was injected and 0.1 μl pure reference standard solution was analyzed in a similar manner. Peak identification was done by comparing the retention times of the pure reference standard and those obtained from the extract. Concentration of the pesticide residues was calculated using a five pointer calibration curve. The analysis was carried out at the American University of Nigeria (AUN), Adamawa state Nigeria.

Quality control and quality assurance

Quality contro land quality assurances were included in the analytical scheme. The recovery, precision and linearity of studied pesticides were evaluated by adding a working mixture to 20g of chopped untreated samples; the spiked samples were made to stand for atleast 1hour before the extraction. Three replicate samples were extracted and analyzed according to the method described by[16].Precisionwas calculated based on daily repeatability of 3samples,whereas reproducibility was carried out on 3different days. Recoveries were calculated for three replicate samples. Per cent recoveries in spiked samples ranged between 79% and 110%. Accordingly, the sample analysis data were corrected for these recoveries. Detection limit(s) of the method were also assessed based on the lowest concentrations of the residues in each of the matrices that could be reproducibly measured at the operating conditions of the GC; whichwere 0.001mg/kg.A blank analysis wasalso carried outinordertocheckanyinterferingspeciesin thereagents[5].

RESULTS AND DISCUSSION

Organochlorine pesticides which arewidely used in agriculture and animal production for the control of various insects and diseases are highly persistent. Most of them have been banned, yet their residues still appear as pollutants in food as well as in the environment.

Levels of Organochlorine Pesticides Residues in Vegetables Cultivated from Hurida

Table 1 showed the prevalence and levels of pesticide residues found in the vegetable samples collected from Hurida. Among the compounds studied in the samples, lambdacyhalothrine is the predominant compound in the vegetables. The detected level of it ranges from 1.023 mg/kg in lettuce and 0.026 mg/kg in tomato, followed by permethrin with highest concentration of 0.006 mg/kg in okra and lowest concentration of 0.001 mg/kg in lettuce. p,p'-DDE and lindane have shown co-occurrence with maximum levels of 0.045 mg/kg and 0.003 mg/kg in spinach and tomato and minimum levels of 0.043 mg/kg and 0.002 mg/kg in spinach and lettuce respectively.

DDT, endrin, endosulfan and methoxiclor were not detected in all the vegetable samples. Meanwhile, p,p'-DDT, endosulfansulfate and dieldrin occur once each.

Table1: Mean Concentration (mg/kg) OCP Residues on Vegetable Cultivated from Hurida, (Mubi north LGA)

Pesticide			Vegetables			
Oganochlorines	Lettuce	Moringa	Okra	Pepper	Spinach	Tomato
p,p'-DDE	0.043±0.001	<0.001	<0.001	<0.001	0.045±0.001	<0.001
DDT	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
p,p'-DDT	<0.001	<0.001	<0.001	0.032±0.001	<0.001	<0.001
Lambda Cyhalothrine	1.023*±0.001	<0.001	0.508±0.001	0.042±0.001	0.051±0.001	0.026±0.001
Endrin	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Endosulfan	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Endosulfan sulphate	<0.001	<0.001	<0.001	<0.001	0.003±0.001	<0.001
Lindane	0.003±0.001	<0.001	<0.001	<0.001	0.002±0.001	<0.001
Methoxyclor	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Permethrin	0.001±0.001	0.003±0.001	0.006±0.001	<0.001	<0.001	<0.001

OCP= Organochlorine pesticide, Detection Limit (0.001), *Indicates values greater than MRL

Levels of Organochlorine Pesticides Residues in Vegetables Cultivated from Wurogude

Table 2 shows the prevalence and levels of pesticide residues found in the vegetable samples collected from Wurogude. Among the compounds studied in the samples, permethrin is the predominant compound detected in the vegetables. The detected level of it ranges from 0.032 mg/kg in pepper and 0.004 mg/kg in tomato, followed by lambdacyhalothrine with maximum concentration of 1.044 mg/kg in tomatoes and minimum concentration of 0.041 mg/kg in pepper.

P,p'-DDE, p,p'-DDT, endosulfansulfate, lindane and dieldrin occurred in two samples each. DDT and methoxiclor was not detected in all the vegetable samples analyzed.While, eldrin and endosulfan occurred only once each in moringa and tomato.

Table 2 Mean Concentration (mg/kg) OCP Residues on Vegetable Cultivated from Wurogude, (Mubi north LGA)

Pesticide			Vegetables			
Organochlorines	Lettuce	Moringa	Okra	Pepper	Spinach	Tomato
p,p'-DDE	0.044±0.001	<0.001	<0.001	<0.001	0.057*±0.001	<0.001
DDT	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
p,p'-DDT	<0.001	<0.001	<0.001	<0.001	0.042 ±0.001	0.033±0.001
Lambda Cyhalothrine	0.750±0.001	<0.001	<0.001	0.041±0.001	<0.001	1.044*±0.001
Endrin	<0.001	0.001±0.001	<0.001	<0.001	<0.001	<0.001
Endosulfan	<0.001	<0.001	<0.001	<0.001	<0.001	0.042±0.001
Endosulfan sulphate	<0.001	<0.001	0.023±0.011	<0.001	0.048±0.001	<0.001
Lindane	0.006±0.001	<0.001	<0.001	<0.001	<0.001	0.004±0.001
Methoxyclor	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Permethrin	0.011±0.001	<0.001	<0.001	0.032±0.001	0.013±0.001	0.004±0.001

OCP= Organochlorine pesticide, Detection Limit (0.001), *Indicates values greater than MRL

Levels of Organochlorine Pesticides Residues in Vegetables Cultivated from Sebore

Table 3 shows the distribution and levels of pesticide residues found in the vegetable samples analyzed. Among the compounds studied in the samples, lambdacyhalothrine and permethrin are the predominant compounds in the vegetables. Maximum levels of 0.604 mg/kg and 0.041 mg/kg were detected in okra and pepper while minimum levels of 0.033 mg/kg and 0.001 mg/kg were detected in pepper and moringa respectively, followed by p,p'-DDE, with highest level of 0.062 mg/kg in lettuce and lowest level of 0.002 mg/kg in pepper.p,p'-DDT, and endosulfan were detected in two samples each.

DDT, endosulfansulfate, methoxiclor and dieldrin were not detected in all the vegetable samples analyzed, while, endrin and lindane were detected in lettuce and spinach respectively.

Table 3 Mean Concentration (mg/kg) OCP Residues on Vegetable Cultivated from Sebore, (Mubi South LGA)

Pesticide			Vegetables			
Oganochlorines	Lettuce	Moringa	Okra	Pepper	Spinach	Tomato
p,p'-DDE	0.062*±0.001	<0.001	<0.001	0.002±0.001	0.027±0.001	<0.001
DDT	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
p,p'-DDT	<0.001	<0.001	<0.001	<0.001	0.042 ±0.001	0.033±0.001
Lambda Cyhalothrine	0.250±0.001	<0.001	0.604±0.001	0.033±0.001	0.053±0.001	0.044±0.001
Endrin	0.003±0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Endosulfan	0.042±0.001	<0.001	<0.001	<0.001	<0.001	0.053*±0.001
Endosulfan sulphate	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Lindane	<0.001	<0.001	<0.001	<0.001	0.001±0.001	<0.001
Methoxyclor	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Permethrin	0.002±0.001	0.001±0.001	<0.001	0.041±0.001	0.023±0.001	0.003±0.001

OCP= Organochlorine pesticide, Detection Limit (0.001), *Indicates values greater than MRL

Levels of Organochlorine Pesticides Residues in Vegetables Cultivated from Tantila

Table 4 shows the distribution and levels of pesticide residues found in the vegetable samples collected from Tantila. Among the compounds studied in the samples, permethrin is the predominant compounds in the vegetables. Maximum levels of it was detected in spinach 0.023 mg/kg and the lowest level was detected in lettuce 0.002 mg/kg followed by p,p'-DDE, with highest level of 0.110 mg/kg in tomato and lowest level of 0.001 mg/kg in okra. Lambdacyhalothrine was detected in lettuce and tomato, while, p,p'-DDT, endrin, endosulfan, endosulfansulfate, lindaneand dieldrin were detected in one sample each (lettuce, spinach and pepper). Levels of DDT and methoxiclor were found to be below detection limit in all the samples analyzed in this area.

Table 4 Mean Concentration (mg/kg) OCP Residues on Vegetable Cultivated from Tantila, (Mubi South LGA)

Pesticide			Vegetables			
Oganochlorines	Lettuce	Moringa	Okra	Pepper	Spinach	Tomato
p,p'-DDE	0.044±0.001	<0.001	0.001±0.001	0.032±0.001	<0.001	0.110*±0.001
DDT	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
p,p'-DDT	0.029±0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Lambda Cyhalothrine	0.250±0.001	<0.001	<0.001	<0.001	<0.001	0.044±0.001
Endrin	<0.001	<0.001	<0.001	<0.001	0.001±0.001	<0.001
Endosulfan	0.041±0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Endosulfan sulphate	<0.001	<0.001	<0.001	<0.001	0.046±0.001	<0.001
Lindane	<0.001	<0.001	<0.001	<0.001	0.008±0.001	<0.001
Methoxyclor	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Permethrin	0.002±0.001	0.013±0.001	<0.001	0.013±0.001	0.023±0.001	0.003±0.001

OCP= Organochlorine pesticide, Detection Limit (0.001), *Indicates values greater than MRL

Table 5.MaximumResidueLevels (MRL)ofOrganochlorinePesticides fortheSelectedVegetables (mg/kg).

Vegetables	Lindane	Methoxychlor	Permerthrin	Dieldrin	Endrin	Endosulfans	DDT&Analolgue	Lambdacyhalothrin
Pepper.	0.01	0.01	0.05	0.02	0.01	0.05	0.05	1.0
Tomato.	0.01	0.01	0.05	0.01	0.01	0.05	0.05	1.0
Okra.	0.01	0.01	0.05	0.01	0.01	0.05	0.05	1.0
Spinach.	0.01	0.01	0.05	0.01	0.01	0.05	0.05	1.0
Lettuce.	0.01	0.01	0.05	0.01	0.01	0.05	0.05	1.0
Moringa.	0.01	0.01	0.05	0.01	0.01	0.05	0.05	1.0

Tables 1 to 4 showed the results obtained from the analysis of pesticide residues on vegetables cultivated from the studied areas. The results showed that pesticide residue levels on the majority of the vegetables analyzed from both the studied areas were in conformity with the MRLs recommended by Codex Alimentarius Commission [3]. However, levels of pesticide residues higher than the MRLs were also recorded.

Organochlorine pesticide residue levels higher than the MRLs recommended by Codex Alimentarius commission was recorded in Lettuce from Hurida1.023 mg/kg lambdacyhalothrine, spinach and tomatofrom Wurogude also gave higher level of p,p'-DDE, 0.057 mg/kg and 1.044 mg/kg lambdacyhalothrine respectively in Mubi north LGA.

Levels of pesticide residue higher than the MRLs was also recorded in lettuce and tomato from Sebore as 0.062 mg/kg p,p'-DDE and 0.053 mg/kg endosulfan respectively. p,p'-DDE level higher than the MRL was also recorded in tomato from Tantila 0.011 mg/kg, in Mubi south LGA.

Residues of organochlorine pesticide higher than the MRLs were found most in tomato 30% followed by lettuce 20% and then spinach 10%. Despite the fact that majority of the vegetables indicated pesticide residue levels below the detectable limits, detection of contamination levels higher than the MRls are strongly indicative of potential health risk to consumers.

Studies' regarding pesticide residue contamination of vegetable in Nigeria is scanty most of the studies on this subject matter centered on dairy product, water and aquatic life (mostly fish). In Ghana, it is estimated that 87% of farmers use pesticides on vegetables; insecticides are the most widely used among the different classes of pesticides [11].

The result of this research work revealed the use of organochlorines pesticide in the cultivation of vegetable crops despite the barn. Several research reports on organochlorines pesticideresidues contamination of vegetables and fruitsindicatedthat despite the barn, organochlorines pesticideare still widelyusedby vegetableproducersindifferent parts of the world [17].Nakata [18] also found elevated levels of organochlorine pesticides residues in fruits and vegetables collected from shanghai and yinxing, China. Similarly, Hura[19] reported that organochlorine pesticide residues were found present on all samples of fruits and vegetables analyzed from Eastern Romania. High levels of organochlorine pesticide residues were also reported by Mukherjee [20] in West Bengal, India.

CONCLUSION

The result of this work showed the contamination levels of some vegetables with organochlorine pesticide residues higher than the MRLs, this is as a result of indiscriminate use of the organochlorines pesticide for the cultivation of vegetable crops in the studied area due to lack of proper knowledge on the harmful effect and weakness on the barn on the importation and use of these harmful pesticides. Therefore; consumption of these vegetables poses a significant health risk to the public. The potential harmful effects could be minimized through proper enforcement of the barn on organochlorine pesticides. This study has provided more information on the pesticide contaminations in foodstuffs in Nigeria, which may be added to the information available on pesticide residues founding the food commodities in Nigeria.

RECOMMENDATIONS

On the basis of the above findings, it is recommended that, public awareness programs should be organized by the relevant authorities in agricultural communities to educate famers and the general public on the health hazard and the risk of consumption of pesticide residue laden food commodities. We also recommend for properenforcementof thebarn on the importation and use of organochlorinepesticides in other to protect the public from indiscriminate exposure to their harmful effects.

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