Original Research Article

ASSESMENT OF PESTICIDE RESIDUES IN FRESH VEGETABLES FROM THREE MAJOR MARKETS IN LAGOS USING QuEChERS METHOD AND GC-MS

ABSTRACT

The study evaluates pesticide residues in fresh vegetables from three major markets in Lagos and verified compliance of these fresh vegetables with the maximum residue levels (MRLs) as specified by Codex Alimentarius Commission. The residues were extracted by means of multi residue method based on the Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) method, Gas Chromatography - Mass Spectrometry (GC-MS) was used to determine two organophosphates (dichlorvos and chlorpyrifos), two organochlorines (endosulfanII and chlorothalonil), two carbamates(carbaryl and carbofuran) and two pyrethroids(alpha-cypermethrin and lambda-cyhalothrin). Fifteen samples of five common vegetables (cabbage, lettuce, fluted pumpkin, bitter leaf and African spinach) were analyzed. The linear ranged used were 0.005, 0.1, 0.25, 0.5 and 1.0mg/l, resulting to r^2 of ≥0.996. While the mean recoveries obtained for two fortification levels (0.1 and 0.25 ppm) in three replicates for lettuce control sample and spinach control sample were 96.7 to 104.3 % , 96.3 to 101.3 %,and 92.0 to 114.3 % ,92.1 to102.1 % respectively. A satisfactory precision of RSD<20% was recorded. The limit of detection (LOD) and limit of Quantitation (LOQ) were between 0.005 -0.050mg/kg and 0.015-0.150 mg/kg respectively. Pesticide residues detected ranges from 0.025-0.529 mg/kg. EndosulfanII, Chlorothalonil, carbaryl and Carbofuran were not detected during the entire study. However, pesticide residues detected were below the MRLs specified by Codex Alimentarius Commission.

Keyword: Pesticide residues, Vegetables, QuEChERS, GC-MS

INTRODUCTION

Vegetables are important components of the human diet since they provide essential nutrients and antioxidants that are required for most of the reactions occurring in the body. A high intake of vegetables has been encouraged not only to prevent consequences due to vitamin deficiency but also to reduce the incidence of major diseases such as cancer, cardiovascular diseases and obesity. Like other crops, vegetables are attacked by pests and diseases during production and storage leading to damages that reduce the quality and the yield. In order to reduce the loss and maintain the quality vegetables harvest, pesticides are used to destroy pests and prevent diseases. The use of pesticides has increased because they have rapid action; decrease toxins produced by food infecting organisms and are less labour intensive than other pest control methods. However, the use of pesticides during production often leads to the presence of pesticide residues in vegetables after harvest. Shrestha *et al*,(2010) reported that commercial vegetable growers opt for the application of a variety of pesticides that belong to diverse chemical class, pesticides such as Parathion-Methyl, Dichlorvos, Carbofuran , Chlorpyrifos, Cypermethrin, Deltamethrin, Dimethoate, Endosulfan, Lambda-Cyhalothrin, Profenofos, and fungicides such as Carbendazim, Mancozeb and Metalaxyl in order to overcome pest resistance and pest resurgence[1]. According

to Petsas A.S *et al*.,(2017), vegetables are capable of retaining large quantities of pesticides[2]. The presence of pesticide residues is a concern for consumers because pesticides are known to have potential harmful effects to other non-targeted organisms. The major concerns are their toxic effects such as interfering with the reproductive systems and fetal development as well as their capacity to cause cancer and asthma [3]. Some of the pesticides are persistent and therefore remain in the body causing long term exposure. Use of pesticides in Nigeria is not well controlled as compare to the developed countries due to ineffective legislation, lack of awareness and inappropriate pesticide management. Global scientific concerns have been raised regarding the potential toxicity of pesticides that have promoted their strict regulation in order to protect consumers, environment and also the users of pesticides. To ensure the safety of food for consumers, numerous legislations such as codex directives (CODEX Committee on Pesticide Residues, 2003) have established maximum residue limits (MRLs) for pesticides in foodstuffs [4]. Maximum residue limits (MRLs) values defined as the highest levels of pesticide residues that are legally tolerated in or on food or feed when pesticides are applied correctly (adoption of Good Agricultural Practices, GAPs) were established internationally by Codex Alimentarius Commission. Different extraction and quantification methods are used by various researchers for estimation of pesticide residues in several vegetables. The main criteria for opting any methodology is that analytical method should be fast, easy, inexpensive and applicable to different matrices. In recent years, gas chromatography-mass spectrometry (GC–MS) and gas chromatography–tandem mass spectrometry (GC–MS/MS) have been a versatile tool use in pesticide analysis. The ability to perform multi-analysis of analytes using GC–MS is another attribute that makes the technique unique in its applications. Analysis of several pesticides in vegetable samples from countries like Kenya, India, Pakistan, Kuwait by GC–MS in one run has been reported[5, 6, 7, 8]. The aim of this study was to evaluate presence of some pesticide residues in fresh vegetable from three major markets in Lagos and to verify compliance of these fresh vegetables with the maximum residue levels' (MRLs) as specified by Codex Alimentarius regulations for products present in the market.

EXPERIMENTAL

Sampling Collection and Storage

A total number of 75 vegetables were sampled from the 3 major vegetable markets. Five samples of each vegetable in each market were collected. The vegetable species are Lettuce, Cabbage, Fluted pumpkin, Bitter leave and spinach. Samples were packed in separated polythene bags, sealed and labeled with a unique sample identity and placed in an ice chest box. All samples were stored at 4^{o}C.The composite samples were prepared by systematic mixing of the five samples of each vegetable in each market. After the vegetable samples were mixed and blended using Stephan blender, the composite sample were then extracted within 24 hours from the time of their collection. Control sample of lettuce and spinach were also collected.

Sample Extraction and Cleanup

The QuEChERS sample preparation method for pesticides was applied to all the samples [9]. A 10 g portion of the homogenized sample was weighed into a 50 ml polytetrafluoroethylene (PTFE) tube added 10 ml of acetonitrile. Then, 4 g magnesiumsulphate (MgSO4), 1 g sodium

chloride (NaCl), 1g sodium citrate tribasic dehydrate($Na_3 C_6H_5O_7.2H_2O$) and 0.5g sodium citrate dibasic sesqihydrate ($C_6H_5Na_2 O_7.1.5H_2O$) were added, and the sample was shaken vigorously for 1 min on a vortex .The samples were then centrifuged at 3000 rpm for 5 min. The supernatant (6 ml) were transferred to a 15 ml PTFE tube to which 900 mg MgSO4, 150 mg PSA and 150 mg GCB were added. The extract was shaken using a vortex mixer for 30 second and centrifuged at 3000 rpm again for 5 min. 2ml of the supernatant were taken into a graduated test tube and 20 microliter of formic acid (HCOOH) was added to adjust the ^{pH}. These extracts were evaporated to dryness under a stream of nitrogen and reconstituted with 2ml of Hexane: Acetone (4:1) and transferred to 2 ml vial and sealed for quantification using gas chromatograph equipped with mass spectrometry (GC-MS).

Preparation of Pesticide Standard Solution

The pesticide reference standards were sourced from Dr Ehrenstorfer, Augsburg, Germany and the purity of standards ranged from 96% to99%. Individual stock standard solution of the studied pesticide were prepared by weighing accurately 10mg of each pesticide into 10ml volumetric flask and dissolved with acetone, yielding a concentration of 1mg/ml. A mixed stock standard solution of pesticides was prepared 5ug/ml in hexane: acetone (4:1).The standard mixture of pesticides was prepared at 0.005, 0.1, 0.25, 0.5 and1.0 ug/ml concentrations by serial dilution technique for preparing the calibration curve. The entire standard was stored at -20^{o}c.

Instrumental method

Chromatographic instrumentation and quantification were carried out by Gas chromatograph-mass spectrometer GC-MS (Shimadzu QP2010 Ultra) with a GC column HP-5MS 5 % phenyl-95 % methyl siloxane, 30x0.25 mm id x 0.25 film thickness. The GC operating conditions: split less injection, injector temperature 250 °C, helium carrier gas (99.9999 purity) at flow rate 1.20 mL min-1 with column head pressure 89.4 kpa, oven temperature from 100°C (1 min hold), then raised to 200 °C at the rate of 10 °C (2min hold) afterwards raised to 300 °C at the rate of10 ^{o}C (5 min hold). The sample was injected in split less modes. The MS system was routinely set in selective ion monitoring (SIM) mode and each compound was quantitated based on peak area using one target and one or two qualifier ion. Mass spectrometer parameter was set as follows: electron impact ionization mode with 70 eV e lectron energy. Ion source temperature 200 °C, MS interface temperature 250 °C.

RESULT AND DISCUSSION

Table 1 show the LOD and LOQ obtained for each pesticide. The LOD and LOQ values obtained ranged from 0.005 to 0.05mg/kg and 0.015 to 0.150mg/kg respectively. The linearity was assessed by the correlation coefficient (R^2) resulted from the five-point calibration curve. The linearity was observed in the range 0.005 -1.0mg/kg and all correlation coefficient were R^2 $\geq$0.996.

Table1: List of Pesticides with retention time (Rt), LOD, LOQ and Correlation coefficient (R^2)

Pesticide Name	Retention time (Rt)min	LOD(mg/kg)	LOQ(mg/kg)	Correlation Coefficient(R^2)
Dichlorvos	6.570	0.005	0.015	0.995860
Chlorpyrifos	13.429	0.010	0.030	0.997184
Endosulfan II	15.557	0.010	0.030	0.998576
Chlorothanolin	11.176	0.050	0.150	0.998796
Carbaryl	10.040	0.050	0.150	0.998460
Carbofuran	10.861	0.010	0.030	0.998063
α-Cypermethrin	21.919	0.005	0.015	0.999423
λ-Cyhalothrin	19.981	0.010	0.030	0.999671

Recovery study was carried out to determine the method accuracy and precision .For each blank matrix (Lettuce and spinach), two concentration levels of 0.1 ug/ml and 0.25 ug/ml at replicate (n=3) were determined. All the studied pesticides, the recoveries of these two spiking levels ranged between 92.0 to114.3 percent. The method was proved to be repeatable with RSD in range of 1.2 to 14.0 % at all spiking levels. Result obtained complied with SANCO guidelines [10]. (Document No. SANCO/12571/2013). Recovery within the range70-120 % and RSD ≤ 20 %.

Table 2`: Average recovery (n=3) and Relative standard deviation (%RSD)

	Lettuce				African Spinach			
Spiking level(ug/ml)								
	0.1		**0.25**		**0.1**		**0.25**	
Pesticide	Recovery(%)	RSD (%)	Recovery(%)	RSD (%)	Recovery(%)	RSD (%)	Recovery(%)	RSD (%)
Dichlorvos	100.7	1.4	98.7	1.6	114.3	11.8	92.7	3.4
Chlorpyrifos	102.3	6.7	99.6	1.2	95.7	7.9	100.3	14.0
EndosulfanII	100.7	2.1	101.1	2.0	102.5	6.4	99.9	7.1
Chlorothanonil	100.7	6.5	96.3	4.0	92.0	13.3	100.3	3.0
Carbaryl	101.3	4.1	98.9	1.5	104.7	8.3	98.0	6.0
Carbofuran	104.3	4,8	101.1	1.4	110.3	9.2	102.1	6.2
α-Cypermethrin	98.0	11.4	99.3	1.3	82.0	1.4	99.2	9.0
λ-Cyhalothrin	96.7	6.9	97.7	2.3	94.9	4.0	101.4	8.5

Table 3: Showing Codex Maximum Residue Limits (MRLs) of studied vegetables mg/kg [21]

Pesticide	Lettuce	Cabbage	Fluted Pumpkin	Bitter leaf	African Spinach
Dichlorvos	0.5	0.5	0.5	0.5	0.5
Chlorpyrifos	0.1	0.05	0.05	0.05	0.05
Carbaryl	10	5.0	3.0	10	10
Carbofuran	0.1	0.5	0.2	0.2	0.2
Endosulfan II	1.0	2.0	2.0	2.0	2.0
Chlorothanolin	1.0	1.0	1.0	1.0	1.0
α-Cypermethrin	2.0	1.0	1.0	1.0	1.0
λ-Cyhalothrin	0.2	0.2	0.2	0.2	0.2

Table 4: Pesticide Residue Concentrations (mg/kg) in fresh vegetable samples collected from Mile12 market.

Pesticide	Lettuce	Cabbage	Fluted Pumpkin	Bitter Leaf	African Spinach
Dichlorvos	0.178	0.268	0.048	0.025	ND
Chlorpyrifos	0.082	0.049	0.044	ND	0.038
Carbaryl	ND	ND	ND	ND	ND
Carbofuran	ND	ND	ND	ND	ND
Endosulfan II	ND	ND	ND	ND	ND
Chlorothanolin	ND	ND	ND	ND	ND
α-Cypermethrin	0.168	0.529	0.094	0.096	0.065
λ-Cyhalothrin	0.118	0.171	0.113	0.075	0.064

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181 Table 5: Pesticide Residue Concentrations (mg/kg) in fresh vegetable samples collected from
182 Oyingbo market.

Pesticide	Lettuce	Cabbage	Fluted Pumpkin	Bitter Leaf	African Spinach
Dichlorvos	0.088	0.058	0.153	0.080	0.077
Chlorpyrifos	0.060	0.044	0.039	0.038	ND
Carbaryl	ND	ND	ND	ND	ND
Carbofuran	ND	ND	ND	ND	ND
Endosulfan II	ND	ND	ND	ND	ND
Chlorothanolin	ND	ND	ND	ND	ND
α-Cypermethrin	0.045	0.072	0.028	0.090	ND
λ-Cyhalothrin	0.129	0.200	0.071	0.150	0.101

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185 Table 6: Pesticide Residue Concentrations (mg/kg) in fresh vegetable samples collected from
186 Oshodi market.

Pesticide	Lettuce	Cabbage	Fluted Pumpkin	Bitter Leaf	African Spinach
Dichlorvos	0.045	0.400	0.058	0.121	0.027
Chlorpyrifos	0.036	0.048	0.038	0.047	0.040
Carbaryl	ND	ND	ND	ND	ND
Carbofuran	ND	ND	ND	ND	ND
Endosulfan II	ND	ND	ND	ND	ND
Chlorothanolin	ND	ND	ND	ND	ND
α-Cypermethrin	0.240	0.045	0.072	0.044	0.032

λ-Cyhalothrin	0.054	0.081	0.075	ND	ND`

The result of GC-MS analysis of pesticide residues in fresh vegetable samples is presented in table 4; table 5 and table 6. Eight different pesticides were investigated and quantified with 54 detections. From table 4, residues of dichlorvos were found in 4 of 5 fresh vegetable samples in order of cabbage (0.268mg/kg)> lettuce (0.178mg/kg) > fluted pumpkin (0.048mg/kg)>bitter leaf (0.025mg/kg). None of the sample had dichlorvos above the Codex recommended MRL value of 0.5mg/kg. Chlorpyrifos was another pesticide detected in 4 of 5 fresh vegetable samples analysed in mile 12 market, This pesticide was not detected in bitter leaf, however, the concentration of chlorpyrifos was found to be in the order of lettuce(0.082mg/kg) >cabbage(0.049mg/kg)>fluted pumpkin(0.044mg/kg) >African spinach(0.038mg/kg). None of the vegetable samples exceeded the MRL value of 0.05mg/kg for cabbage, fluted pumpkin, African spinach and 0.1mg/kg stipulated for lettuce. Alpha cypermethrin was detected in all the five fresh vegetable samples from mile 12 market, detected concentration in vegetable samples werein the range of cabbage(0.529 mg/kg) > lettuce(0.168mg/kg)>bitterleaf(0.096 mg/kg)>flutedpumpkin(0.094 mg/kg)>African spinach(0.065 mg/kg) all the vegetable samples did not exceed the 1.0mg/kg MRL stated for cabbage, fluted pumpkin, bitter leaf ,African spinach and 2.0mg/kg stated for lettuce. Lambda-cyhalothrin was detected in all the five vegetable samples from mile 12 market in the order of cabbage (0.171 mg/kg)>lettuce (0.118 mg/kg)>fluted pumpkin(0.113 mg/kg)>bitter leaf(0.075 mg/kg) >African spinach(0,064mg/kg) and were all below the Codex MRL of 0.2mg/kg. EndosufanII, chlorothanolin, carbaryl and carbofuran were not detected in all the five fresh vegetable samples from mile 12. A similar finding by Adyel, T.M *et al.*,(2013) did not detect any carbofuran residues in vegetable samples[11].

Table 5 ; Result from Oyingbo market followed similar trend with that of mile 12 market, dichlorvos was detected in all the five fresh vegetable samples from Oyingbo market ranging from the fluted pumpkin(0.153 mg/kg)>lettuce(0.088 mg/kg)> bitter leaf(0.080 mg/kg) > African spinach(0.077 mg/kg) >cabbage(0.058 mg/kg). The residues of dichlorvos detected were below the MRL of 0.5 mg/kg stipulated by Codex. Chlorpyrifos residue was detected in 4 of the 5 fresh vegetable samples from Oyingbo market. The concentrations were in the order of lettuce (0.060mg/kg) > cabbage (0.044 mg/kg) > fluted pumpkin (0.039 mg/kg) > bitter leaf(0.038mg/kg). Residues in these vegetable samples were not above the MRL of 0.05 mg/kg and 0.1mg/kg for lettuce. African spinach was free of chlorpyrifos residues. This study revealed that 4 out of the 5 fresh vegetables from Oyingbo market were contaminated with α-cypermethrin in the order of bitter leaf (0.090 mg/kg)> cabbage(0.072 mg/kg) > lettuce(0.04 5mg/kg) > fluted pumpkin(0.028 mg/kg). None of the detected fresh vegetable samples exceeded the MRL of 1.0mg/kg and 2.0 mg/kg for lettuce. The concentrations of λ- cyhalothrin in this study were significantly below their MRLs of 0.2 mg/kg. Their contamination followed the order of cabbage (0.200 mg/kg)> bitter leaf (0.150 mg/kg) > lettuce(0.129 mg/kg) > African spinach(0.101mg/kg)> fluted pumpkin(0.071mg/kg). EndosulfanII, chlorothanolin, carbaryl and carbofuran were not detected. The finding of this study is comparable to the findings of the study conducted by Mahugija J.A. *et al.*,(2017) [12].

Table 6 illustrated the result of pesticide residues obtained from Oshodi market. Dichlorvos was detected in all the 5 vegetable samples from Oshodi market. The order of contamination were found to be cabbage (0.400mg/kg) > bitter leaf (0.121mg/kg)>fluted pumpkin (0.058mg/kg) >lettuce (0.045mg/kg)> African spinach(0.027mg/kg). The concentration of dichlorvos residues were not above the MRLs 0.5mg/kg set by codex. In same way, chlorpyrifos was detected in all the 5 vegetable samples ,the order of magnitude of chlorpyrifos residues in vegetable samples were as follows: cabbage(0.048 mg/kg) >bitter leaf(0.047 mg/kg)>African spinach (0.040 mg/kg)> fluted pumpkin(0.038 mg/kg) > lettuce (0.036 mg/kg). The residues of chlorpyrifos in all the vegetable samples did not exceed the MRLs of 0.05mg/kg and 0.1mg/kg set for lettuce. Similarly, α-cypermethrin was detected in all the 5 vegetable samples analysed. The result showed that lettuce (0.240 mg/kg)>fluted pumpkin(0.072 mg/kg) >cabbage (0.045 mg/kg)>bitter leaf(0.044 mg/kg) > African spinach(0.032 mg/kg).α-cypermethrin residues in this study were below the MRLs of 1.0 mg/kg and 2.0 mg/kg set for lettuce. In the same vein, λ-cyhalothrin was detected in 3 of 5 of the vegetable samples analysed. The findings revealed that cabbage (0.081mg/kg) >fluted pumpkin (0.075 mg/kg)>lettuce (0.054 mg/kg). None of the sample detected exceeded the MRLs of 0.2mg/kg set by Codex. Endosulfan II, chlorothanolin, carbaryl and carbofuran were not detected in all the samples.

This study shows the evidence of the presence of pesticide residues in vegetable samples in Lagos market. The samples analyzed contained pesticide residues below the MRLs. All the five fresh vegetable samples investigated in this study were free from contamination of endosulfan II and chlorothanolin. These results were not surprising since most of organochlorine pesticides were banned or their use is severely restricted in Nigeria. Carbaryl and carbofuran were not detected in all the vegetable samples analyzed in this study. This suggests that they were not used for the vegetables studied or there was no significant contamination due to these compounds. The occurrences of multiple residues in all the samples analyzed were likely to be a consequence of the application of different types of pesticides to protect vegetables against insect pests and diseases [1]. The incidence of multi-residue pesticide contamination in different vegetable samples has also been reported in other studies [1,13]. Pesticide residues detected in this study were similar to those detected in other studies [14, 15, 16, 17, 18, 19].

From the results in this study, it is plausible to state that vegetable growers from studied area applied good agricultural practice (GAP) and being cautious of with-hold period (WHP). A withholding period is the minimum time for vegetables treated with pesticide must wait before harvesting, such a period allows enough time for the pesticide to degrade to an acceptable level.[1,20]The results of this study were interpreted in relation to maximum residue limits(MRLs) requirements set by Codex Alimentarius Commission for vegetables.

CONCLUSION

The results indicated that all the five vegetable samples were contaminated with two or more pesticide residues with concentrations below the maximum residues limits(MRLs).From a health perspective, the observed levels of pesticide residues did not pose a potential health risk to consumers. Hence, the consumption of vegetables is safe.

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