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Fast and Easy Method of 55 Pesticide Residues Determination in Commonly Fruits and Vegetables Collected from Egyptian Local Markets

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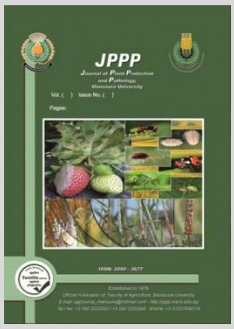

ABSTRACT

The highest concerns of consumers who need food safety are pesticide residues in fruits and vegetables. In this study, a total fifty-five pesticide residues from different chemical groups including insecticides, fungicides, herbicides, acaricides and others compounds were detected in different kinds of fruits (apple, grapes, and guava) and vegetables (squash, cucumber, tomatoes, pepper and cantaloupe) collected from local markets in Cairo Governorate. The method is based on a modified QuEChERS procedure (quick, easy, cheap, effective, rugged & safe) using acetonitrile for extraction and partition.. Analytical determinations of the 55 pesticides were carried out with gas chromatography using micro electron capture detector (µECD) and flame photometric detector (FPD).. The data obtained indicated that, the mean recoveries of the pesticides were between 81.20 and 98.70 %. The detection limits (LOD) and quantification limits (LOQ) of pesticide residues for the method were ranged (0.002 - 0.009 µg/g) and between 0.01 - 0.05 (µg/g), respectively. Of the 55 pesticides analyzed,32 (58.2%), 31(56.4), and 34(61.8) of pesticide residues were detected in the apple, grapes and guava respectively, while 30(54.5%), 31(56.4%), 30(54.5%) 24(43.6) and 27(49.1) of pesticide residues were detected in the squash, cucumber, tomatoes, pepper and cantaloupe respectively. The percentages of pesticide residues that exceeded the MRLs in both fruits and vegetables (apple. grape and guava) were12.7%, 16.4% and 16.4 (squash, cucumber, tomatoes pepper and cantaloupe) were 5.5%, 7.3%, 3.6%, 9.1% and 3.6% respectively.

Keywords: Fruits, vegetables, pesticides, QuEChERS,MRLs,GC-ECD.

INTRODUCTION

Pesticides play a significant role in food production. Pesticides are used to protect crops against insects, weeds, fungi and other pests (Guler*et al* 2010). They protect or increase yields and the number of times per year a crop can be grown on the same land. This is particularly important in countries that face food shortages.

Pesticides may remain in small amounts (called residues) in or on fruits, vegetables, grains, and other foods. To ensure the safety of the food supply for human consumption, Environmental Protection Agency (EPA) regulates the amount of each pesticide that may remain in and on foods. Pesticide residues on the food we eat are highly regulated. Although some residues may remain at the time of harvest, residues tend to decline as the pesticide breaks down over time. The pesticide residues are the deposit of active ingredient of pesticides, its metabolites or degradation products existing in the food after spraying of the pesticides. The pesticides residues entering the human diet are of major concern today (Abdulhamid *et al.*, 2015).

Fruits and vegetables are vital components of the human diet. They provide critical nutrients required for the important reactions happening in the body. The intake of five or more servings per day is considered essential for a good health and it is encouraged for vitamin deficiency prevention and also different diseases such as cancer or obesity (Keikoltlhaile and Spanoghe 2011). Also, pests and diseases which attacked fruits and vegetables during production and storage periods leads to damage and reduce the quality and the yield of these crops. A wide range of pesticides are applied for crops protection in fruits and vegetables cultivation because of heavy pests' infestation (Yu *et al*., 2016). Pesticides causes hazardous and toxicity to human health, as remaining of pesticide residue in fruits and vegetables can cause certain diseases to humans (Hassain and Siddiqe 2010). It is significant to identify and quantify the pesticides that can be remaining in fruits and vegetables after pesticide applications. Food contaminated by pesticide residues turn out to be a significant issue in the world (Park *et al*., 2016). Studies have indicated that regulation of pesticide residue limits (MRLs) in commodities is established but not fully enforced in many countries (Maclachlan and Hammiliton2010; Ambrus and Yang 2016) Therefore, it became essential analyzing the pesticide residues in both of fruits and vegetables, as well as environmental commodities. One of the major disadvantages of pesticides use is their residues that may remain on /in food and feed with amounts exceeded the maximum residue limits (MRLs) this could pose health hazards to consumers.

The QuEChERS (quick, easy, cheap, effective, rugged& safe) method is a technique for extraction has

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verified its utility. It has a great possible in the extraction of polar and particularly basic compound and for pesticide residue analysis in plant material, with excessive achievement. It has become the main analytical means in most pesticide monitoring laboratories, because it can simultaneously achieve high qualityresults for a wide range of pesticides and it presents practical benefits desired by all laboratories over most traditional methods of analysis (Lehotay*et al*., 2010;Camino-Sanchez *et al*., 2011). By applying QuEChERS to analyze of pesticides in the agricultural crops, matrix effects are removed and high recoveries of pesticides are possible. Acetonitrile used as the extraction solvent of the residues of pesticides in different food stuff, because the obtained extracts contain few interfering substances, additionally, acetonitrile can be separated fairly easily from water (salting out), therefore it is the preferred extraction solvent in the QuEChERSprocedure (Ankiewicz, 2019).

Many researchers have assessed the pesticide residues (PRs) in various fruits and vegetables including banana, mango, apple, peach, watermelon, grape, orange, lemon, pear, strawberry, kiwi fruit, beet, papaya, cabbage, spring tomato and white cabbage, (Kocoureke*t al*., 1998;Ortelli *et al.,* 2004;Gambacorta *et al.,* 2005;Ferrer and Thurman 2007;Zhang 2007; Boon *et al*., 2008;Kruve *et al.,* 2008; Gonzalez-Rodriguez *et al.*,2008 ; Chen *et al.*, 2009 ;Cunha *et al*., 2009;Hernandez-Borges *et al*., 2009 ;Knezevic and serdar 2009 ; and Banerjee *et al*.,2010)reported the amounts of pesticide residues to be even more than maximum residue level (MRL) values recommended by European Union (EU), World Health Organization (WHO) and Food and Agricultural Organization (FAO).

The main objective to assess the pesticide residues levels in the commonly consumed fruits and vegetables to compare these residues with the (EU) maximum residues limits.

MATERIALS AND METHODS

Samples collection

Samples of fruits and vegetables crops were taken from Egyptian local markets in great Cairo governorates during 2018.Three popular fruit crops and five popular vegetable crops and were used in this study.Apple (*Maluspumila);* grapes *(Vitisvinifera);* guava *(Psidiumguajava),* (cantaloupe) melon *(Benin casahispida);* cucumber *(Cucumissativus);* Squach *(Cucurbitapepo);* tomatoes *(Solanum.Lycopersicum)* and pepper *(Capsicum annuum).*For pesticide residue analysis 1-2 kg of each commodity was prepared according to Codex guidelines. Samples were stored at -18 ^{o}C before grinding, extraction and analysis. Samples were analyzed for 55 pesticides which included different groups as,organophosphorus, organochlorine, pyrethroids and others compounds.

Chemicals and reagents

All solvents used in thiswork were HPLC grade and supplied by Alliance Bio, USA. Primary and secondary amine (PSA, 40µmBondesil) was supplied from(Supelco, Bellefonte, USA). Analytical grade of anhydrous magnesium sulfate was supplied fromMerck, Darmstadt,Germany.Disodiumhydrogencitratesesquihydrate and trisodiumcitrate dehydrate (Sigma-Aldrich).Analytical grade of sodium chloride supplied from El-Naser Pharmaceutical Chemicals Company (Egypt).

Stock solutions of active ingredients.

Analytical standards of 55 tested pesticides with purity of 97.4-99.7% were supplied from Riedel-de Haen (Seelze, Germany) and Dr. Ehrenstorfer (Augsburg, Germany). Stock standard solutions (100 µg /ml) were prepared individually with ethyl acetate, acetone, n-hexane, methanol and acetonitrile according to their polarity and solubility. All standard solutions were stored in the dark at 4ºC.

Extraction and clean up

Extraction and clean-up processes were done by QuEChERS methods according to (EN 15662:2008).

The process of the extraction and cleanup summarized as,add of 10 g of the tested sample into a 50 ml centrifuge tube; 10 ml of acetonitrile (1% acetic acid), and the mixture shaked for 1 min. then added thesalt mixture which contain (4 g of anhydrous magnesium sulfate, 1 g of sodium chloride, 1 g oftrisodium citrate dehydrate and 0.5 g of disodium hydrogencitratesesquihydrate), and shakedthe mixture for another 1 min, and centrifuged the extract for 5 min at (> 3,000 rpm). Six ml of the supernatant was transferred to new clean 15 ml centrifuge tube containing 150 mg PSA and 900 mg anhydrous magnesium sulfate. The samples were shaked again for 1 min and then centrifuged for 5 min at (>4,000 rpm). Filtered 2 ml ofan aliquot through a 0.45 µm filter (Millipore, USA). The samples were ready to inject in Gas chromatography.

Quality Assurance procedures

All analytical methods and instructions were carefully validated as a part of the laboratory quality assurance system according to Document SANCO/12571/2013 (SANCO, 2013). Low level fortification (0.01-0.05 µg/g of all samples with the contaminants of interest has been carried out to ensure that the method performed satisfactory for the particular food examined. Analysis of triplicate of samples represents precision of analysis.

Determination of pesticides

Qualitative and quantitative determination of pesticide residues in food sample depends on using two different GC detectors (FPD and µECD). The injection standard technique was followed for the quantitative determination.µECD was used for organochlorine, pyrethroidsand halogenated compounds and FPDwas used for organophosphorus compounds.

GC-µECD Determination

Gas chromatograph HP 6890 with autosampler HP 7673 equipped with double micro electron capture detector (µECD) with capillary, column, (30 m x 0.32mmx0.25µm) 5% phenylmethylpolysiloxane (HP5).Injector temperature 280^{o}C, detector temperature 300^{o}C, flow rate of N_2(3ml/min). Temperature programs of GC was as follows; Initial temperature 190^{o}C for 5 min, rise 5^{o}C/min up to 220^{o}C and held for 5 min, then 5^{o}C/min up to 240^{o}C and held to 5 min, 10^{o}C/min up to 260^{o}C and held for 5 min, 10^{o}C/min up to 280^{o}C and held for 5 min

GC-FPD Determination

Gas chromatograph HP 6890 equipped with flame photometric detector (FPD) with capillary, column, (30 m x 0.32mmx0.25µm) PAS- 1701.Injector temperature 240^{o}C, detector temperature 250^{o}C, flow rate of N_2(4ml/min).Temperature programs was as follows; Initial temperature 160^{o}C for 2 min, then rise 6^{o}C/min up to 260^{o}C and held for 30 min.

RESULTS AND DISCUSSION

Quality assurance

Linearity of the method was determined by constructing calibration curves prepared by triple injection (n= 3) for each of the five concentrations of tested pesticides, i.e. 0.01 to 3.0 µg/ml. The calibration curve was plotted automatically.The method showed good linearity with determination coefficient (R^2) ranged 0.96-.0.99.The results showed that, the mean recoveries of the pesticides were between 81.20 and 98.70 %. The detectionlimits (LOD) and quantification limits of pesticide residues for the method were ranged (0.002 - 0.009 µg/g) and between 0.01 to 0.05 (µg/g), respectively. From these results the method was valid to estimate pesticide residues in fruit and vegetable (Table 1).

Table 1. Validation parameters to determine of pesticide residues in fruits and vegetables using GC-ECD/FID

No of samples.	Pesticides	Type of Pesticides	LOD (µg/g)	LOQ(µg/g)	Recovery (%)
1	Bromuconazole	F	0.002	0.01	93.50
2	Epoxiconazole	F	0.002	0.02	85.50
3	Diniconazole	F	0.006	0.01	94.45
4	Penconazole	F	0.004	0.02	93.00
5	Difenoconazole	F	0.002	0.01	97.20
6	Propiconazole	F	0.003	0.01	93.90
7	Tetraconazole	F	0.003	0.01	87.30
8	Triticonazole	F	0.003	0.01	89.80
9	Triflumizole	F	0.005	0.01	90.45
10	Dimethoate	I	0.005	0.02	89.90
11	Malathion	I	0.008	0.01	85.30
12	Profenofos	I	0.007	0.01	97.55
13	Triazophos	I	0.005	0. 03	93.60
14	Dichlorvos	I	0.007	0.02	86.15
15	Fenamiphos	I	0.008	0.01	81.65
16	Chlorpyrifos	I	0.004	0.05	93.63
17	Diazinon	I	0.007	0.01	90.95
18	Cadusafos	I	0.007	0. 02	81.40
19	Ethion	I	0.009	0.02	85.45
20	Pirimiphos Methyl	I	0.005	0.01	91.35
21	Prothiofos	I	0.005	0.01	87.45
22	Propetamphos	I	o.oo7	0.01	89.35
23	Chlorpyrifos-Methyl	I	0.005	0.01	86.45
24	Quinalphos	I	0.009	0.01	90.85
25	Azinphos-Methyl	I	0.007	0.02	90.95
26	Cypermethrin	I	0.002	0.01	81.20
27	Deltamethrin	I	0.001	0.01	87.65
28	Esfenvalerate	I	0.005	0.01	83.95
29	Permethrin	I	0.005	0.01	94.35
30	Tetramethrin	I	0.009	0.01	89.60
31	Fenarimol	F	0.006	0.01	94.00
32	Myclobutamyl	F	0.005	0.01	95.25
33	Triforine	F	0.009	0.01	82.15
34	Dicloran	F	0.007	0.01	84.90
35	Metribuzin	H	0.009	0.01	90.95
36	Dicofol	I	0.009	0.02	96.00
37	Bioallethrin	I	0.005	0.01	97.25
38	Hexythiazox	A	0.005	0.01	83.25
39	Clodinafop	H	0.008	0.01	95.00
40	Cyfluthrin	I	0.005	0.01	85.15
41	Lambda Cyhalothrin	I	0.003	0.01	94.90
42	Thiocyclam	I	0.006	0.01	95.30
43	Butralin	H	0.005	0.02	86.75
44	Pendimethalin	H	0.006	0.01	86.25
45	Fluazinam	F	0.005	0.01	84.40
46	Bifenazate	A	0.003	0.01	86.15
47	Proquinzid	F	0.007	0.01	82.30
48	Azoxystrobin	F	0.007	0.01	98.20
49	Chlorfenpyr	I,	0.003	0.01	86.20
50	Captan	F	0.008	0.01	91.00
51	Thiamethoxam	I	0.003	0.01	98.70
52	Acetochlor	H	0.007	0.05	90.90
53	Chlorfluazuron	I	0.005	0.01	87.95
54	Cyflufenamid	F	0.007	0.01	96.55
55	γ-Cyhalothrin	I	0.008	0.03	90.90

LOD, Limit of detection ,LOQ, Limit of quantificationF, Fungicide – I , Insecticide – H , Herbicide -A, Acaricide.

Determination of pesticide residues in different types of fruits and vegetables.

Pesticide residues details and the mean contamination ranges of different fruits (apple, grape sand guava) and vegetables (squash, cucumber, tomatoes, pepper and cantaloupe) collected from Egyptian local markets in Cairo Governorates are given in Table (2) and Fig (1) .It is clear from data, the results indicated that, the pesticide residues were found to be 32(58.2%), 31(56.4%) and 34(61.8%)) for apple, grapes and guava respectively. While for the ,squach, cucumber, tomatoes pepper and cantaloupe were found to be contaminated with 30(54.5%), 31(56.4%), 30(54.5%) 24(43.6%)and 27(49.1%), respectively. Also, 10 (18.2%) of the 55 pesticide, including, triticonazole ,triazophos, dichlorvos, cadusafos, propetamphos, quinalphos, azinphos-methyl, dichloran, butralin and acetochlor were not detected in any of different fruits and vegetables as shown in Table 2.

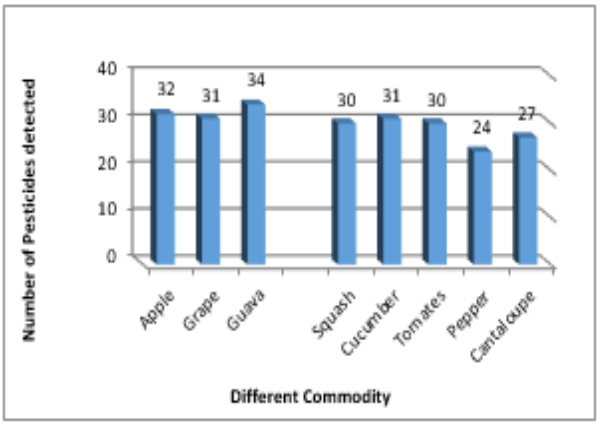

Fig. 1. Number of pesticides detected inthe different types of fruits and Vegetables

In Contrast, only 11 (20%) out of 55 pesticide including, triflumizole, cypermethrin, deltamethrin, fenarimol, myclobutamyl, hexythiazox, bifenazate, proquinzid, captan, thiamethoxam, and cyflufenamid were found in all different types of fruits (apple, grapes and guava) and vegetables (squach, cucumber, tomatoes, pepper and cantaloupe). Only three pesticides out of the 55 pesticides including prothiofos (0.25mg/kg), tetramethrin (0.01mg/kg) and bioallethrin (0.01mg/kg were found only in grape, guava and tomatoes, respectively. Difenoconazole was detected in different types fruits and vegetables except of apple, clodinafop was detected in different types (fruits and vegetables) except of pepper, tetraconzoale was detected in different types of fruits and vegetables except of pepper but lambda- cyhalothrin was detected in both fruits and vegetables except of pepper.

A total of 55 pesticide residues in different types of fruits subjected to analysis. The results showed that, in fruits 18 (32.72%) of the 55 pesticide residues were not detected in three different types of fruits. Also, 25 (45.45%) of the 55 pesticide residues were determined in all three different types of fruits. Moreover, 8 (14.2%) of the 55 pesticide residues were detected in only two different types of fruits. The results also indicated that 4 (7.3%) of the 55 pesticide residues were detected in only one type of fruits.

Of the 55 pesticide residues in different of vegetables subjected to analysis. Data revealed that, in vegetables 14 (25.54%) of the 55 pesticide residues were not detected in five different of vegetables. Also, 14 (25.54%) of the 55 pesticide residues were found in all five different of vegetables, while 7 (12.72%) of the 55 pesticide residues were detected in only four different types of vegetables, 8 (14.54%) of the 55 pesticide residues were detected in only three different types of vegetables, Furthermore,8 (14.54%) of the 55 pesticides were detected in two type of vegetables. Finally, 4 (7.3%) of the 55 pesticides were detected in only one type of vegetables (Table2).

The same results were previously obtained by (Srivastava*et al.*,2011) for 20 vegetables including leafy samples. They extracted their samples using the QuEChERS method, measured with a GC-ECD/NPD and obtained a recovery of 70-96% for the 35 pesticides analyzed. Also, The results of this study are consistent with the study carried out by (Dogheim *et al.*,2001), who analyze multi-residues of pesticides in 1579 samples of Egyptian fruits and vegetables collected from 8 local markets in a 6 governorates during 1996 for 53 pesticide residues including organophosphorus and organonitrogen compounds as well as some pyrethroids. They found that 23.9% of samples contained detectable residues, for individual crops contaminated samples ranged from 0 to 96% of the number of samples analyzed.

Also, (Nasiri*et al.*, 2016)reported that among the 60 analyzed Iranian cucumbers samples, 41.7% of them were contaminated with pesticide residues which 31.7% of the samples had pesticide residues lower than maximum residue limit and 10% of samples had residue higher than maximum residue limit. As well as (Reddy *et al*., 2011) reported that the insecticide residues in market samples of grapes were chlorpyriphos, monocrotophos, acephate, methamidophos and quinalphos. Moreover, the seresults are in accordance with those of (Yu *et al*., 2016**)** who monitored organophosphorus pesticides (OPs) in leafy vegetables in Changchun, China. Results of the study showed that7% of the total samples were free from pesticide residues, whereas 23% and 68% of the total samples contained OPs above and below the MRLs, respectively. Also, (Farg*et al*., 2011) who analyzed 141 pesticide residues in 31 orange samples from the local markets in Cairo , 22 (66.7%) of them were contaminated with 8 different pesticide residues. including fenpropathrin and chlorpyrifos, which were also detected in this work.

The maximum residue limits (MRLs) are defined as the highest concentrations of pesticide multi-residues (mg/kg) in fruits and vegetables. However, MRLs are not safety limits, and exposure to residues in excess of an MRL does not automatically imply a hazard to health (Sadto*etal*.2007). Also, (Anwar*et al.*, 2011**)** estimated the residues of seven organophosphate, three pyrethroid and two organochlorine pesticides in fruit samples purchased from the local markets of Nawabshah district by Gas Chromatography (GC). The results revealed that thepesticide residues of banana samples were below the determination limit of tested pesticides and the apple samples were exceeded the maximum residue limits (MRL) of Codex Alimentarius Commission.

The quality control of analytical methods considered one important factor for pesticide residues analysis. The quality control data of residues analysis in this study were in agreement with the findings of (Hadian *et al*. 2019) who determined forty-eight pesticide residues from different chemical structures including organochlorine, organo phosphorus, organo nitrogen, dicarboximides, strobilurin, triazine, pyrethroids, and other chemical groups in 85 fruits and vegetables samples. The pesticide was extracted with ethyl-acetate, then, the extracts cleaned using high performance gel permeation column chromatography (GPC) and solid phase column (SPE). The mean recoveries of the pesticides were between 81 and 136%.

Table 2. Concentrations pesticide residues in fruits and vegetables samples analyzed.

Detected pesticide	Residues in fruits commodity µg/g			Residues in vegetables commodity µg/g				
	Apple	Grapes	Guava	Squash	Cucumber	Tomatoes	Pepper	Cantaloupe
Bromuconazole	ND	0.02	0.01	0.02	0.01	ND	ND	ND
Epoxiconazole	ND	ND	ND	0.03	0.01	0.05	ND	ND
Diniconazole	0.01	0.01	0.01	ND	ND	ND	0.01	0.02
Penconazole	0.05	0.09	0.02	ND	ND	ND	ND	0.05
Difenoconazole	ND	0. 1	0. 1	0.01	0.05	0. 5	0. 10	0.01
Propiconazole	ND	ND	ND	0.01	0.01	0. 6	ND	ND
Tetraconzoale	0. 1	0. 1	0.01	0. 1	0.15	0.03	ND	0.02
Triticonazole	ND	ND	ND	ND	ND	ND	ND	ND
Triflumizole	0.44	2	0,03	0.09	0.1	0.06	0.01	0.05
Dimethoate	0.03	0.02	0.06	0.05	0.1	ND	0.21	ND
Malathion	0.04	0,1	0.05	0.01	ND	ND	0.02	0.01
Profenofos	0,01	0.06	0.01	ND	ND	2	0.10	0.01
Triazophos	ND	ND	ND	ND	ND	ND	ND	ND
Dichlorvos	ND	ND	ND	ND	ND	ND	ND	ND
Fenamiphos	0,01	ND	0.01	0.02	0.01	0,01	ND	ND
Chlorpyrifos	0.05	0.005	0.09	ND	ND	0.1	0.03	ND
Diazinon	0.02	0.04	ND	0.01	0.02	ND	0.02	0.01
Cadusafos	ND	ND	ND	ND	ND	ND	ND	ND
Ethion	0.05	ND	0.02	ND	ND	ND	ND	ND
Pirimiphos-Me	ND	ND	ND	0.02	0.03	0.05	0.01	ND
Prothiofos	ND	0.25	ND	ND	ND	ND	ND	ND
Propetamphos	ND	ND	ND	ND	ND	ND	ND	ND
Chlorpyrifos-Me	0.5	0.04	0.01	ND	ND	0.1	0.28	0.01
Quinalphos	ND	ND	ND	ND	ND	ND	ND	ND
Azinphos- Me	ND	ND	ND	ND	ND	ND	ND	ND
Cypermethrin	0.06	0.02	0.05	0.1	0,2	0.3	0.1	0.09
Deltamethrin	0.06	0.02	0.01	0.1	0.1	0.07	0.07	0.01
Esfenvalerate	0.03	0.5	0.02	0.01	0.01	0.03	ND	0.01
Permethrin	ND	ND	ND	0.02	0.01	0.03	0.01	ND
Tetramethrin	ND	ND	0.01	ND	ND	ND	ND	ND
Fenarimol	0.05	0.09	0.01	0.1	0.1	0.01	0.02	0.05
Myclobutamyl	0.01	0.02	0.02	0.04	0.01	0.03	0.5	0.05
Triforine	ND	ND	ND	0.01	ND	0.05	ND	ND
Diclooran	ND	ND	ND	ND	ND	ND	ND	ND
Metribuzin	ND	ND	ND	0.05	0.01	0.02	0.01	0.02
Dicofol	0.34	0.02	0.09	ND	0.11	ND	0.15	ND
Bioallethrin	ND	ND	ND	ND	ND	0.01	ND	ND
Hexythiazox	0.09	0-8	0.1	0.1	0.1	0.2	0.08	0.02
Clodinafop	0.01	0.02	0.02	001	0.01	0.01	ND	0.04
Cyfluthrin	0.01	0.02	0.01	ND	0.01	0.01	ND	ND
Lambda Cyhalothrin	0.01	0.01	0.03	0.01	0.02	0,01	ND	0.02
Thiocyclam	0.01	ND	0.01	ND	ND	ND	ND	0/01
Butralin	ND	ND	ND	ND	ND	ND	ND	ND
Pendimethalin	0.01	0.02	0.03	0,02	ND	ND	ND	0,01
Fluazinam	0.1	ND	ND	0.01	0.02	0,02	0.01	0.01
Bifenazate	0.2	0.4	0.03	0.02	0.2	0.07	0,5	0.01
Proquinzid	0.05	0.1	0.3	0.03	0.02	0.08	0.01	0.01
Azoxystrobin	ND	0,5	0.01	ND	0.5	0.03	ND	ND
Chlorfenpyr	001	ND	ND	ND	0.01	ND	ND	ND
Captan	2	0.02	0.01	0.02	0.01`	0.5	0.01	0.03
Thiamethoxam	0.25	0.2	0.01	0.3	0.3	0.1	0.4	0.1
Acetochlor	ND	ND	ND	ND	ND	ND	ND	ND
Chlorfluazuron	ND	0.01	001	0.02	0.01	ND	ND	0.01
Cyflufenamid	0.03	0.06	0.01	0.03	0.01	0.01	0.04	0.02
ɣ-Cyhalothrin	0.04	ND	0.03	ND	ND	ND	ND	ND

ND= Not detected

In this study, the data in Tables (3,4) and Fig. (2) illustrates the percentages of pesticide residues that exceeded the MRLs of fruits (apple, grape and guava) were (7)12.7%, (9) 16.4%, and (9)16.4% of the 55 pesticide residues, respectively and of vegetables (squash, cucumber, tomatoes ,pepper and cantaloupe) were (3) 5.5%, (4) 7.3%, (2) 3.6% (5), 9.1% and (2) 3.6% of the 55 pesticide residues, respectively. While, most of the pesticide residues for different types of fruits and vegetable were found blew the MRLs for apple, grape and guava were (48)87.3%, (46) 83.6%, and (46)83.6% and for squash, cucumber, tomatoes ,pepper and cantaloupe were (52) 94.6%, (51) 92.3%, (53) 96.4% (50),90.9% and (53) (96.4% of the 55 pesticide residues, respectively.

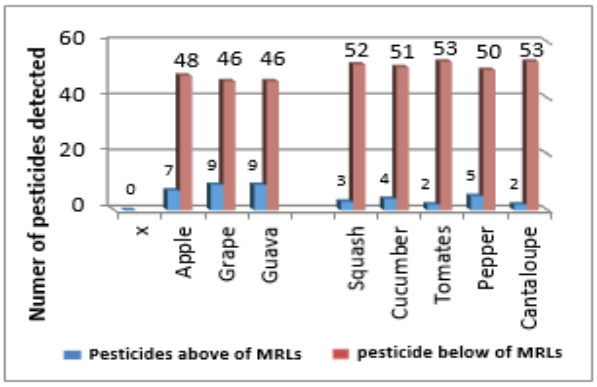

Fig .2. Number of pesticides above and below of MRLs in the different types of fruits and vegetables

Tables (3,4) revealed that, the results most detected pesticides in different of fruits and vegetables were below the (MRL). But, dimethoate, malathion, chlropyrifos, diazinon, ethion, dicofol and γ-cyhalothrin were exceeded the MRLs which detected in apple 12.7% (7 out of 55). In contrast, residues of tetraconazole, dimethoate, malathion, profenfos, diazinon, prothiofos, esfenvalerate, proquinzid and cyflufenamid were found to exceeded the MRLs in grape 16.4% (9 out of 55).On the other side, diniconazole and clodinafop were found above the MRLs in cantaloupe 3.6% (2 out of 55). In guava 16.4% (9 out of 55) of the detected pesticide residues including dimethoate, malathion, chlropyrifos ,ethion, dicofol, hexythiazox, lambda-cyhalothrin, binfenazate and proquinidexceeded the MRLs. In Table (2) and Table (3), a total of 5.5% (3/55) pesticide residues including dimethoate, chlorfluazuron and pirimiphos-methyl were detected at concentrations that exceeded the MRLs in squash. 7.3%(4/55) including dimethoate, diazinon, pirimiphos-methyl and dicofol of pesticide residues detected from cucumber exceeded the MRLs, 3.6% (2/55) pirimiphos-methyl and triforine were above the MRLs in tomatoes 5 out of 55 (9.09%) of the detected pesticide residues including difenconazole, dimethoate, chlorpyrifos, dicofol and cypermethrin exceeded the MRLs in pepper.

This is might be attributed to these pesticides being misused, overused or unnecessarily used by farmers who have very limited or no information about how to apply them or their health implications when present in foodstuffs and are just after a bumper harvest. In addition, the good agricultural practices (GAP) may not be well followed. Furthermore, a wrong pesticide choice made by the farmers for pest control in chard or wrong advice from pesticide retailers (Wang *et a*l., 2013).

Fruits and vegetables can be contaminated by pesticides used for the protection of their culture. The use of pesticides to control pests in fruits and vegetables can lead to the presence of pesticide residues (Ibrahim *et al*., 2018). The level of these residues can be below the maximum residue limit (MRL) if good agricultural practices (GAP) were used. The presence of residues with level exceeding MRLs should be interpreted as violation of GAP. In many reports, pesticide residues are present in the majority of fruits and vegetables; they are more detected in fruits than in vegetables. The percentage of exceeding MRLs is less than 20% in most monitoring programs (Mebdoua2018).

Table 3. MRLs of pesticides detected in fruits.

Commodity	Detected pesticide	MRL* (ppm)	Commodity	Detected pesticide	MRL (ppm)	Commodity	Detected pesticide	MRL (ppm)
	Diniconazole	0.01		Bromuconazole	0.50		Bromuconazole	0.05
	Penconazole	0.20		Diniconazole	0.01		Diniconazole	0.01
	Tetraconazole	0.30		Penconazole	0.4		Penconazole	0.05
	Triflumizole	0.5		Difenoconazole	3.0		Difenoconazole	0.10
	Dimethoate	0.01		Tetraconazole	0.5		Tetraconazole	0.02
	Malathion	0.02		Triflumizole	3.0		Triflumizole	0.10
	Profenofos	0.01		Dimethoate	0.01		Dimethoate	0.01
	Fenamiphos	0.02		Malathion	0.02		Malathion	0.02
	Chlorpyrifos	0.01		Profenofos	0.01		Profenofos	0.01
	Diazinon	0.01		Chlorpyrifos	0.01		Fenamiphos	0.02
	Ethion	0.01		Diazinon	0.01		Chlorpyrifos	0.01
	Chlorpyrifos-Me	1.0		Prothiofos	0.01		Ethion	0.01
	Cypermethrin	1.0		Chlorpyrifos-Me	1.0		Chlorpyrifos-Me	0.01
	Deltamethrin	0.20		Cypermethrin	0.5		Cypermethrin	0.05
	Esfenvalerate	0.10		Deltamethrin	0.2		Deltamethrin	0.01
	Fenarimol	0.10		Esfenvalerate	0.3		Esfenvalerate	0.02
Apple	Myclobutamyl	0.6	Grape	Fenarimol	0.3	Guava	Tetramethrin	0.01
	Dicofol	0.02		Myclobutamyl	1.0		Fenarimol	0.02
	Hexythiazox	1.0		Dicofol	0.02		Myclobutamyl	0.02
	Clodinafop	0.02		Hexythiazox	1.0		Dicofol	0.02
	Cyfluthrin	0.20		Clodinafop	0.02		Hexythiazox	0.50
	Lambda Cyhalothrin	0.08		Cyfluthrin	0.3		Clodinafop	0.02
	Thiocyclam	0.01		Lambda Cyhalothrin	0.08		Cyfluthrin	0.02
	Pendimethalin	0.05		Pendimethalin	0.05		Lambda Cyhalothrin	0.01
	Fluazinam	0.30		Bifenazat	0.7		Thiocyclam	0.01
	Bifenazate	0.70		Proquinzid	0.5		Pendimethalin	0.05
	Proquinzid	0.08		Azoxystrobin	3.0		Bifenazate	0.02
	Chlorfenpyr	0.01		Captan	0.03		Proquinzid	0.02
	Captan	10.0		Thiamethoxam	0.4		Azoxystrobin	0.01
	Thiamethoxam	0.30		Chlorfluazuron	0.01		Captan	0.03
	Cyflufenamid	0.05					Thiamethoxam	0.01
	γ-Cyhalothrin	0.01		Cyflufenamid	0.15		Chlorfluazuron	0.01
							Cyflufenamid	0.02

(EU, 2016).

Table 4. MRLs of pesticides detected in vegetables.

Commodity	Detected pesticide	MRL (ppm)	Commodity	Detected pesticide	MRL (ppm)	Commodity	Detected pesticide	MRL (ppm)
	Bromuconazole	0.05		Bromuconazo le	0.05		Epoxiconazole	0.08
	Epoxiconazole	0.05		Epoxiconazole	0.05		Difenoconazole	2.0
	Difenoconazole	0.30		Difenoconazole	0.30		Propiconazole	3.0
	Propiconazole	0.01		Propiconazole	0.01		Tetraconazole	0.10
	Tetraconazole	0.20		Tetraconazole	0.20		Triflumizole	1.0
	Triflumizole	0.20		Triflumizole	0.20		Profenofos	10.0
	Dimethoate	0.01		Dimethoate	0.01		Fenamiphos	0.04
	Malathion	0.02		Fenamiphos	0.02		Chlorpyrifos	0.10
	Fenamiphos	0.02		Diazinon	0.01		Pirimiphos-Me	0.01
	Diazinon	0.01		Pirimiphos-Me	0.01		Chlorpyrifos-Me	1.0
	Pirimiphos-Me	0.01		Cypermethrin	0.20		Cypermethrin	0.5.0
	Cypermethrin	0.20		Deltamethrin	0.20		Deltamethrin	0.07
	Deltamethrin	0.20		Esfenvalerate	0.02		Esfenvalerate	0.10
	Esfenvalerate	0.02		Permethrin	0.05		Permethrin	0.05
	Permethrin	0.05		Fenarimol	0.20		Fenarimol	0.02
Squach	Fenarimol	0.20	Cucumber	Myclobutamyl	0.20	Tomatoes	Myclobutamyl	0.30
	Myclobutamyl	0.20		Metribuzin	0.10		Triforine	0.01
	Triforine	0.01		Dicofol	0.02		Metribuzin	0.10
	Metribuzin	0.10		Hexythiazox	0.50		Bioallethrin	0.01
	Hexythiazox	0.50		Clodinafop	0.02		Hexythiazox	0.50
	Clodinafop	0.02		Cyfluthrin	0.10		Clodinafop	0.02
	Lambda Cyhalothrin	0.15		Lambda Cyhalothrin	0.05		Cyfluthrin	0.05
	Pendimethalin	0.05		Fluazinam	0.01		Lambda Cyhalothrin	0.07
	Fluazinam	0.01		Bifenazat	0.50		Bifenazate	0.50
	Bifenazate	0.50		Azoxystrobin	1.0		Proquinzid	0.15
	Proquinzid	0.05		Chlorfenpyr	0.01		Azoxystrobin	3.0
	Captan	0.03		Captan	0.03		Captan	1.0
	Thiamethoxam	0.50		Thiamethoxam	0.50		Thiamethoxam	0.20
	Chlorfluazuron	0.01		Chlorfluazuron	0.01		Chlorfluazuron	0.01
	Cyflufenamid	0.05		Cyflufenamid	0.04		Cyflufenamid	0.02

(EU, 2016)

Continued

Commodity	Detected pesticide	MRL (ppm)	Commodity	Detected pesticide	MRL (ppm)
	Diniconazole	0.01		Diniconazole	0.01
	Difenoconazole	0.80		Penconazole	0.10
	Triflumizole	0.10		Difenoconazole	0.20
	Dimethoate	0.01		Tetraconazole	0.05
	Malathion	0.02		Triflumizole	0.10
	Profenofos	0.10		Malathion	0.02
	Chlorpyrifos	0.01		Profenofos	0.01
	Diazinon	0.05		Diazinon	0.01
	Pirimiphos-Me	0.01		Chlorpyrifos-Me	0.01
	Chlorpyrifos-Me	1.0		Cypermethrin	0.20
	Cypermethrin	0.50		Deltamethrin	0.02
	Deltamethrin	0.20		Esfenvalerate	0.02
	Permethrin	0.05		Fenarimol	0.05
Pepper	Fenarimol	0.02	Cantaloupe	Myclobutamyl	0.20
	Myclobutamyl	0.50		Metribuzin	0.10
	Metribuzin	0.10		Hexythiazox	0.50
	Dicofol	0.02		Clodinafop	0.02
	Hexythiazox	0.50		Cyfluthrin	0.02
	Fluazinam	0.01		Lambda Cyhalothrin	0.06
	Bifenazate	3.0		Pendimethalin	0.05
	Proquinzid	0.02		Fluazinam	0.01
	Captan	0.03		Bifenazat	0.50
	Proquinzid	0.02		Proquinzid	0.02
	Thiamethoxam	0.70		Captan	0.03
				Thiamethoxam	0.15
	Cyflufenamid	0.04		Chlorfluazuron	0.01
				Cyflufenamid	0.04

(EU, 2016).

Finally, the results of this study shows that the concentrations of all the pesticides detected in both fruits and vegetables samples were found to be much lower than the MRLs. This implies that the samples are safe for human consumption as far as the investigated residues are concerned. However, they should be cleaned and washed properly. This is because washing of agricultural produce is known to reduce the levels of residues, which can be dissolved or physically dislodged from the raw product (Chin, 1991). It is recommended that there be continuous survey and monitoring programs for pesticides in food commodities in order to protect the end-user from indiscriminate exposure to pesticides.

CONCLUSION

This study provides scientific evidences of detected residues of many pesticides in the commonly fruits and vegetables taken from Egyptian local markets .The data are helpful for the risk assessment of consumers exposure to those pesticide residues. The QuECHERS sample preparation is suitable for determination of several classes of pesticide residues in matrices with high sugar content, more lipids and steroids contents. This method is considered as a flexible, sample preparation concept based on application of different solvents, salts, buffers and sorbents. This method also used in this study showed to be a simple, dynamic and effective procedure that can be run in any laboratory, since it eliminates slow and difficult steps used in other methods of pesticide extraction. The analytical procedure provides accurate results and it is applicable for routine analysis of many other fruits and vegetable matrices. The large number of pesticides belonging to different chemical can be analyzed.

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الكشف عن متبقيات 55من مبيدات الآفات في الخضروات والفواكه الشائعة في الأسواق المحلية المصرية

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يعتبر تقدير متبقيات المبيدات فى الخضروات والفاكهه من الأمور المهمة والتى تتعلق بالمستهلكين والغذاء الآمن. وفى هذه الدراسة تم رصد 55 من متبقيات المبيدات من مجاميع كيماوية مختلفة تشمل المبيدات الفوسفورية والفطرية والبيروثروويد والكلورونية فى مختلف الخضروات (كوسه – خيار – طماطم – فلفل– كنتالوب) والفاكهه (تفاح – عنب – جوافه) من الأسواق المحلية المصرية. حيث تم الأستخلاص وفقا لطريقة QuEChERS (وهى من أحدث الطرق المستخدمة فى الأستخلاص سريعة, سهلة, غير مكلفة, فعالة,مرنة وأمنة) مع استخدام جهاز الغاز الكروماتوجرفى المزود بكشاف قابض الالكترونات وكشاف طيف اللهب . وقدأوضحت النتائج المتحصل عليهاأن إجمالى متبقيات المبيدات التى تم رصدها 55 مبيد فى مختلف الخضروات والفاكهة وكان متوسط نسبة الاسترجاع يتراوح ما بين 81,20% الى 98,70 %. قدرت متوسطات حدود الكشف والتقدير فى مدى 0.002 الى 0.009 ميكروجرام / جرام و0.01 الى 0.05 ميكروجرام /جرام على الترتيب. وأوضحت النتائج من بين 55 متبقي مبيد تم رصدها فى الفاكهه والخضر كان 32 , 31 , 34 متبقى مبيد تم رصدها فى عينات التفاح و العنب و الجوافه على الترتيب بينما 30 , 31 ,30,24,27 متبقى مبيد تم رصدها فى الخضر (عينات كوسه و خيار و طماطم و فلفل و الكنتالوب على الترتيب). وكانت نسبة الحد الاقصى لمتبقيات المبيدات التى تم رصدها فى الفاكهه والخضر فكانت نسبة المبيدات الاعلى من من الحد الاقصى فى عينات التفاح والعنب الجوافه 12,7 و 16,4و 16,4 % على الترتيب بينما فى الخضر كانت نسبة متبقيات المبيدات الاعلى من الحد المسموح به 5,5 ,7,3 ،3,6, 9.1, 3,6% فى عينات كوسه و خيار و طماطم و فلفل و الكنتالوب على الترتيب من اجمالى 55 متبقى مبيد تم تقدرهم فى الخضر والفاكهة.