



Monitoring of Pesticide Residues in Imported Fruits and Vegetables in United Arab Emirates during 2019

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Authors' contributions

This work was carried out in collaboration among all authors. Author NMAEM designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Authors IIAA and ASJ managed the analyses of the study. Author ASJ managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

An accurate, rapid and reliable method for the simultaneous determination of pesticide multi-residues in 9724 samples imported fresh fruits and vegetables by a method of liquid chromatography coupled with tandem mass spectrometry LC-ESI (+)-MS/MS operating in multiple reaction monitoring (MRM) mode, two transitions for each compound in the presence of internal standards and modified quick, easy, cheap, effective, rugged, safe (QuEChERS) method was used for the determination of 343 pesticides.

The performance of the analytical method was validated in accordance with EU SANCO guidelines (SANTE/12682/2019) for monitoring pesticide multi-residues. Residues of 93 compounds, mainly fungicides and insecticides, were detected in 3548 samples.

In this article, The potential applications of Liquid Chromatography and Tandem Mass spectrometry (MS/MS) techniques on monitoring pesticide residues of target samples of imported fruits and vegetables in United Arab Emirates (UAE) during the year 2019 is reported. The need for a continuous monitoring program of pesticide residues in imported fruits and vegetables is highly recommended and continuous process control for health and safety practices and to adhere to international agricultural and environmental policies of the country.

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1. INTRODUCTION

Vegetables and fruits are important components of human diet since they supply essential nutrients that are required for many of the biochemical reactions occurring within the physical body. Like other crops, fruits and vegetables are attacked by pests and diseases during production and storage, leading to damages that reduce the quality and the harvest. In order to reduce the loss and maintain the quality of fruits and vegetables harvest, pesticides are used [1]. However, the use of pesticides during production often leads to the presence of pesticide residues after harvest [2].

Consumers are concerned about pesticide residues on fresh fruits and vegetables because some pesticides have been associated with some health hazards such as headaches and nausea to chronic impacts like cancer, reproductive harm and endocrine disruption [3].

Governments and international organizations regulate, and monitoring processes exist to prevent or minimize such adverse health effects. Additionally, there has been widespread concern of the maximum residue limits (MRLs) and total dietary intakes of pesticide residues in food commodities [4,5]. The MRL is that the maximum amount of pesticide or related residues (metabolites and coadjutants) that is still officially in food, due to the correct application during a specific phase, from its production to consumption. Therefore, MRL values represent the maximum concentration of pesticide residue that is legally allowed in food products, and thus, can be legally marketed. The MRL is expressed in parts of the pesticide (by weight), or their residues per million parts of feed (by weight) (ppm or mg.kg^{-1}) [3]. MRLs have been established for agricultural products in many countries to avoid the hazard caused by pesticide residues.

The monitoring and evaluation of the impacts in the use of pesticides should be seen as vital activities, ensuring the sustainability of the agricultural production systems that use pesticides, in addition to ensuring compliance

with regulations [6], to assure that the applications of pesticides be made according to the proposed good agricultural practices (GAP) [6,7], resulting in a safe product for the consumer [8,9]. If GAP is applied efficiently, the number of pesticide residues will be below the corresponding to the maximum residue level [9].

According to WHO, on average, 30% of the diet consists of fruits and vegetables [10,11], being consumed mainly raw or semi-processed [10], and consequently, they are expected to contain higher levels of pesticide residues [10-12] in comparison with other food groups of animal origin [11]. It is impossible to eliminate pesticide residues from the vegetables' internal parts, due to the pesticide's ability to penetrate inside of the leaves and pulps [13,14]. Studies have indicated that regulation of pesticide maximum residue limits (MRLs) in commodities is established but not fully enforced in many countries [15,16].

Reliable residue analysis data resulting from monitoring programs in foods, even if limited, could also be of great value indicating the possible risks of pesticide exposure on human health and on international trade. Such data could help decision makers in reviewing and reconsidering the registration and use of pesticides in the country.

The UAE imports a substantial and increasing portion of its fruits and vegetables. The National Laboratories of Ministry Climate Change and Environment inspects import shipments. Expecting from exporting countries to comply allowable pesticide residue levels in UAE. This monitoring programs implemented in UAE are carried out by the Ministry of Climate Change and Environment, the purposes are to check whether residues found in imported fruits and vegetables are compliance with national MRLs. And the findings will provide scientific evidence for UAE agriculture.

2. MATERIALS AND METHODS

Reagents and chemicals: Certified analytical standards were purchased from Dr Ehrenstofer (Germany), with purity between 92.0 and 99.5%.

Acetonitrile (Merk, Germany), methanol (LC-MS grade, Scharlab), Formic acid (Honeywell, Germany).

Ready-made QuEChERS kits were purchased from Supelco; Supel™ QuE citrate extraction tube contains 4.0 g MgSO₄, 1.0 g NaCl, 0.5 g sodium Citrate dibasic sesquihydrate, 1.0 g Na Citrate tribasic dehydrate. Supel™ QuE PSA/ENVI-CARB (EN) tube 2 contains 150.0 mg Supelclean PSA, 45.0 mg Supelclean ENVI-Carb, 900.0 mg MgSO₄. The solutions were prepared with Ultrapure demineralized water Milli-Q plus system (Merck-Millipore Corporations, USA).

Standard preparation: Individual analytical stock solutions (1000 mg L⁻¹) of each pesticide were prepared in methanol, considering the purity of each pesticide standard. These analytical solutions were diluted in methanol to 100 mg L⁻¹. All solutions were stored in amber flasks at -18°C. Afterwards, a mixture with the concentration of 10 mg L⁻¹ containing all pesticides was prepared, that was diluted to 1 mg L⁻¹ and was kept in refrigerator at 4.0 ± 2.0°C

Internal standard solution: A solution of Triphenyl phosphate (TPP), analytical grade, was used as an internal standard.

Apparatus: Agilent 6460 triple quadrupole, on a reversed-phase column and detected by tandem mass spectrometry (MS/MS) using electrospray ionization (ESI). The pesticide residues determined with positive ESI only. The total chromatographic run time was 32 minutes. Injection volume was 2.0 µL and the column temperature was set at 60°C. The Agilent Mass Hunter Workstation software B.04.00 Features was used for data analysis. All pesticides were detected in the multiple reaction monitoring modes (MRM). Each pesticide has precursor ion there were two product ions determined. One product ion used for quantification and other one was used for qualification, detected pesticides are shown in Table1.

Samples collection: The pesticide residue random monitoring program aimed to take and analyse various samples of fresh vegetables and fruits, and a total of 9724 samples were collected from January to December 2019 from imported consignments in all over UAE Emirate's ports.

The percentage of vegetable samples taken and analysed reached 44.66% (4343 samples) of the

total samples, and the percentage of the fruit taken and analysed 55.33% (5381 samples). An illustrates commodity groups and representative commodities vegetables and fruits shown in Table 2. Vegetable group, 4343 samples included Potatoes, Tomatoes, Cucumbers, Squash, Eggplant, Okra, Lettuce, Parsley, mint, Carrot, Beans, Garlic, Onions, Coriander, Spinach, pepper while Fruit group, 5381 samples included Dates, Grapes, Melons, Oranges, Lemons, Bananas, Pomegranate, Guava, Apple, Mango, Strawberry, Peach, Apricot as shown in Table 2.

Samples took by the Ministry's inspectors in accordance with the general principles and standard specifies the method of sampling fresh fruits and vegetables UAE's GSO 125:1990 for establishing MRLs in food commodities. Each representative vegetable or fruit sample was a composite of 10 subsamples of the same commodity collected through random sampling. All the samples (1–2 kg each) were placed in sterile polythene bags, in an ice chest box, to avoid contamination, deterioration, labelled, and transported to the laboratory under appropriate transport conditions within 24 hours for analysis.

Sample preparation: The used sample preparation method was developed at the National Laboratories Department, Ministry of Climate Change and Environment (MOCCAE), which is accredited by UKAS (The United Kingdom Accreditation Service) according to ISO 17025:2017 (International Organization for Standardization, 2017) for the analysis of pesticides in several foodstuffs. Inedible parts are removed from the samples, and the vegetable and fruit samples are mixed, homogenized for analysis. Blank samples (Pesticide free samples) were acquired from the consumer market used for validation experiments. The samples were stored at ambient temperature (20°C) till withdraw for analysis.

The pesticide residues were analysed using the method approved by the European Union to determine the level of Pesticides in food products QuEChERS method reported by Anastasiadis et al. [17] by using LCMSMS (liquid chromatography tandem mass spectrometry) the following sample extraction and clean up steps were conducted as follows.

Table 1. List of studied pesticides

Abamectin	Carbaryl	Demeton S methyl sulfone	Etoxazole	Fuberidazole	Metconazole	Phenmedipham	Quinoxyfen	Tri-allate
Acenaphthene	Carbendazim	Demeton-S-methyl	Etrimfos	Furalaxyl	Methabenzthiazuron	Phenthoate	Quizalofop-ethyl	Triazamate
Acephate	Carbetamide	Desmedipham	Famoxadone	Furathiocarb	Methidithion	Phosalone	Rotenone	Triazophos
Acetamiprid	Carbofuran	Diazinon	Fenamidone	Halfenprox	Methiocarb	Phosmet	Secbumeton	Trichlorfon
Acibenzolar S methyl	Carbofuran-3-Hydroxy	Dichlofluanid	Fenamiphos	Halofenozide	Methiocarb sulfone	Phosphamidon	Siduron	Tricyclazole
Acrinathrin	Carboxin	Dichlorvos	Fenamiphos sulfone	Heptenophos	Methomyl	Picolinafen	Silthiofam	Tridemorph
Alachlor	Chlorantraniliprole	Diclobutrazol	Fenarimol	Hexaconazole	Methoprotryne	Picoxystrobin	Simazine	Trifloxystrobin
Alanycarb	Chlorfenapyr	Dicloran	Fenazaquin	Hexaflumuron	Methoxychlor, o,p'-	Piperonyl butoxide	Simetryn	Triflumizole
Aldicarb	Chlorfenvinphos	Dicrotofop (Dicrotophos)	Fenbuconazole	Hexazinone	Methoxychlor, p,p'-	Pirimicarb	Spinetoram	Triflumuron
Aldicarb sulfone	Chlorfluazuron	Dicrotophos	Fenhexamid	Hexythiazox	Methoxyfenozide	Pirimiphos-ethyl	Spinosad	Trifluralin
Aldicarb sulfoxide	Chloridazon	Diethofencarb	Fenitrothion	Hydramethylnon	Metobromuron	Pirimiphos-Methyl	Spiromesifen	Triticonazole
Ametryn	Chlorobenzilate	Difenoconazole	Fenoxycarb	Imazalil	Metribuzin	Prochloraz	Spirotetramat	Uniconazole
Aminocarb	Chlorothalonil	Diflubenzuron	Fenpiclonil	Imidacloprid	Mevinphos	Procymidone	Spiroxamine	Vamidothion
Amitraz	Chlorotoluron	Diflufenician	Fenpropathrin	Indoxacarb	Mexacarbate	Profenofos	Sulfentrazone	Vinclozolin
Atrazine	Chloroxuron	Dimethenamid	Fenpropimorph	Ipconazole	Molinate	Promecarb	Sulfotep	Zoxamide
Azaconazole	Chlorpropham	Dimethoate	Fenpyroximate	lprobenfos	Monocrotophos	Prometon	Tebuconazole	Triticonazole
Azinphos ethyl	Chlorpyrifos	Dimethomorph	Fenthion	lprodione	Monolinuron	Prometryne	Tebufenozide	Uniconazole
Azinphos methyl	Chlorpyrifos-methyl	Dimoxystrobin	Fenthion sulfoxide	lprovalicarb	Monuron	Propachlor	Tebufenpyrad	Vamidothion
Azoxystrobin	Chlorthal-dimethyl	Diniconazole	Fenuron	Isocarbophos	Moxidectin	Propamocarb	Tebutam	Vinclozolin
Beflubutamid	Clethodim	Dinotefuran	Fenvalerate	Isofenphos	Myclobutanil	Propanil	Tebuthiuron	
Benalaxyl	Clodinafop-propargylester	Dioxacarb	Fipronil	Isoprocarb	Napropamide	Propaquizafop	Tecnazene	
Bendiocarb	Clofentezine	Dioxathion	Flamprop-methyl	Isoproturon	Neburon	Propargite	Teflubenzuron	
Benfuracarb	Clomazone	Diphenylamine	Flonicamid	Isoxadifen-ethyl	Nitenpyram	Propazine	Tefluthrin, cis-	

Abamectin	Carbaryl	Demeton S methyl sulfone	Etoxazole	Fuberidazole	Metconazole	Phenmedipham	Quinoxyfen	Tri-allate
Benomyl	Clothianidin	Diuron	Fluazifop-butyl	Ivermectin	Norflurazon	Propetamphos	Temephos	
Benzoximate	Coumaphos	DMST	Fluazifop-p-butyl	Kresoxim-methyl	Novaluron	Propham	Terbumeton	
Bifenazate	Cyanazine	Doramectin	Fluazinam	Lenacil	Nuarimol	Propiconazole	Terbutryne	
Bifenthrin	Cyazofamid	Emamectin	Flubendimide	Linuron	Ofurace	Propoxur	Tetrachlorvinphos	
Bitertanol	Cycloxydim	Endosulfan -beta isomer	Fluidioxonil	Lufenuron	Omethoate	Propyzamide	Tetraconazole	
Boscalid	Cycluron	Endosulfan sulfate	Flufenacet	Malaoxon	Oxadiazon	Proquinazid	Tetradifon	
Bromacil	Cyfluthrin I	Endosulfan-alpha isomer	Flufenoxuron	Malathion	Oxadixyl	Prosulfocarb	Tetramethrin	
Bromopropylate	Cyhalothrin (lambda)	EPN	Fluometuron	Mandipropamid	Oxamyl	Prothiofos	Thiabendazole	
Bromoxynil	Cymoxanil	Epoxiconazole	Fluoxastrobin	Mecarbam	Oxyfluorfen	Pymetrozine	Thiacloprid	
Bromuconazole	Cypermethrin	EPTC	Fluquinconazole	Mefenacet	Paclobutrazol	Pyracarbolid	Thiamethaoxam	
Bupirimate	Cyproconazole	Ethiofencarb	Flusilazole	Mefenpyr-diethyl	Paraoxon ethyl	Pyraclostrobin	Thidiazuron	
Buprofezin (Z-isomer)	Cyprodinil	Ethion	Flutolanil	Mepanipyrim	Paraoxon methyl	Pyraflufen-ethyl	Thiobencarb	
Butafenacil	Cyromazine	Ethiprole	Flutriafol	Mepronil	Parathion-methyl	Pyrazophos	Thiodicarb	
Butocarboxim	DDD-p,p'	Ethirimol	Folpet	Mesotrione	Penconazole	Pyridaben	Thiophanate-methyl	
Butoxycarboxim	DDE-p,p'	Ethofenprox	Foramsulfuron	Metaflumizone	Pencycuron	Pyrifenox	Tolclofos-methyl	
Buturon	DDT-o,p'	Ethofumesate	Forchlorfenuron	Metalaxyl	Pencyuron	Pyrimethanil	Tolyfluanid	
Cadusafos	DDT-p,p'	Ethoprophos	Formetanate	Metamitron	Pendimethalin	Pyriproxyfen	Triadimefon	
Cafentrazone ethyl	Deltamethrin	Ethoxyquin	Fosthiazate	Metazachlor	Permethrin	Pyridaphenthion	Triadimenol	

Ten grams of each samples were homogenized and dissolve in 10.0 ml of acetonitrile and shake vigorously for one minute. A buffer solution - salt mixture made by adding (1.0 g sodium chloride, 1.0 g disodium hydrogen citrate sesquihydrate, 0.5 g trisodium citrate dehydrate, and 4.0 g magnesium sulphate anhydrous) to the homogenized sample, stirred vigorously for 60 s. The organic phase solution was separated from the inorganic phase solution after centrifugation at 5000 rpm for 5 minutes. Transfer 10.0 mL of the upper extract clear solution into a single-use polypropylene centrifuge tube containing 150 mg Supelclean PSA, 45.0 mg Supelclean ENVI-Carb, 900 mg of magnesium sulphate ($MgSO_4$) vortex the mix and centrifuge it for 5 min at 5000 rpm. Evaporate one ml of the supernatant solution to complete dryness using vacuum concentrator at 40°C. Finally, the residues of pesticide were re-dissolved in 1 ml of deionized water: acetonitrile (9:1) and filtered using at PTFE syringe filter 0.22 µm, the reconstituted sample (2 µL) was then injected into a Liquid chromatograph coupled to tandem mass spectrometry (LC-MS/MS).

In-house method validation: The selected parameters for in-house validation in compliance with the document SANTE/12682/2019 [18] “Guidance document on analytical quality control and method validation procedures for pesticide residue analysis in food and feed” which was issued by the European Commission Directorate General for Health and Food Safety, and became effective on January 1, 2020. The reliability of the method was evaluated by estimating the accuracy (expressed as recovery percentage) and precision (% relative standard deviation): The main goal of the recovery experiments is to determine the method accuracy, via comparison of the real concentration of each pesticide measured by performing the complete procedure with the known pesticide concentration initially added to the matrix. The method precision is expressed as the repeatability (RSD%) of the recovery determinations at the two different spiking levels (0.01 and 0.1 mg/kg). Five spiked samples were analysed at each level and the blank. Recovery range of about 75-105% with corresponding relative standard deviations <20% is deemed satisfactory in pesticide residue analysis. Guidance documents for monitoring of pesticide residue analysis within the European Union (SANTE/2019) set mean recoveries for initial validation in the range of 70-120%.

3. RESULTS AND DISCUSSION

Monitoring pesticide residues in fruit and vegetables remains a key priority for UAE food safety. Increasing imports from some countries, with substantially different regulations in these countries, highlights the need for stringent pesticide monitoring.

During the year 2019, 9724 samples of imported fresh vegetables and fruits were taken and analysed from all shipments all over UAE Emirate's ports. The results showed that 93.75% of the samples conformed to the approved standard specifications, while 6.25% of the samples exceeded the permissible limits for pesticide residues as shown in Table 2.

Ministry of Climate Change and Environment, represented by the National Laboratories Department, conducted survey to monitor pesticide residues in imported fresh vegetables and fruits by collecting samples from imported consignments at all shipments all over UAE Emirate's ports to ensure that pesticide residues (if any) not exceed the maximum permissible limit for these products according to the regulations, specifications and circulars in force.

The sampling and analysis of samples for monitoring pesticide residues was completed in 2019, and the results were compared to the maximum permissible limits (MRL) for pesticide residues in the UAE standard (maximum limits for pesticide residues in agricultural and food products) issued by Emirates Authority for Standards and Metrology (ESMA), UAE Standards UAE.S 19:2016, and the list of maximum limits for pesticides residues in the Codex Alimentarius Commission (Codex) and European specifications. (<https://ec.europa.eu/food/safety>).

3.1 Pesticide Residues in Analysed Fruits and Vegetables Samples

The percentage of samples free of pesticide residues were 63.69%, while the percentage of samples containing pesticide residues higher (EU MRL or Codex MRL) than the permissible limit 6.47% (607 sample) of the total samples established by EU or Codex [19] Table 2.

The results showed that vegetables samples contained pesticide residues exceeding permissible limits by 7.52%, while fruits group

contained pesticide residues exceeding permissible limits by 5.43% as shown in Table 2.

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It is reported that total percentages of pesticide residues in fruits and vegetables in the United Kingdom, kingdom of Saudi Arabia [20] and Australia are higher than European Union [21] and United State of America is lower than those countries as shown in Table 3 [22].

No pesticide residues were detected in any of the radish, yam, cassava and leek samples. It was found that (16) ginger samples (59.2%) from the total examined samples contains residues above the permissible limit, followed by the curry leaves (57.4%). Whereas, beetroot (1.8%) of the samples contained pesticide residue above the MRLs as in Table 4.

Vegetable samples represent the lowest percentage of the total number of samples taken in monitoring pesticide residues, as it reached 44.66% of the total number of samples. Where 4343 samples of vegetables were taken from Table 2.

The fruit group samples constituted 55.33% of the total number of samples, as 5381 samples were taken from fruit. Table 5.

The results showed that 4028 samples (92.74%) were in compliance with the specifications approved by the monitoring program, 2842 of which a sample (65.43%) was free from pesticide residues, while the number of samples containing pesticide residues did not exceed the maximum permissible limit of 1186 samples (27.31%), and 315 samples (7.52%) contained pesticide

The results of the analysis showed that the percentage of samples conforming to the specifications is 94.57%, the percentage of samples free from pesticide residues was 61.96%, while 5.43% of samples had pesticide residues slightly exceeding the permissible limits, however; few selected fruits samples such as Grapefruit (80.8%), Mandarins (86.2%), Orange (85.5%), pomelo (84.6%), Apricots (80.8%) and Lemon (87%) showed higher percentage of contaminated as shown in Table 5.

Table 2. Overview of the results of monitoring pesticide residues in vegetable and fruit samples

Commodities' group	Total	Samples free of pesticide residues		Samples containing pesticide residues within MRL		Samples containing pesticide residues above MRL	
		number	%	number	%	number	%
Vegetables	4343	2842	65.43	1186	27.31	315	7.52
Fruits	5381	3334	61.96	1755	32.61	292	5.43
Total	9724	6176	63.69	2941	29.96	607	6.47

Table 3. Percentages of samples exceeding the permissible limits in the programs for monitoring pesticide residues in food in some countries

Country	Percentage above MRL	The Program	Preparation by	Date
kingdom of Saudi Arabia	8.71	report of the National Pesticide Residue Monitoring Program performed	Saudi Food and Drug Authority	2019 (20)
European Union	7.2	European Union report on pesticide residues in food	European Food Safety Authority	2016
United Kingdom	4.25	Pesticide Residues in Food (PRiF) Annual Report 2016.	The Expert Committee on Pesticide Residues in Food (PRiF) 2016	2016
United State of America	9.8	Pesticide Residue Monitoring Program	U.S. Food and Drug Administration	2016

Table 4. Summary of the vegetable's samples used, (Pesticide free samples)

Vegetables	Samples	Number of samples	Samples with residues above MRLs		Samples within MRL		Samples free	
			No	Rate %	No	Rate %	No	Rate %
Leafy vegetables	Coriander	43	5	11.63	6	13.95	32	74.41
	Chard	7	0	0.00	2	28.57	5	71.42
	Parsley	108	32	29.63	15	13.89	61	56.48
	Spinach	9	1	11.11	2	22.22	6	66.66
	Watercress	14	4	28.57	0	0.00	10	71.42
	Lettuce	298	21	7.05	122	40.94	155	52.01
	Radish leaves	23	0	0.00	0	0.00	23	100
	Mint	20	1	5.00	7	35.00	12	60.00
Fruiting Vegetables, Cucurbits	Courgette	323	19	5.88	123	38.08	181	56.03
	Cucumbers	112	8	7.14	38	33.93	66	58.92
	Melons	326	17	5.21	111	34.05	198	60.73
	Pumpkins	27	3	11.11	3	11.11	21	77.77
	Squash	23	0	0.00	2	8.72	21	91.30
	Gourd	114	6	5.26	22	19.3	86	75.43
Fruiting Vegetables, other than Cucurbits	Capsicums	256	39	15.23	121	47.27	96	37.50
	Cherry tomato	12	0	0.00	3	25.00	9	75
	Eggplant	79	2	2.53	22	27.85	55	69.62
	Tomato	698	38	5.44	302	43.27	358	51.28
	Pepper	175	28	16.00	63	36.00	84	48.00
	Okra	28	1	3.57	5	17.86	22	78.57
Root and Tuber Vegetables	Potato	179	8	4.47	9	5.03	162	90.50
	Carrots	231	11	4.76	31	13.42	189	81.81
	Cassava	3	0	0.00	0	0.00	3	100.00
	Taro	47	2	4.26	2	4.26	43	91.48
	Yam	7	0	0.00	0	0.00	7	100.00
	Turnip	22	0	0.00	2	9.09	20	90.90
	Beetroot	54	1	1.85	3	5.56	50	92.59
Brassica	Cauliflower	153	4	2.61	21	13.73	128	83.66
	Cabbage	329	14	4.26	65	19.76	250	75.98
	Broccoli	28	0	0.00	5	17.86	23	82.14
Bulb vegetables	Garlic	145	4	2.76	3	2.07	138	95.17
	Onion	225	17	7.56	16	7.11	192	85.33
	Leek	2	0	0.00	0	0.00	2	100.00
	Fennel	3	1	33.33	1	33.33	1	33.33
Legume vegetables	Peas	53	2	3.77	3	5.66	48	90.56
	Beans-Green	114	5	4.39	42	36.84	67	58.77
Stalk and stem vegetables	Artichoke	2	0	0.00	1	50.00	1	50.00
	Celery	17	1	5.88	5	29.41	11	64.70
Herbs and Spices	Curry leaves	7	4	57.14	2	28.57	1	14.28
	Ginger rhizomes	27	16	59.26	6	22.22	5	18.51
Total		4343	315	7.52	1186	27.31	2842	65.43

3.2 MRL Exceedances of Pesticides in Analysed Samples

Of the 343 pesticides (including metabolites) studied, 93 pesticides were detected above the MRLs in the analyzed fruit and vegetable samples as in Table 6 and 7.

Data in Table 6 showed that 58 pesticides were detected in the fruits samples, 26 of which are registered in the United Arab Emirates, 14 are not Registered and not restricted pesticides, while 18 banned pesticides were found according to the list of the Ministry of Climate Change and Environment.

While in vegetables samples data showed that 79 pesticides were detected, 30 of which are registered in the United Arab Emirates, 15 are not Registered and not restricted pesticides, while 34 banned pesticides were found according to the list of the Ministry of Climate Change and Environment as shown in Table 7.

In general, as mentioned above the list of disclosed pesticides in this monitoring were contained 93 pesticides distributed into three lists registered, not registered and banned, the common detected pesticides above MRL's, 34 pesticides.

It is banned in the UAE and also in one or more international organizations, and it must not be present in food that is higher than the permissible limits. Moreover; when considering non-banned pesticides registered in UAE, compared to monitored pesticides samples, in this study the permissible limits have been limited to 21 pesticides shown in Tables 6 and 7.

In all monitoring fruits samples, 24.13% and 31.03 % from detected pesticides above MRL's were unregistered and banned pesticides, respectively. While in vegetables samples, 18.98 and 43.03 % from detected pesticides above MRL's were unregistered and banned pesticides, respectively.

Table 5. Summary of pesticides residues in fruits samples, (Pesticide free samples)

Fruits	Samples	Number of samples	Samples with residues above MRLs		Samples within MRL		Samples free	
			No	Rate %	No	Rate %	No	Rate %
Pome Fruits	Apple	885	4	0.45	225	25.42	656	74.12
	Pear	294	2	0.68	150	51.02	142	48.30
Stone fruits	Apricots	47	4	8.51	34	72.34	9	19.16
	Nectarine	43	4	9.30	17	39.53	22	51.16
	Peach	97	12	12.37	41	42.27	44	45.36
	Plum	112	2	1.79	37	33.03	73	65.18
	Prunes	1	0	0.00	0	0.00	1	100.00
Tree nuts	Coconut	38	0	0.00	3	7.89	35	92.11
Berries and other small fruits	Berries	15	3	20.00	6	40.00	6	40.00
	Grapes	294	15	5.10	160	54.42	119	40.48
Citrus fruits	Grapefruit	59	4	6.78	47	79.66	8	13.56
	Orange	444	41	9.23	340	76.58	63	14.19
	Pomelo	39	23	58.97	10	25.64	6	15.38
	Lemon	263	24	9.13	162	61.60	77	29.28
	Mandrins	254	33	12.99	186	73.23	35	13.78
Assorted tropical and sub-tropical fruits - inedible peel	Avocado	32	0	0.00	3	9.38	29	90.63
	Papaya	2	0	0.00	1	50.00	1	50.00
	Banana	197	6	3.05	60	30.46	131	66.50
	Kiwifruit	109	2	1.83	3	2.75	104	95.41
	Mango	289	10	3.46	34	11.76	245	84.78
	Guava	51	10	19.61	3	5.88	38	74.51
	Litchi	5	0	0.00	0	0.00	5	100.00
	Pineapple	144	0	0.00	105	72.92	39	27.08
	Persimmon	24	3	12.50	0	0.00	21	87.50
	Pomegranate	220	16	7.27	51	23.18	153	69.55
Assorted tropical and sub-tropical fruits - edible peel	Durian	3	1	33.33	0	0.00	2	66.67
	Fig	36	0	0.00	3	8.33	33	91.67
	Dates	1384	73	5.27	74	5.35	1237	89.38
Total		5381	292	5.43	1755	32.61	3334	61.96

As shows in Table 6 in fruits samples, most of the residues in banned pesticides above MRL (118 samples) found in positive samples analyzed were had level between 0.03 and 32 mg/kg and the majority of the positive pesticides residues of Imazalil and Thiophanate-methyl had residue levels 32.0 and 15.0 mg/kg respectively.

While in non-registered pesticides samples (22 samples) above MRL were had level between 0.018 and 17.0 mg/kg and the majority of the positive pesticide's residues of Thiabendazole and Dimethomorph had residue levels 17.0 and 4.1 mg/kg, respectively.

Also in Table 7, in vegetables samples, most of the residues in banned pesticides above MRL (34 samples) found in positive samples analyzed were had level between 0.029 and 21.9 mg/kg and the majority of the positive pesticides residues of Triazophos and Fenpropathrin had residue levels 21.9 and 17.5 mg/kg, respectively.

Since the current study was conducted on pesticide residues in imported fruit and vegetables samples, then it is suggested that the fruit producers should regard the pre harvest periods (e.g. five days for diazinon; four days for permethrin, and 13 days for chlorpyrifos) before distributing the crops into the markets. As the previous studies showed, considering pre harvest period on the fruits and vegetables decreased the level of different pesticide residues [8,9,10]. MRL exceedance may be due to GAP non-compliance, cross-contamination or spray drift, contamination from a previous use of persistent pesticides, and/or unexpectedly slow degradation of residues [23].

3.3 The Frequency of Detection

Pesticide residue concentrations above the MRLs stipulated by CODEX and EU regulation [24,25]. Tables 8 and 9 presents the detection frequency of the pesticides that frequently occurred in the analysed samples.

Table 8 establishes a relationship of monitoring the most frequently detected pesticides and fruits samples exceeding the MRLs in force, the most frequent were Bifenazate, detected in 67 samples: oranges (33), mandarins (22), lemon (8), mango (1) and Grapefruit (1) followed by Spirodiclofen in 31 samples: date (31); Carbendazim in 13 samples: date (2), lemon (1),

grape (1), pomegranate (5) apple (1), banana (1) and nectarine (2); Acetamiprid in 11 samples: orange (1), grape (1), Pomegranate (1), peach (1), Guava (4), Grapefruit (2) and pear (1); many pesticides (Boscalid, Chlorfenapyr, Chlorpropham, Amitraz, Azoxystrobin, Cycloxydim, Cyhalothrin, Difenconazole – Dimethirimol, Diphenylamine, Famoxadone, Fenobucarb, Fipronil, Fosthiazate, Mandipropamid, Paraoxon-methyl, Phenthoate, Propargite, Thiamethoxam, Tridimenol and Triflumuron), which exceeded MRLs were found occasionally in one or two samples.

Table 9 establishes a relationship; of monitoring the most frequently detected pesticides and vegetables samples exceeding the MRLs in force, the most frequent were Chlorpyrifos, detected in 22 samples: Tomatoes (2), Parsley (1), Lettuce (2), Melons (3), Cucumbers (2), Beans-Green (2), Gourd (1), Taro (2) and Peas (2) followed by Acetamiprid in 19 samples: capsicum (10); Tomatoes (2), Coriander (3), Gourd (1) and Watercress (3); Phenthoate in 18 samples: Tomatoes (5) and Onion (13) and many pesticides which exceeded MRLs were found occasionally in one or two sample.

As summary of analysis results in this study the percentage of samples with residues above the maximum residue levels (MRL) were 7.25% and 5.42% in vegetables and fruits samples, respectively. whereas samples with residues within MRL were 27.30% and 32.61% in vegetables and fruits samples, respectively. A total of 6176 samples (63.51%) were free from detectable residues. The most fruits contain residues above the limit of detection were dates, orange, Mandarins, Lemon, Grapes and Pomegranate. The Most vegetables contain residues above the limit of detection were Capsicum, Tomatoes, parsley, Onion and Lettuce. Out of the 343 pesticides tested, 34 pesticides were found above the limit of detection, according to UAE, Codex and European regulations. The most frequently pesticides detected above the MRL in fruits samples were Bifenazate, Spirodiclofen, Carbendazim, Acetamiprid Deltamehrin, Ethion and Imazalil, respectively. While in vegetables samples The most frequently pesticides detected above the MRL in fruits samples were Chlorpyrifos, Acetamiprid, Phenthoate, Acephate, Tebufenpyrad, Chlorfenapyr, Metalaxyl, Carbendazim and Dimethoate, respectively.

Table 6. Distribution of pesticides residues above MRL in all fruits samples monitored

Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs	Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs	Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs			
Bifenazate	17	81	Registered	Thiabendazole	17	4	Not Registered	Imazalil	32	24	Banned
Spirodiclofen	3.1	36	Registered	Paclobutrazol	0.06	3	Not Registered	Carbendazim	0.78	15	Banned
Chlorpyrifos	2.03	14	Registered	Triadimenol	0.21	3	Not Registered	Fenpropathrin	0.37	14	Banned
Deltamethrin	0.35	12	Registered	Tebufenpyrad	0.82	2	Not Registered	Dimethoate	0.4	10	Banned
Acetamiprid	3.4	11	Registered	Chlorpropham	0.1	1	Not Registered	Ethion	1.5	10	Banned
Pyridaben	0.73	7	Registered	Cycloxydim	0.141	1	Not Registered	Thiophanate- methyl	15	8	Banned
Pyrimethanil	11	7	Registered	Dimethirimol	0.025	1	Not Registered	Acephate	0.5	7	Banned
Metalaxyl	2.45	5	Registered	Dimethomorph	4.132	1	Not Registered	Profenofos	0.145	7	Banned
Imidacloprid	0.8	5	Registered	Diphenylamine	0.08	1	Not Registered	Hexaconazole	0.1	5	Banned
Clothianidin	1.11	4	Registered	Famoxadone	0.03	1	Not Registered	Omethoate	0.09	5	Banned
Abamectin	0.05	3	Registered	Fenobucarb	0.22	1	Not Registered	Oxadixyl	0.1	3	Banned
Azoxystrobin	2.39	3	Registered	Fosthiazate	0.03	1	Not Registered	Propargite	0.075	3	Banned
Bifenthrin	1.2	3	Registered	Paraoxon-methyl	0.41	1	Not Registered	Phenthoate	0.08	2	Banned
Clofentezine	0.6	3	Registered	Quinalphos	0.018	1	Not Registered	Chlorfenapyr	0.04	1	Banned
Difenoconazole	0.63	3	Registered					Fipronil	0.075	1	Banned
Fenpyroximate	0.89	3	Registered					Malathion	0.03	1	Banned
Thiamethaoxam	1.5	3	Registered					Methomyl	0.04	1	Banned
Etoxazole	0.6	2	Registered					Penconazole	0.25	1	Banned
Fludioxonil	0.311	2	Registered								
Lufenuron	0.27	2	Registered								
Mandipropamid	2.08	2	Registered								
Propiconazole	15	2	Registered								
Boscalid	0.052	1	Registered								
Cyhalothrin	0.1	1	Registered								
Piperonyl butoxide	0.03	1	Registered								
Triflumuron	0.3	1	Registered								

Table 7. Distribution of pesticides residues above MRL in all vegetables samples monitored

Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs	Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs	Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs
Acetamiprid	5.6	27 Registered	Tebufenpyrad	6.7	13 Not Registered	Phenthoate	0.54	19 Banned
Chlorpyrifos	12.4	23 Registered	Famoxadone	1.37	8 Not Registered	Acephate	3.6	12 Banned
Pyridaben	2.6	14 Registered	Thiabendazole	0.4	8 Not Registered	Chlorfenapyr	0.26	12 Banned
Clothianidin	3.8	10 Registered	Triadimenol	0.416	6 Not Registered	Carbendazim	1.9	11 Banned
Metaxyl	0.3	9 Registered	Bromopropylate	0.9	3 Not Registered	Fenpropathrin	17.5	9 Banned
Bifenazate	8	8 Registered	Quinalphos	0.1	3 Not Registered	Malathion	1.5	9 Banned
Thiamethoxam	9.04	7 Registered	Carbofuran -3-Hydroxy	0.15	1 Not Registered	Dimethoate	0.53	7 Banned
Imidacloprid	5.02	6 Registered	Carboxin	0.3	1 Not Registered	Hexaconazole	0.16	7 Banned
Clofentezine	0.4	5 Registered	Cymoxanil	0.1	1 Not Registered	Methomyl	5.8	7 Banned
Diflubenzuron	0.4	4 Registered	Emamectin	0.08	1 Not Registered	Omethoate	0.09	7 Banned
Tebuconazole	5.1	4 Registered	Fenproidol	0.03	1 Not Registered	Penconazole	4.5	7 Banned
Propiconazole	0.66	3 Registered	Flutriafol	0.32	1 Not Registered	Ethion	1.79	6 Banned
Fenazaquin	0.335	3 Registered	Malaoxon	0.14	1 Not Registered	Carbofuran	0.07	5 Banned
Cyromazine	1.06	2 Registered	Phenylphenol	0.4	1 Not Registered	Oxadiazon	2.4	4 Banned
Difenoconazole	17	2 Registered	Prochloraz	0.169	1 Not Registered	Oxadixyl	9.6	4 Banned
Fenpyroximate	0.32	2 Registered				Profenofos	4.27	4 Banned
Pirimiphos-Methyl	1.9	2 Registered				Propargite	0.37	4 Banned
pyriproxyfen	0.9	2 Registered				Chlorfenvinphos	0.23	3 Banned
Spirodiclofen	0.38	2 Registered				Imazalil	0.268	3 Banned
Bifenthrin	0.3	2 Registered				Linuron	0.68	3 Banned
Lufenuron	0.14	2 Registered				Fipronil	0.3	2 Banned
Azoxystrobin	0.017	1 Registered				Sulfotep	0.05	2 Banned
Boscalid	0.052	1 Registered				Myclobutanil	0.188	2 Banned
Cyfluthrin	0.7	1 Registered				Thiophanate-methyl	2.4	1 Banned
Etoazole	1	1 Registered				Triazophos	21.9	1 Banned
Piperonyl Butoxide	0.03	1 Registered				Ethirimol	0.081	1 Banned
Pyrimethanil	0.25	1 Registered				Fenamiphos	0.029	1 Banned
Triflumuron	0.04	1 Registered				Hexythiazox	0.6	1 Banned
Thiacloprid	1.3	1 Registered				Mepanipyrim	0.1	1 Banned

Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs	Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs	Pesticides	Maximum value detected (mg/kg)	Samples with residues above MRLs
Acrinathrin	0.13	1 Registered				Methamidophos	0.05	1 Banned
						Monocrotophos	0.199	1 Banned
						Procymidone	0.04	1 Banned
						Cyproconazole	0.07	1 Banned
						Diazinon	0.142	1 Banned

Table 8. Pesticide frequencies and exceed MRLs in the analyzed fruit samples

Pesticides																				Total	
	Dates	Orange	Mandrins	Lemon	Grapes	Pomegranate	Peach	Mango	Guava	Pomelo	Apple	Banana	Apricots	Nectarin	Grapefruit	Strawberry	Persimmon	Plum	Pear		Kiwifruit
Bifenazate		33	22	8				1			2				1						67
Spirodiclofen	31																				31
Carbendazim	2			1	1	5				1	1			2							13
Acetamiprid		1			1	1	1		4						2				1		11
Deltamehrin	9													1							10
Ehion	4					3				3											10
Imazalil		3	5	1				1													10
Chlorpyrifos	5			1				2	1												9
Fenpropathrin					1	1	5	1						1							9
Dimethoate					1		3		1		3										8
Profenofos		1	1	3						1											6
Thiophanate-methyl	1				1					1	2	1									6
Pyridaben	5																				5
Acephate					2			2													4
Hexaconazole				1	1								2								4
Imidacloprid	2							1												1	4
Pyrimethanil			2	1													1				4

Table 8. (continued)

Pesticides	Dates	Orange	Mandirins	Lemon	Grapes	Pomegranate	Peach	Mango	Guava	Pomelo	Apple	Banana	Apricots	Nectarin	Grapefruit	Strawberry	Persimmon	Plum	Pear	Kiwifruit	Total		
Abamectin	3																					3	
Bifenthrin	1					1	1																3
Clofentezine	3																						3
Etoxazole				1												2							3
Fenpyroximate	3																						3
Omethoate			1					2															3
Paclobutrazol				3																			3
Clothianidin					2																		2
Metalaxyl	1				1																		2
Oxadixyl										1					1								2
Thiabendazole																		2					2
Fludioxonil																	2						2
Boscalid						1																	1
Chlorfenapyr									1														1
Chlorpropham							1																1
Amitraz																					1		1
Azoxystrobin																			1				1
Cycloxydim		1																					1
Cyhalothrin					1																		1

Table 8. (continued)

Pesticides	Dates	Orange	Mandrins	Lemon	Grapes	Pomegranate	Peach	Mango	Guava	Pomelo	Apple	Banana	Apricots	Nectarin	Grapefruit	Strawberry	Persimmon	Plum	Pear	Kiwifruit	Total	
Difenoconazole								1														1
Dimethirimol											1											1
Diphenylamine					1																	1
Famoxadone				1																		1
Fenobucarb				1																		1
Fipronil					1																	1
Fosthiazate	1																					1
Mandipropamid							1															1
paraoxon-methyl				1																		1
Phenthoate						1																1
Propargite																1						1
Thiamethoxam	1																					1
Tridimenol													1									1
Triflumuron	1																					1
Total	73	39	31	23	14	13	12	9	9	6	5	5	4	4	4	3	3	2	2	2	2	263

Table 9. Pesticide frequencies and exceed MRLs in the analyzed vegetable samples

Pesticides	Capsicums	Tomato	Parsley	Onion	Lettuce	Ginger rhizomes	Melons	Cabbage	Carrots	Courgette	Potato	Cucumbers	Beans-Green	Coriander	Gourd	Curry leaves	Cauliflower	Pumpkins	Watercress	Garlic	Taro	Peas	Total	
Chlorpyrifos		8	1		2		3					2	2		1						2	1	22	
Acetamiprid	10	2												3	1				3					19
Phenthoate		5		13																				18
Acephate	2						3	2			5													12
Tebufenpyrad	1					10																		11
Chlorfenapyr	1	9								1														11
Metalaxyl		1					1			3	1							3						9
Carbendazim	1	1	4		1						1			1										9
Dimethoate							1	2		2			2				2							9
Clothianidin	4	1					2									1								8
Pyridaben	3				5																			8
Famoxadone	2				3			2																7
Thiabendazole				1	1			1		1					1		1							6
Bifenazate	1		2	3																				6
Ethion	5																							5
Penconazole			5																					5
Carbofuran					1										2								1	4
Diflubenzuron		3								1														4

Table 9. (continued)

Pesticides	Capsicum	Tomato	Parsley	Onion	Lettuce	Ginger rhizomes	Melons	Cabbage	Carrots	Courgette	Potato	Cucumbers	Beans-Green	Coriander	Gourd	Curry leaves	Cauliflower	Pumpkins	Watercress	Garlic	Taro	Peas	Total
Imidacloprid	1						1									2							4
Clofentezine	2											1											3
Fenpropathrin								3															3
Hexaconazole	3																						3
Linuron			1						2														3
Methomyl	1	1										1											3
Omethoate					1			1									1						3
Oxadiazon									2					1									3
Profenofos	1						1		1														3
Chlorfenvinphos	1											1											2
Cyfluthrin	1					1																	2
Fenazaquin						2																	2
Malathion	1	1																					2
Myclobutanil									2														2
Tebuconazole			2																				2
Pirimiphos-Methyl			1						1														2
Thiophanate-methyl					1							1											2
Propargite	1						1																2

Table 9. (continued)

Pesticides																					Total		
	Capsicums	Tomato	Parsley	Onion	Lettuce	Ginger rhizomes	Melons	Cabbage	Carrots	Courgette	Potato	Cucumbers	Beans-Green	Coriander	Gourd	Curry leaves	Cauliflower	Pumpkins	Watercress	Garlic		Taro	Peas
Triadimenol			1						1														2
Quinalphos		1	1																				2
Bromopropylate	2																						2
Cyproconazole	1																						1
Cyromazine						1																	1
Diazinon	1																						1
Difenoconazole	1																						1
Ethirimol									1														1
Fenamiphos											1												1
Etoxazole	1																						1
Fenproidin			1																				1
Flutriafol													1										1
Imazalil		1																					1
Lufenuron								1															1
Malaoxon																				1			1
Mepanipyrim			1																				1
Oxamyl			1																				1
Monocrotophos																1							1

Table 9. (continued)

Pesticides																					Total			
	Capsicums	Tomato	Parsley	Onion	Lettuce	Ginger rhizomes	Melons	Cabbage	Carrots	Courgette	Potato	Cucumbers	Beans-Green	Coriander	Gourd	Curry leaves	Cauliflower	Pumpkins	Watercress	Garlic		Taro	Peas	
Oxadixyl			1																				1	
Phenylphenol		1																						1
Prochlora	1																							1
Procymidone		1																						1
Spirodiclofen										1														1
Triflumuron		1																						1
Bifenthrin			1																					1
Boscalid																				1				1
Carbofuran -3-Hydroxy	1																							1
Carboxin												1												1
Total	50	35	25	17	15	14	13	12	10	9	8	6	6	5	5	4	4	3	3	2	2	2		250

This study provides an additional perspective by estimating quantities of pesticide residues to which consumers could be exposed in UAE specifically, also the percentage of samples contain pesticides residues may exceeding the MRLs slightly that could remain on imported fruits and vegetables to UAE, if exporters adhered to the maximum allowable application rates based on their own market guidelines rather than those of the UAE. Moreover, not adhered by pre harvest intervals.

4. CONCLUSIONS

Food safety is a core priority of the UAE's food security system. The Ministry of Climate Change and Environment continues its efforts to provide healthy and safe food to consumers in line with global best practices and the objectives of the UAE National Agenda and the UAE Vision 2021. Enhancing food safety and sustaining local production are strategic priorities for MOCCA. Furthermore, the Ministry is also keen to ensure that all foodstuffs and products in the country, both domestically produced and imported, are safe for consumption.

The UAE imports a significant and rising portion of its fruits and vegetables, Pesticide residues not only endanger human health and lead to concerns about food safety problems, but also seriously affect the import and export trade of agricultural products. Therefore, pesticide residues have become an important issue in the field of food safety in UAE and a primary concern in UAE society. Residual pesticide monitoring of fruit and vegetables is a key tool for ensuring conformity with regulatory requirements and compliance with GAP. Most samples tested in UAE over 2019 period (93.75%) were compliant and were comparable with national and with similar pesticide residue monitoring programs conducted elsewhere. The current study findings showed that the multi-residue method with LC-MS/MS and GC-MS/MS could detect a large number of pesticide residues at the same time with high accuracy. Therefore, it is recommended that this method be employed for all vegetables and fruits in order to assess their pesticide residue levels before reaching the markets.

In conclusion, in this study we would like to recommend continued monitoring of pesticide

residues in imported food to control and improve import food safety and reduce pesticide exposures in originating countries. We recommend also, expand ministry of climate change and environment related to pesticide residue monitoring programs to include a greater number of samples analyzed annually, with an emphasis on imported products, and to develop complementary strategy to avoid duplication with other laboratories all over the country.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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