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Monitoring of Pesticide Residues in Imported Datepalm Fruits in United Arab Emirates

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

Article Information

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ABSTRACT

The occurrence of pesticide residues in representative samples collected from imported dates palm fruits during 2020 to United Arab Emirates (UAE) was investigated to ensure compliance with the standard specifications and requirements by the regulatory and supervisory authorities, maintain the health and safety of consumers and improve food safety. An accurate, rapid, and reliable method for the simultaneous determination of pesticide multi-residues in 230 samples imported dates by liquid chromatography coupled with tandem mass spectrometry (LC–ESI (+)-MS/MS) operating in multiple reaction monitoring (MRM) mode and modified quick, easy, cheap, effective, rugged, safe (QuEChERS) method was used. The performance of the analytical method was validated in accordance with EU SANCO guidelines (SANTE/12682/2019) for monitoring pesticide multi-residues level of 343 compounds were determined in 230 samples. Results indicated that the percentage of samples with residues above the maximum residue levels (MRL) was 4.34% in dates samples, whereas samples with residues within MRL were 7.39% in dates samples. A total of 230 samples of 88.26% were free from detectable residues. Out of the 343 pesticides tested, 11 pesticides were found above the limit of detection, according to UAE, Codex, and

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European regulations. The main purpose of this work is to inform citizens and traders who have concerns about food safety on the capability of the MOCCAE on quality assurance regarding pesticide residue in imported food.

Keywords: Determination; Dates; Multi residue; pesticides; liquid chromatography; tandem mass spectrometry (MS/MS); QuEChERS; UAE.

1. INTRODUCTION

Dates are the fruit of the date palm tree(Phoenix dactylifera L.), which is grown in many tropical regions of the world. The importance of the date palm for human nutrition is mainly due to its health benefits of dates such as lowering protein-rich, cholesterol, rich in vitamins. bone improving health, strengthening the nervous system, rich in iron, promoting digestion, improving skin, fixing hangovers and assists in weight gain [1]. Dates are considered as principal food in the Arab peninsula.

The residual effect of pesticides in food commodities has become a major cause of concern all over the world. Food safety is crucial and consumers have to be assured that they are not exposed to an unacceptable level of pesticide residues. After the establishment of the world (WTO) and other trade order General Agreements on Tariffs and Trade (GATT), Sanitary and phytosanitary SPS, the presence of the residues above the permissible level is also a major bottleneck in the international trade of food commodities.

In order to ensure food safety for consumers and protect human health, many organizations and countries around the world have established maximum residue limits (MRLs) for pesticides in food commodities [2, 3, 4]. The MRL is the extreme level of a pesticide residue (expressed in mg·kg⁻¹) that is legally permitted in or on food or animal feed [3]. Therefore, the pesticide residues in food have been strictly regulated by governments all over the world to determine whether the concentration of pesticides used exceeds their MRLs.

The Ministry of Climate Change and Environment (MCCE), United Arab Emirates(UAE) is keen to ensure that all foodstuffs and products in the country, both domestically produced and imported, are safe for consumption and to strengthen infrastructure at Quarantine stations to prevent the entry of food commodities which have pesticide residues above the MRLs, Testing/Certification of pesticide residue in export/import consignments [4,5].

The maximum permitted levels of pesticide residues in foods are stipulated by regulatory bodies in the UAE. National Laboratories Department, MOCCAE has monitored the residual effects of Pesticides in imported food commodities which was accredited by the British Commission for Accreditation (UKAS) [6].

To fulfill the rule of the central lab of the MOCCAE, this study was carried out to ensure that pesticide residues in imported dates are not exceeding the maximum permissible limit of pesticide residues in the country, which is considered a safe limit for the consumer approved by the local and international specialized agencies. To enhance food safety in the UAE and ensure that the highest-rated food products are traded only, this monitoring program was carried out.

2. MATERIALS AND METHODS

2.1 Chemicals and Standard Solutions

Certified analytical standards-tested pesticides were purchased from Dr. EhrenstoferGmbH(Germany), with purity between 92.0 and 99.5%, Acetonitrile (Merck, Germany), methanol (LC-MS grade, Scharlab), Formic acid (Honeywell, Germany).

Ready-made QuECHERS kits were purchased from Suplco; Supel[™] QuE citrate extraction tube (contains 4.0 g MgSO₄, 1.0 g NaCl, 0.5 g sodium Citrate dibasic sesquihydrate, 1.0 g NaCitrate tribasic dehydrate), Supel[™]QuE PSA SPE clean up Tube (contains 150.0 mg Supelclean PSA, 900.0 mg MgSO₄.) The solutions were prepared with Ultrapure demineralized water Milli-Q plus system (Merck-Millipore Corporations, USA).

2.1.1 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. Afterward, a mixture with the concentration of 10 mg L⁻¹ containing all

pesticides was prepared, which was diluted to 1 mg L^{-1} and was kept in the refrigerator at 4.0 ± 2.0°C (Table 1). Internal standard solution: A solution of Triphenylphosphate (TPP), analytical grade, was used as an internal standard.

2.1.2 Apparatus

Analysis of final extracts Agilent 6460 triple quadrupole, on a reversed-phase column and detected by tandem mass spectrometry (MS/MS) using electrospray ionization (ESI). The pesticide residues were determined with positive ESI only. The total chromatographic run time was 32 minutes. The 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 a precursor ion there were two productions determined. One production was used for quantification and the other one was used for qualification. Detected pesticides are shown in Table 1.

2.2 Sampling

The pesticide residue random monitoring program aimed to collect samples from all imported agricultural consignments at UAE border ports according to plans based on scientific foundations with the aim of laboratory analysis, to assess the extent to contain pesticide residues of pesticides in accordance with the Technical Regulations issued by United Arab Emirates Authority for Standardization and Metrology No. «UAE.S MRL 1-2019» regarding maximum limits [7]. A total of 230 date samples were collected from January to December 2020 from imported consignments in all over UAE ports.

Samples were taken by the Ministry'inspectors; each sample was approximately 1 Kg weight was placed in sterile polythene bags in an ice chess box to avoid contamination, deterioration, and labeled and transferred to the laboratory under appropriate transport conditions within 24 hours for analysis.

The used 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), and accreditation was

gradually extended for the analysis of pesticides in several foodstuffs and compliance with the document SANTE/12682/2019 [8]. A guidance document on analytical quality control and method validation procedures for pesticide residue analysis in food and feed was issued by the European Commission Directorate-General for Health and Food Safety and became effective on January 1, 2020. Samples were labeled and crushed after seeds were discarded and then stored at 4°C until analysis. Blank samples (Pesticide free samples) were acquired from the consumer market used for validation experiments.

2.3 Sample Preparation

The pesticide residues were analyzed using the method approved by the European Union (EU) to determine the level of Pesticides in food products QuECheRS method reported by Anastassiadeset al. [9] by using LCMSMS (liquid chromatography-tandem mass spectrometry).

This method is validated to use at theNational Laboratories, MOCCAE, UAE. It employs dispersive solid-phase extraction (SPE) followed by chromatographic analysis of the extracts. Liquid Chromatography coupled with tandem Mass Spectrometry was adopted to achieve qualitative and quantitative analysis.

The following sample extraction and clean-up steps were conducted: A homogenate date sample (5 g) in a 50-mL PTFE centrifuge tube was extracted with 10 mL of water, shake then, 10 ml acetonitrile add and shaken for 1 min. Following this step, a mixture of extraction salts (4 g of magnesium sulfate; 1 g of sodium chloride; 1 g of trisodium citrate dihydrate, and 0.5 of disodium hydrogen citrate g sesquihydrate) were added to each sample. After being shaken vigorously for 1 min, tubes were centrifuged for 10 min at 4000 rpm to separate solid from acetonitrile supernatant which was ready to dispersive Solid Phase Extraction with 150.0 mg Supelclean PSA and 900.0 mg MgSO₄.The final extract was stabilized by 5% formic acid in acetonitrile solution. The sample was then injected into a liquid chromatography coupled to tandem mass spectrometry (LC-Recovery compliance with MS/MS). the document SANTE/12682/2019 [8] representative portions of the previously homogenized samples were spiked homogenously with the appropriate amount of the working standard solution in methanol (0.1 mg kg⁻¹).

Table 1. List of active ingredients tested for their residues

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3. RESULTS AND DISCUSSION

3.1 Monitoring the Presence of Pesticides Residues

Work here is a statistical representation of the situation of pesticide residues in imported date fruits to UAE market, considering dates as the most widely consumed fruit in many Arab Gulf and Arab countries.

This study analyzed the presence of 343 pesticide residues using the LC-ESI (+)-MS/MS method, which combines LC chromatography with tandem mass spectrometry. In (Table 1) 230 samples accessible from UAE entry ports were tested using the multi-residue method in imported dates to the UAE throughout the year 2020. After optimization of the analytical conditions, a total of 343 pesticides were detected with good sensitivity by the multiresidue analysis method. According to the European Commission's (SANTE/12682/2019) requirements for validation parameters, the followina parameters were investigated: recovery, linearity, and accuracy of peak regions. Under replicated conditions, good linearity was obtained with regression coefficients (R2 > 0.99), recovery results ranged from 70% to 120%, and precision based on relative standard deviations (RSD) ranged from 2 to 20%; these values are considered acceptable according to the European Commission's requirements (2019). No residues were detected in the blank date palm fruits samples at the LOD of the method.

Maximum amounts of certain pollutants in food are specified by a European Commission regulation. This regulation is often updated, regardless of the specific limits of general foodstuffs, and there are a number of specific limits for pollutants for specific products, including dates. Pesticide residues are the most frequent requirements for date pollutants [10].

Herein the results showed that 95.65% of the samples (220 samples) were in conformity with

the specifications, total88.26% samples were free of pesticide residues, while the percentage of samples that contain pesticide residues exceeded the permissible limits (MRL) was were 4.34% (10 samples) of the total number of samples (Table 2) and considered as noncompliant with CODEX and EU legislation on pesticide residues [11].

Results show the percentage of samples that exceeded the permissible limits decreased, as the percentage in the first quarter of the year was 8.21%, while in the fourth quarter of the year it amounted to 2.56%. On the contrary, no MRL exceedance was reported in the second quarter for pesticides that were found to exceed the legal limit in the first quarter e. g. Carbendazim, Deltamethrin, Ethion, Fipronil, Diflubenzuron, Indoxacarb and Triflumuron.

In summary; the year2020 was slightly lower in the first quarter, percentage of samples exceeding the limits allowed in the program 4.34% (Table 2). For pesticides with MRL, exceedances during 2020 the year a significant decrease of MRL the highest rate was observed within the fourth quarter 2020. Continuous consumption of food products even with moderate pesticide contamination may have negative consequences on human health in the long term [12].

Several countries have relied on applications of the broad spectrum of synthetic pesticides, such as Amitraz, Fenpropythrine, Finperochemate, Ttradyphon, Finazakuen, Propargate, and Hexithiasoxy[13], Studies have detected pesticide residues in dates marketed, in Saudi Arabia within 2018 [14] Pesticide residues were detected in 36 (18%) within the MRL allowed but 15 samples (7.5%) exceeded the maximum residue levels, in Tunisia monitor pesticide residues in dates sampled from Tunisian oasis during the agricultural season 2017-2018 and to check compliance with existing regulations especially for European Community [15].

Table 2. The percentage of samples that are compatible and exceed the permissible limitsduring the year 2020

Year/quarter	Free from pesticide residues limits	Within pesticide residues limits	Exceed pesticides residues limits
Quarter 1 2020	80.82	10.95	8.21
Quarter 2 2020	82.35	17.64	0.00
Quarter 3 2020	88.63	6.81	4.54
Quarter 4 2020	93.59	3.84	2.56
Total	88.26	7.39	4.34

MRLs are based on good agricultural practices and examined to ensure that acceptable daily intake or acute reference dose is not exceeded. In the light of these results, action has been taken on samples containing MRL overrun in accordance with approved standards requiring the importer to re-export or destroy the product.to maintain food safety in imported date palm fruits.

3.2 Residues Measured above the MRL

A total of 10 samples with residues exceeding the legal limit (above MRL) of one or several pesticides were detected as shown in Table 3.

In this study residues exceeding the legal limits were related to 11 different pesticides in date samples as shown in the above table were Acetamiprid. Carbendazim. Deltamethrin. Diflubenzuron, Ethion, Fipronil, Imidacloprid, Indoxacarb, Pyriproxyfen, Spirodiclofen, and Triflumuron, accounted 47.8% (11/23) and out of 11 pesticides that exceeded the MRLs, banned pesticides for use in agriculture were detected in 3 (27.27%) namingCarbendazim, Ethion, and Fipronil. The most frequently detected pesticides exceeded MRLswere Acetamiprid. Deltamethrin and Indoxacarb. The fact that 4.34% of samples exceeded the MRLs and contained 3 banned pesticides, suggested that banned pesticides might still be frequently used in date for exported countries. These results reflect that a certain proportion of farmers may not follow good agricultural practices (GAP).

3.3 Residues of more than One Pesticide (Multiple residues)

In this study, some samples contained only one pesticide residue, but 40% (4 out of 10)

exceeded the permissible limits samples had multiple residues from more than one insecticide present in the same sample, while 17.64% (3 out of 17) within MRL. More than one residue has been detected in date samples, and in recent years, indicating the mixed-use of pesticides due to pest resistance, multiple pesticides have been widely applied.

For the Prohibition of Pesticides toxic to humans, as well as pesticides that remain for the longest period in the environment and protect public health by setting limits on pesticide residues in food. All pesticides that have a significant threat to human health and the environment are banned in the UAE.

Of the pesticide residues detected above EU residue limits (MRLs), according to the MOCCAE list of pesticides (MOCCAE), a total of 11 pesticides are incompatible with regulations exceeding the legal limit, including 6 pesticides permitted in the UAE, two restricted pesticides, and 3 unapproved substances (banned pesticides) (Table 4).

Although WHO classified fipronil and Ethionby hazard as moderately hazardous (Class II) [16]. Fipronil has been associated with human Possible Human Carcinogen according to EPA classified.

According to UAE food regulations Carbendazim, Ethion and Fipronil are not allowed to be used, they have banned compounds (Table 4) according to Ministerial Decree No. (36) of 2018 regarding banned and restricted pesticides in the United Arab Emirates.

Sample	Pesticide Detected	Residue Detected	MRL*	EU Pesticides Database**	
no .		(mg/kg)	(mg/kg)		
1	Carbendazim	1.016	0.1	Reg. (EU) No 559/2011	
	Spirodiclofen	0.13	0.02	Reg. (EU) 2016/1902	
2	Deltamethrin	0.166	0.01	Reg. (EU) 2018/832	
	Ethion	0.134	0.01	Reg. (EU) No 310/2011	
	Fipronil	0.037	0.005	Reg. (EU) 2019/1792	
4	Diflubenzuron	0.64	0.01	Reg. (EU) 2019/91	
	Indoxacarb	0.01	0.02	Reg. (EU) 2015/845	
5	Deltamethrin	0.04	0.01	Reg. (EU) 2018/832	
6	Triflumuron	0.1	0.01	Reg. (EU) 2018/1516	
7	Indoxicarb	0.29	0.02	Reg. (EU) 2015/845	
8	Imidacloprid	0.477	0.05	Reg. (EU) No 491/2014	
9	Acetamiprid	0.12	0.01	Reg. (EU) 2019/88	
	Pyriproxyfen	0.14	0.05	Reg. (EU) 2020/856	
10	Acetamiprid	0.489	0.01	Reg. (EU) 2019/88	
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Table 3. List of dete	ected pesticides	above the MRL
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*MRL maximum residue level ** EU Pesticides Database

Table 4. Distribution of pesticides residues above MRL in samples monitored

Pesticides	Maximum value detected (mg/kg)	Status of Pesticides	Pesticides	Maximum value detected (mg/kg)	Status of Pesticides	Pesticides	Status of Pesticides
Deltamethrin		Allowed	Imidacloprid		Restricted	Carbendazim	Banned
Acetamiprid		Allowed	Spirodiclofen		Restricted	Ethion	Banned
Triflumuron		Allowed				Fipronil	Banned
Indoxacarb		Allowed					
Diflubenzuron		Allowed					
Pyriproxyfen		Allowed					

*According to List of Registered Pesticides in the Ministry (MOCCAE) Last update 13 August 2021

Carbendazim is classified as a pesticide unlikely to present acute hazard in normal use is also detected in some of the samples. Fipronil (0.037 mg kg⁻¹) to be detected above the upper limit of the maximum (MRLs level) allowed residue set by Reg. (EU) 2019/1792 (0.005 mg kg⁻¹) for date palm fruits, these results give an indication of possible misuses of non-approved active substances, should follow-up on these national monitoring programs and take corrective measures where appropriate.

4. CONCLUSION

Pesticide residue monitoring program is a compliance program used to monitor the level of chemical residues of pesticides in imported palm fruits in the UAE during 2020 to ensure that they do not exceed MRL limits using liquid mass spectrometry (MS/MS) techniques because of improper use pesticides leads to professionalism, environmental and food security risks, and monitoring pesticide residues helps to assess the risks of consumer exposure to such residues and ensure a high level of consumer protection.

Although pesticides are used to control pests and diseases, it is therefore recommended that the pesticide residue control program should continue in imported dates in the UAE while maintaining the analytical scope as widely as possible, taking into account the fact that the residues of unappointed active substances in the 2020 control program are low. On the other hand, there is control of all pesticide residues in all imported food products, and the high observed rate of sample compliance with the regulation that does not exceed their MRL in palm fruit dates of good agricultural practices (GAP) follows

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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