



Risk Assessment of Pesticide Residues in Dates in the Emirate of Abu Dhabi

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ABSTRACT

Pesticides are applied to protect plants from pest infestation and subsequent damage caused resulting in undesirable effects. Nevertheless, human exposure to residues from these pesticides may pose health implications. Residue monitoring programs have been established in many countries to assess the use of pesticides in accordance with good agricultural practices. The current study is an effort to report on the status of pesticide residues in date fruit and to conduct a risk assessment based on dates consumption among adults in the UAE. A total of 464 date fruit samples were obtained in 2024 as part of this survey in the Emirate of Abu Dhabi and analyzed for 365 pesticide residues. Pesticide extraction was performed using the QuEChERS method, followed by detection through liquid chromatography coupled with tandem mass spectrometry. The results from the validation data indicate that the method has sufficient accuracy for the detection of pesticide residues in date palm fruits. A total of 292 (63%) samples did not contain detectable quantity of pesticides (ND) while 172 samples (37%) contained pesticide residues exceeding the regulatory limits (>MRL). Deltamethrin of pyrethroid family was the predominant insecticide found in 41% of the positive samples. In addition, residues of Cypermethrin, Pyridaben, Lambda-Cyhalothrin, Chlorpyrifos, Spirodiclofen, Thiamethoxam, Carbendazim, Imidacloprid, Fenzaquin, Fenvalerate, Matrine, Chlorantraniliprole, Clothianidin Fenpyroximat, Acetamiprid, Abamectin, Acirathrin and Ethion were also detected. The hazard index data indicates that the dietary exposure to the assessed pesticides is within acceptable safety limits. The study emphasizes the significance of regular monitoring and its value in food safety management.

Keywords: Dates, Pesticide; Residues; LC-MS-MS; hazard index.

Article History

Article # 25-533

Received: 09-Sep-25

Revised: 25-Sep-25

Accepted: 07-Oct-25

Online First: 12-Dec-25

INTRODUCTION

Pesticides are chemical or biological substances intended to protect plants from pests that may otherwise cause disease or undesirable impact in plants and plant products (Zafar et al., 2020). Insecticides, herbicides, fungicides and rodenticides are some of the typical examples of pesticides. They are classified based on their target organisms. Insecticides are chemicals that controls or kill insect pest while herbicides control or kill unwanted plants (Zafar et al., 2022; Kamal et al., 2024). Rodenticides are used to kill rats and mice and fungicides kill fungi, such as molds and mildews. The intended use of pesticide is to manage the pests and associated diseases failing which can cause significant harm to crop. At the same time, overuse can cause adverse effects to health and

environment (Ahmad et al., 2024). Hence, it is very important to ensure that the chemical and biological substances used in pesticides as active ingredients do not risk humans, animals or the environment (Ren et al., 2019). In this context, the Codex Alimentarius has established the maximum residue levels (MRL) which states that "a maximum concentration of a pesticide residue to be legally permitted in food commodities and animal feeds" (FAO, 2015). A nationwide pesticide residue monitoring program has also been established in many countries to monitor the use of pesticides in accordance with good agricultural practices (GAP) (Eslami et al., 2021; Razzaq et al., 2023).

In the United Arab Emirates (UAE), regulation of pesticides is based on federal laws, ministerial decisions and national standards that together govern activities related to pesticides. The Ministry of Climate Change and

Cite this Article as: Siddiqui A, Marzouqi AASA, Hosani AMAHA and Jagadeesan P, 2026. Risk assessment of pesticide residues in dates in the Emirate of Abu Dhabi. International Journal of Agriculture and Biosciences 15(2): 496-501. <https://doi.org/10.47278/ijab/2025.214>



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Environment (MOCCAE) is the competent federal authority for all services such as registration, trade, permitted uses, labeling, safe handling, disposal and residue limits in food. Pesticides product registration requires technical dossiers, product specifications and other related documentation. Import consignments of pesticides are subject to prior permits and customs control. The country maintains lists of banned and restricted pesticides with periodical updates on those lists. The permissible limits of pesticide residues in agricultural and food products are covered in the standards (UAE.S GSO 382 & 383).

The date palm fruit is a nutritional powerhouse enriched with vitamins, minerals, and sugars. It's cultivation dates to the earliest civilization of the middle east (Fuller & Stevens, 2019; Basij et al., 2025). Today, UAE is one of the largest date fruit producers in the world with 40 million date trees and 200 cultivars approximately (Al-Muaini et al., 2019). About one third of the production is consumed locally while a sizable portion is exported to other countries in the world. Date palm cultivation is challenged by biotic factors such as insect pests and other microbial diseases resulting in poor yields. Nature and severity are influenced by factors like cultivar, location, weather etc. Globally, about 132 species of insects and mites are associated with date palms of which the red palm weevil, date dust mite and dubas bug are of economic importance (El-Shafie et al., 2017; Asl et al., 2022).

In the UAE, several preventive measures are being applied to contain red palm weevil consistently. For instance, cleaning, pruning of palm trees, visual inspection to find infested areas and application of integrated pest management are employed (Hammami et al., 2024). Insect infestation further facilitates microbial infection resulting in additional damage to the plants. Black scorch disease caused by *Thielaviopsis punctulata* and sudden decline syndrome (SDS) caused by *Fusarium* spp., are two important microbial species threatening the date palm cultivars. The latter species is a highly pathogenic form with limited chemical fungicide for the management (Alblooshi et al., 2022). Therefore, the application of pesticides has become an inevitable tool in the management of insect pests and the damage caused by microbes. On the contrary, overuse also poses environmental challenges due to the toxic chemicals present as active ingredients, necessitating regular monitoring to ensure residues found in foods comply with national MRLs (Abd El-Mageed et al., 2020). Recently, a new ministerial decision (116/2024) on the technical regulations for MRLs in agricultural and food products in the UAE has been issued (UAES MRL 1:2024), replacing the older regulation "UAES MRL1:2019. The revised standard contains updated MRLs for substances such as chlorpyrifos, imazalil, metalaxyl and fenpyroximate. The competent authorities and stakeholders are given a transition period of 180 days to implement the new standard. In general, these regulations are based on Codex Alimentarius and the Gulf Cooperation Council (GCC). With this background, the current study is undertaken to assess the status of pesticide residues in date fruit and to conduct a risk assessment based on

date's consumption among adults in the UAE. The exposure levels may provide further insights into their long-term health and environmental effects.

MATERIALS & METHODS

Samples

A total of 464 date fruit samples were obtained in 2024 as part of this survey in the Emirate of Abu Dhabi and analyzed for 365 pesticide residues.

Extraction

Date fruit samples were homogenized, and $10g \pm 0.1g$ was taken in a 50mL extraction tube containing 10mL of deionized water and shaken well for 2min. An internal standard, Tri phenyl phosphate (TPP) of 150ul ($10\mu g/ml$) was added followed by the addition of 10mL of Acetonitrile and then shaken vigorously for approximately 2min. QuEChERS AOAC extraction salt containing 6g magnesium sulphate and 1.5g sodium acetate was added and shaken vigorously for approximately 1min. The samples were centrifuged at 5000rpm for 5min and 5mL of supernatant was taken for cleanup into a 50mL extraction tube containing QuEChERS-d-SPE kit (50mg PSA, 150mg $MgSO_4$). The samples were shaken vigorously for 1min and centrifuged for 5min at 5000rpm. The supernatant was filtered through 0.22 or $0.45\mu m$ Millipore filter into a 2mL tube. For LCMSMS, 0.25mL of the filtrate was taken into two 2mL tubes and added 0.75mL of Mobile A to the first tube while 0.75mL of Mobile C was added to the second tube followed by the injection into the LCMSMS.

Standards and Reagents

Methanol, (MS grade), acetonitrile (HPLC grade), Ammonium formate, Formic Acid, Acetic acid, Sodium hydroxide, Magnesium sulphate and sodium acetate was purchased from Merck (Darmstadt, Germany) and water purification was obtained through Milli-Q- System. All pesticide standards were purchased from AccuStandard, Inc. (New Haven, CT, USA).

LC-MS/MS Analysis

For the separation of compounds, liquid chromatograph (Agilent 1200 Series) equipped with a reversed phase ACE Excel 3 Super C18 column was used. The column oven temperature was $40^\circ C$ and the analytes were separated using mobile phase A (for each 1L, mix the following: 900mL Deionized water, 100mL MeOH, 0.63g Ammonium formate to get 10mM), and 1mL Formic Acid (to get 0.1%). pH value should be within range of 3.25 to 3.5) and mobile phase B (for each 1L 900mL MeOH, 100mL Deionized water, 0.63g Ammonium formate to get 10mM) and 1mL Formic Acid (to get 0.1%). pH value should be 4.67 to 4.9. Mobile phases for Negative Mode, Mobile C (ACN: $H_2O = 90:10$), and 0.1mL Acetic Acid (to get 0.01%). Mobile D: For each 1L to Mix the following: 1000mL Acetonitrile, and 0.1mL Acetic Acid (to get 0.01%).

The flow rate was $0.6\mu L/min$, and the total analysis run time was 22min, with an injection volume of $5\mu L$. For tandem mass spectrometry, an integrated triple

quadrupole mass spectrometer (SCIEX API 3200) was used. The ion source worked in positive ionization mode, and the scheduled multiple reaction monitoring (sMRM) was applied. The ion source conditions were set as follows: temperature – 550°C, ion spray voltage - 5500V, curtain gas - 20psi, collision gas - 5, ion source gas 1 - 40psi, ion source gas 2 –60psi. For data acquisition, Sciex analyst Software version 1.5.2 was used.

Method Validation

The method validation parameters included specificity, accuracy, precision, limit of detection, limit of quantitation (LOD and LOQ), linearity of response, measurement uncertainty, robustness and suitability. The accuracy of the method was calculated by analyzing 5 spiked samples at 2 different spiking levels (10 and 100ppb) by different analysts and reported as a percentage of recovery.

The detection limit was determined as the lowest concentration of the residues (10ppb) that could be reproducibly measured under the method's operating conditions. Blank samples were also tested to check reagent interferences. Limit of quantification was calculated as the standard deviation (estimated in measuring limit of detection) of the lowest concentration level multiplied by a factor of 10. Linearity was calculated by spiking the target analytes at 5 different concentrations (0.01, 0.02, 0.05, 0.075, and 0.1mg/kg) to create a matrix-matched calibration curve. The relationship between response and concentration was measured by the correlation coefficient (R^2), which was set to ≥ 0.99 . The residual standard deviation is calculated and represented as bias%, which should be $\leq 20\%$. Repeatability, ($n=8$) and internal reproducibility ($n=24$) were determined as the relative standard deviations (%RSDs) of five replicate measurements on the same day and three consecutive days, respectively.

The measurement uncertainty at the 95% confidence level was calculated using the formula:

$$RSD (\%) = SD/X * 100$$

$$U (\%) = k * RSD (\%)$$

Where: U = uncertainty; k = coverage factor (For 95% confidence a factor of 2 is used); SD =standard deviation; X =Mean of concentration; RSD =relative standard deviation

Risk Assessment

To assess the risk associated with dietary exposure to residues through dates consumption, the following formula was used.

Dietary exposure =

$$\frac{\text{concentration of residue in mg /kg} \times \text{date fruit consumption (kg)}}{\text{body weight (kg)}}$$

The daily consumption pattern for the Emirati population was obtained from research study which reported an average daily consumption of 8 and 10 dates in the range of 72–114g (Ismail et al., 2006; Qazaq & Al Adeeb, 2010). The assessment considered that the average body weight of an adult was 70 kg since the targeted population was aged between 18-60 years.

Hazard Index (HI)

The collective risks associated with pesticide were

calculated through the hazard index (HI) method by adding the hazard quotients (HQ) of each pesticide.

$$HQ = \text{exposure}/ADI; HI = \sum HQ$$

Acceptable Daily Intake (ADI) = the amount of a substance (a pesticide residue or any contaminant) that can be consumed every day over a lifetime, without appreciable risk to health.

RESULTS AND DISCUSSION

The average recovery values of the analytes for 10 & 100µg/kg were 97.3 and 98.2% and the precision data showed an overall average of RSD < 20%. The selectivity of the method was evaluated by comparing the chromatograms of the matrix solution and the matrix-matched standard solution for each pesticide. It was observed that the relative intensities of the quantifier and qualifier ions fell within the tolerance level of $\pm 30\%$ compared to the reference standard. The calibration curve was constructed for each pesticide using 5 different concentration levels and the curves were best fitted to a linear matrix matched calibration with $R^2 \geq 95$ for all compounds.

The limit of detection (LOD) and limit of quantitation (LOQ) calculated from the standard deviation of the response (y-intercept) and slope of the calibration curves at 5 different concentrations suggest the suitability of the method for regulatory compliance (Table 1). The expanded measurement uncertainty calculated by multiplying the standard uncertainty (u) by a coverage factor (k) of 2 corresponding to a confidence level of 95% was below 50% suggesting that the data generated by the assay was reliable. The overall results indicate that the method has sufficient accuracy for the quantification of multiple residues in date fruit samples.

Table 1: Validation parameters of the pesticides

Test Name	R^2	Dynamic range (µg kg ⁻¹)	LOD (µg kg ⁻¹)	LOQ (µg kg ⁻¹)	Matrix effect (%)
Abamectin	0.998	10-50	6.858	22.860	-8.89
Acetamiprid	0.999	10-50	1.570	5.234	3.41
Acrinathrin	0.995	10-50	0.006	0.020	9.96
Carbendazim	0.997	10-50	3.441	11.470	-11.40
Chlorantraniliprole	0.999	10-50	3.075	10.249	-7.36
Chlorpyrifos	0.999	10-50	0.001	0.005	9.56
Clothianidin	0.998	10-50	2.139	7.129	4.24
Cypermethrin	0.995	10-50	0.005	0.017	8.44
Deltamethrin	0.999	10-50	0.005	0.017	6.44
Fenpyroximate	0.999	10-50	3.358	11.193	9.33
Fenvalerate	0.997	10-50	0.006	0.021	10.62
Fenazaquin	0.998	10-50	0.030	0.0900	5.62
Imidacloprid	0.998	10-50	2.778	9.260	2.62
Lambda Cyhalothrin	0.999	10-50	0.005	0.018	11.09
Matrine	0.997	10-50	0.004	0.013	7.41
Pyridaben	0.995	10-50	0.005	0.017	2.47
Spirodiclofen	0.999	10-50	2.226	7.420	16.90
Thiamethoxam	0.999	10-50	2.409	8.029	1.38

The dates samples from the Emirate of Abu Dhabi containing the pesticide and their quantities are presented in Table 2. Out of the 464 dates samples tested, 292 (63%) did not contain detectable quantity of pesticides while 172 samples (37%) were found to contain pesticides exceeding the regulatory limits (>MRL). About 70% of the samples

contained 2 pesticides while 26% had multiple residues. Very few samples (4%) had single pesticide residues. Deltamethrin of pyrethroid family was the predominant insecticide found in 41% of the positive samples. It is found effective against red palm weevil causing significant damage to date palm (Rasool et al., 2024). Cypermethrin is the second dominant insecticide accounting for 13% followed by Pyridaben and Lambda-Cyhalothrin with a detection frequency of 11% and 7% respectively. The remaining 11 pesticides had a frequency of Chlorpyrifos (4%), Spirodiclofen (3.5%), Thiamethoxam (3%), Carbendazim (3%), Imidacloprid (3%), Fenazaquin (2%), Fenvalerate (2%), Matrine (1.5%), Chlorantraniliprole (1%), Clothianidin (1%), Fenpyroximate (1%) and others 8 with a detection frequency (<1%) which included Acetamiprid, Abamectin, Acrinathrin and Ethion.

Table 2: Distribution and concentration of pesticides in dates

Pesticide	Range ($\mu\text{g kg}^{-1}$)	Mean	MRL
Carbendazim	0.16-1.21	0.24	0.1
Chlorantraniliprole	0.01-0.27	0.07	0.01
Chlorpyrifos	0.01-0.04	0.02	0.01
Clothianidin	0.01-0.02	0.01	0.01
Cypermethrin	0.01-0.9	0.09	0.05
Deltamethrin	0.01-0.3	0.03	0.01
Fenpyroximate	0.04-0.2	0.09	0.01
Fenvalerate	0.01-0.03	0.02	0.01
Fenazaquin	0.01-0.3	0.09	0.01
Imidacloprid	0.01-0.07	0.02	0.05
λ -Cyhalothrin	0.01-0.4	0.06	0.01
Matrine	0.01-0.014	0.01	0.01
Pyridaben	0.01-1.68	0.3	0.01
Spirodiclofen	0.01-0.63	0.08	0.02
Thiamethoxam	0.01-0.14	0.04	0.01

IN: insecticide; FU: fungicide; AC: acaricide

The current findings are comparable to a previous study conducted to monitor pesticide residues in imported dates in the UAE. The study reported 11 pesticides exceeding the permissible limits (>MRL) which included Acetamiprid, Carbendazim, Deltamethrin, Ethion, Fipronil, Imidacloprid, Pyriproxyfen and Spirodiclofen. The most frequently detected pesticides were Acetamiprid, Deltamethrin and Indoxacarb. The study also reported more than one pesticide in many samples, corroborating with a previous study (Osaili et al., 2022). In another study comprising samples from imported fresh fruits in the UAE, Chlorpyrifos, Carbendazim, Cypermethrin and azoxystrobin were frequently detected exceeding the MRL (Abd El-Mageed et al., 2021). A study from Egypt on 3 dates cultivars comprising more than 200 samples reported pesticides residues in more than 50% of the sample of which 25.29% exceeded MRL. The most frequently detected pesticide residues were Malathion followed by Chlorpyrifos, Cypermethrin, Cyfluthrin and Carbendazim (Ahmed et al., 2022). Recently, health risk determination of Carbosulfan, Phenmedipham, Carbaryl, Propoxur, Propamocarb, Aminocarb, Ethiofencarb, Pirimicarb, Bendiocarb, Fenoxycarb, Carbofuran, Methomyl, Desmedipham and Methiocarbamate residues in date palm fruits was evaluated in 55 samples from various farms and market across UAE. According to this study, most of the studied carbamate residues were below the maximum

residue levels except for carbosulfan, propoxur and carbofuran. However, assessment of health risks associated with some of the detected carbamates revealed HI <1.0 indicating that date adult consumers applied in this study are not at risk (Morsi et al., 2024). A similar finding was observed in our study with Carbendazim having low health risk index. All these studies stress the significance of regular monitoring and its value in food safety management. According to the data from a study in Iran, inclusion of washing date fruits with distilled water has been found to reduce the amount of organophosphorus pesticide residues in both ripe and unripe fruits. Statistically significant differences in the levels of pesticides in both unripe and ripe dates before and after washing were observed suggesting the importance of washing to reduce the pesticide exposure as observed in many other studies (Wu et al., 2019; Acoglu and Omeroglu 2021; Mosallaei et al., 2024) involving other fruits and vegetables.

A considerable amount of reduction ranging from 3% and up to 68% has been observed. It is worthwhile considering the application of Good Agriculture Practices (GAP), which is a collection of principles to apply for on-farm production and post-production processes, resulting in safe and healthy food and non-food agriculture products, while considering economic, social and environmental sustainability (Arabameri et al., 2022).

With regards to the risk associated with the use of pesticides and its residue, the current study analyzed 464 samples in which no traces of residue were detectable (ND) in 292 samples (63%) suggesting no or negligible exposure. The exposure levels to various pesticides and corresponding hazard index are presented in Table 3. It is evident from the data that all the pesticides detected have hazard index values below 1 at the 95th percentile, indicating that the estimated exposure levels are safe for the majority of the population. However, Pyridaben and Spirodiclofen show relatively higher hazard index values (0.1718 and 0.1537, respectively) than those of other pesticides. Though it is well below 1, these pesticides may pose a higher risk and require closer monitoring. On the contrary, Imidacloprid, Acetamiprid, and Fenvalerate have very low hazard index values (0.0008, 0.0005, and 0.0015, respectively), indicating minimal risk at the current exposure levels.

Table 3: Residue levels hazard index

Pesticide	Residue level	Dietary Exposure	Hazard Index
Deltamethrin	0.03365	5.43×10^5	0.0054
Cypermethrin	0.08087	1.31×10^4	0.0131
Chlorpyrifos	0.01785	2.88×10^5	0.0288
Pyridaben	0.27443	4.43×10^4	0.1477
λ -Cyhalothrin	0.05373	8.67×10^5	0.0173
Fenpyroximate	0.09480	1.53×10^4	0.0765
Fenvalerate	0.01650	2.66×10^5	0.0013
Fenazaquin	0.09200	1.49×10^4	0.0297
Imidacloprid	0.02415	3.90×10^5	0.0006
Matrine	0.01171	1.89×10^5	0.0019
Spirodiclofen	0.08188	1.32×10^4	0.1322
Thiamethoxam	0.03800	6.13×10^5	0.0123
Abamectin	0.01350	2.18×10^5	0.0109
Acetamiprid	0.01867	3.01×10^5	0.0004
Acrinathrin	0.02725	4.40×10^5	0.0147
Carbendazim	0.22550	3.64×10^4	0.0364

Conclusion

Assessment of date fruits from the UAE market revealed no measurable pesticide residues in 292 (63%) samples while 172 samples (37%) were above the regulatory limits. Deltamethrin was the most frequent insecticide found followed by Cypermethrin, Pyridaben, Lambda-Cyhalothrin, Chlorpyrifos, Spirodiclofen, Thiamethoxam, Carbendazim, Imidacloprid, Fenazaquin, Fenvalerate, Matrine, Chlorantraniliprole, Clothianidin Fenpyroximat, Acetamiprid, Abamectin, Acirathrin and Ethion. The cumulative exposure for pesticides through consumption of date fruit was in the range of 8.67×10^5 to 1.3×10^4 mg for adults. Overall hazard index indicates that the dietary exposure to the assessed pesticides is within acceptable safety limits for most of the population. However, specific pesticides like Pyridaben and Spirodiclofen warrant closer attention due to their relatively higher hazard indices. Regulatory bodies should consider these findings to ensure continued protection of public health and to address any potential risks associated with pesticide exposure.

DECLARATIONS

Funding: This study did not get any financial support from any organization/agency.

Acknowledgement: The authors gratefully acknowledge the support and encouragement of the Abu Dhabi Quality and Conformity Council. Gratitude is also extended to the management of Central Testing Laboratories (CTL) for invaluable support.

Competing Interest: The authors declare that they have no known competing financial interests or personal relationships that could influence the work reported in this paper.

Conflict of Interest: The authors declare no conflict of interest.

Data Availability: All the data is available in the article.

Ethics Statement: This study did not involve humans or animals and therefore did not require official ethical approval.

Author's Contribution: Abdullah Siddiqui and Ali Abdallah Sultan Al Marzouqi were involved in method validation and sample analysis; Ayeda assisted in method validation and interpreting the results. Premanandh Jagadeesan, the corresponding author participated in data processing, analysis and in the preparation of the manuscript.

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