Türk. entomol. derg., 2024, 48 (3): 319-326
DOI: http://dx.doi.org/10.16970/entoted.1510753

ISSN 1010-6960
E-ISSN 2536-491X

Original article (Orijinal araştırma)

Investigation of insecticide residues in fig and health risk assessment[1]

İncirlerde insektisit kalıntılarının araştırılması ve sağlık risk değerlendirmesi

Ali YELADI[2] **Kenan KARA[2]** **Tarık BALKAN[2*]**

Abstract

This study aimed to detect insecticide, acaricide, and nematicide residues in 92 fresh and 50 dried fig samples collected from locations with intensive fig, *Ficus carica* L. (Rosales: Moraceae) production in Aydın, Türkiye in 2022. The analysis method was validated according to SANTE 11312/2021 guidelines. The recoveries ranged between 70% and 120%, with repeatability (RSDr) and within-laboratory reproducibility (RSDwR) ≤ 20% and expanded measurement uncertainties below 50% for all insecticides indicating satisfactory analytical performance. In total, 114 different insecticides were screened, revealing residues in 27 samples. Bifenazate was detected in 13 samples, etoxazole in 3 samples, spiromesifen in 5 samples, and both bifenazate and spiromesifen in 6 samples. No detectable residues were found in the dried fig samples. All samples in which bifenazate was detected were above the European Union Maximum Residue Limits (EU-MRL). Etoxazole and spiromesifen, unauthorized in figs, showed etoxazole residues above EU-MRLs, while spiromesifen residues were below EU-MRLs. The acute and chronic health risk indices for the insecticides were found to be below 1, indicating low health risks associated with fig consumption. The risk assessment suggests that fig consumption is safe for consumers.

Keywords: Dried fig, fig, LC-MS/MS, pesticide residue, QuEChERS

Öz

Bu çalışma 2022 yılında Aydın ilinde yoğun incir, *Ficus carica* L. (Rosales: Moraceae) üretiminin yapıldığı lokasyonlardan alınan 92 yaş ve 50 kuru incir numunesinde insektisit, akarisit ve nematisit kalıntılarının tespiti için gerçekleştirilmiştir. Analiz metodu SANTE 11312/2021'e göre doğrulanmıştır. Ortalama geri kazanımlar %73,2 ile %119,6 arasında olup yöntemin tekrarlanabilirliği (RSDr) ve laboratuvar içi tekrar üretilebilirliği (RSDwR) için ≤ %20 ve genişletilmiş ölçüm belirsizlikleri %50'nin altındadır. Bu veriler tatmin edici analitik performansı göstermektedir. Toplamda 114 farklı insektisitin tarandığı çalışmada, 92 yaş incir numunesinin 13'ünde bifenazate, 3'ünde etoxazole, 5'inde spiromesifen, 6'sında ise hem bifenazate hem spiromesifen olmak üzere toplam 27 numunede pestisit kalıntısı kaydedilmiştir. Kuru incir örneklerinde tespit edilebilir kalıntıya rastlanmamıştır. Bifenazate tespit edilen örneklerin tamamı Avrupa Birliği Maksimum Kalıntı Limitlerinin (AB-MRL) üzerindedir. Etoxazole ve spiromesifen etken maddeleri incirde ruhsatsız olup, numunelerde tespit edilen etoxazole kalıntıları AB-MRL üzeri, spiromesifen ise AB-MRL altıdır. Tespit edilen insektisitlerin akut ve kronik sağlık risk indeksi 1'den düşük bulunmuştur. Risk değerlendirmesi incir tüketiminin tüketiciler için güvenli olduğunu göstermiştir.

Anahtar sözcükler: Kuru incir, incir, LC-MS/MS, pestisit kalıntısı, QuEChERS

[1] This study was a part of the Master thesis of the first author, and supported by Tokat Gaziosmanpaşa University, Scientific Research Unit, Tokat, Türkiye, Grant Project No: 2022/112.
[2] Tokat Gaziosmanpaşa University, Faculty of Agriculture, Department of Plant Protection, 60250, Tokat, Türkiye
* Corresponding author (Sorumlu yazar) e-mail: tarik.balkan@gop.edu.tr
Received (Alınış): 05.07.2024 Accepted (Kabul ediliş): 05.10.2024 Published Online (Çevrimiçi Yayın Tarihi): 06.10.2024

Introduction

Fig, *Ficus carica* L. (Rosales: Moraceae) is an important agricultural product with a trademark value for Türkiye, and in 2006, it obtained a geographical indication specific to the province of Aydın. Figs are fruits that are characteristic of the Mediterranean climate, spreading from Anatolia to the whole world. Global fig production amounted to 1.321 million tons in 296 thousand hectares in 2021. Türkiye ranks first in fig production with 320 thousand tons, followed by Egypt with 211 thousand tons, and Morocco holds the third position with 144 thousand tons of production (FAO, 2021). Of the 148 thousand tons of dried figs produced worldwide in 2020/2021, 85.5 thousand (58%) were produced in Türkiye, 25 thousand tons (17%) in Iran, and 10 thousand tons (7%) in Spain. Türkiye holds the largest share of dried fig exports in Europe. The export of dried figs in 2020 amounted to about 43 thousand tons, with revenues reaching $158 million. Approximately 68 thousand tons of dried figs were exported in 2021, generating $263 million, while in 2022, exports increased to 70 thousand tons, contributing $278 million in foreign exchange earnings (Hasdemir, 2022).

The fig is affected by several important pests such as *Ceroplastes rusci* (L., 1758) (Hemiptera: Coccidae), *Tetranychus urticae* Koch,1836 (Acari: Tetranychidae), *Carpophilus* spp. Stephens, 1829 (Coleoptera: Nitidulidae), *Drosophila* spp. Fallen, 1823 (Diptera: Drosophilidae), *Otiorhynchus dauricus* Stierlin, 1862 (Coleoptera: Curculionidae), and *Poecilimon sanctipauli* Brunner von Wattenwyl, 1878 (Orthoptera: Tettigoniidae) (Akşit et al., 2003). Due to constantly changing climate and weather conditions in recent years, intensive pesticide applications are sometimes necessary to combat these pests. Although licensed (pyriproxyfen and spinosad) or temporarily licensed (azadirachtin-a, cyantraniliprole, deltamethrin, and pyrethrin) insecticides are applied against the pests (Anonymous, 2024), some producers prefer unlicensed pesticides. The use of these toxic chemicals leads to residue problems in products and can cause health issues for both applicators and consumers in the long term. Hence, different organizations regulate pesticide residues by setting maximum residue limits (MRLs), aiming to mitigate health risks (EU-MRL, 2022; TGK-MRL, 2022). The regulatory framework governing pesticide residue levels in food products within European Union countries is defined by European Parliament and Council Regulation No. 396/2005 (EC, 2005), which establishes MRLs. Türkiye has implemented the Turkish Food Codex Regulation to regulate MRLs in food products, aligning with EU regulations as part of efforts to harmonize with European Union legislation (Anonymous, 2023).

Studies on monitoring of pesticide residue in fig, are important for food safety, and these studies also help to evaluate the potential risks on consumers' health. The Rapid Alert System for Food and Feed (RASFF) reported that chlorpyrifos-methyl and cypermethrin were detected in dried figs (RASFF, 2024). Yet studies reporting on risk assessment and pesticide residues in figs are limited (Aydin & Ulvi, 2019; Soydan et al., 2021; Rosas-Sánchez et al. 2023).

This study aims to evaluate the presence of insecticide, acaricide, and nematicide residues in fig sampled in Aydın, Türkiye. For this purpose, method verification was conducted following the SANTE guidelines (SANTE/11312/2021) to detect and quantify 114 active ingredients in samples prepared for analysis with the QuEChERS (quick, easy, cheap, effective, rugged, and safe) sample preparation method. The validated method was successfully applied to analyze 92 fresh and 50 dried fig samples collected in Aydın provinces in September and October 2022. In addition, the potential health risks posed by pesticide residues in figs were evaluated.

Materials and Methods

Chemicals and reagents

The pesticide reference standards were supplied by Dr. Ehrenstorfer GmbH (Augsburg, Germany). Methanol (CH_4O) and acetonitrile (C_2H_3N), both of gradient grade for liquid chromatography (≥99.9% purity), acetic acid ($C_2H_4O_2$ >99% purity), and ammonium formate (CH_5NO_2 ≥99% purity) were procured

from Merck (Darmstadt, Germany). QuEChERS materials were purchased from Restek (Bellefonte, PA, USA). QuEChERS extraction [6 g magnesium sulfate anhydrous ($MgSO_4$ >98% purity); 1.5 g sodium acetate anhydrous ($C_2H_3NaO_2$ ≥99% purity)] and clean-up kits [1.2 g $MgSO_4$, 400 mg primary and secondary amines (PSA, 40 µm particle size) and 400 mg C_{18}] were used.

Sample collection and storage

Insecticide, acaricide, and nematicide residue analysis was conducted on 92 fresh and 50 dried figs collected from Aydın, Türkiye in 2022. Harvest-ripe fresh and sun-dried figs were gathered from five locations (Nazilli, Germencik, İncirliova, Köşk, and Bozdoğan) known for their high commercial production. The number of fresh and dried fig samples taken from Nazilli, Germencik, incirliova, Köşk and Bozdoğan are 38-19, 24-12, 12-8, 10-6 and 8-5, respectively. According to the Commission Directive 2002/63/EC, fresh fig samples from the medium-sized fruit and berry fruit category were collected weighing 1 kg (at least 10 units), while dried fig samples from the processed plant products category were collected weighing 0.5 kg (EC, 2002). Samples were promptly stored in the vehicle's refrigerator under appropriate conditions and delivered to the laboratory in polyethylene bags within a maximum of 24 hours. Upon arrival, the samples were kept in a deep freeze at -18°C until they were ready for analysis.

Sample preparation, extraction, and clean-up

The extraction and clean-up steps were performed following the QuEChERS AOAC Method 2007.01, as described by AOAC (2007). A 4-blade blender (Groupe SEB, France) was used to homogenize the fig samples. Figure 1 provides an illustration of the QuEChERS protocols. Analyses of both fresh figs and dried figs were conducted in triplicate using LC-MS/MS.

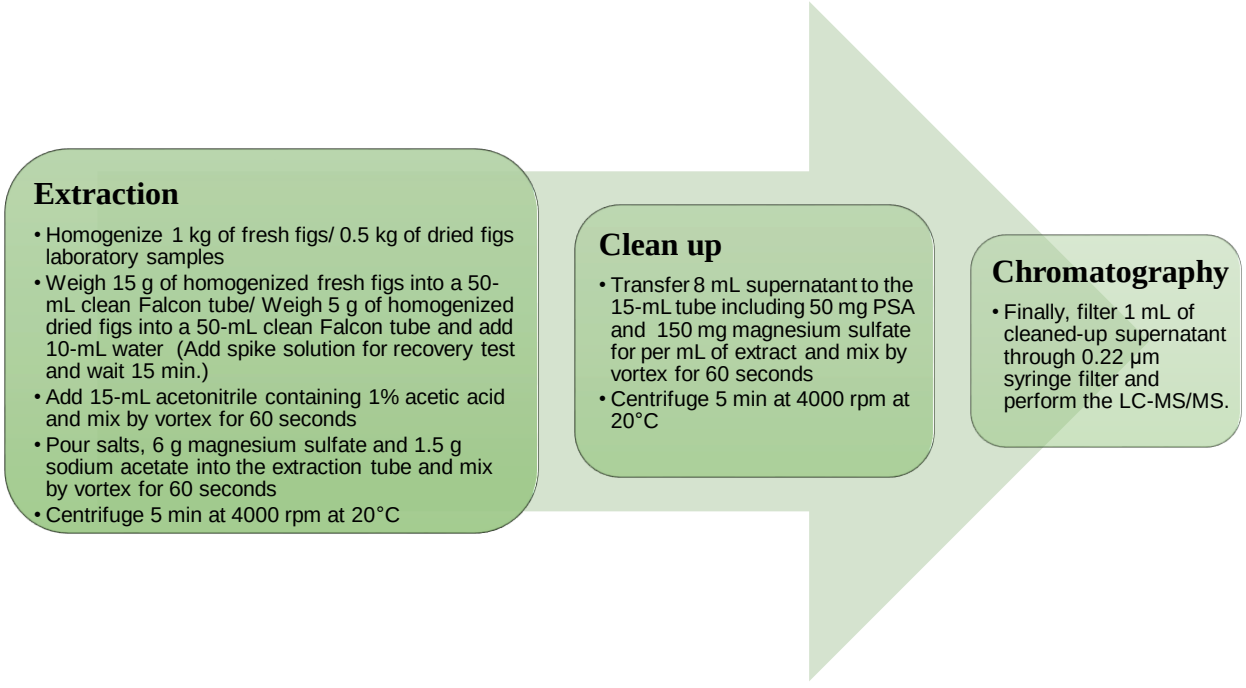

Figure 1. Analytical steps of the QuEChERS-AOAC Official Method 2007.01.

Instrumentation and optimization for LC-MS/MS

A Shimadzu UHPLC Nexera X2 system coupled with an LCMS-8050 triple quadrupole mass spectrometer equipped with electrospray ionization (ESI) was employed for the analyses. The LC column was C_{18} Inertsil (ODS-IV) column (2.1 mm × 150 mm, 3 µm particle size) (GL Sciences, Japonya). The mobile phase comprised 10 mmol/L CH_5NO_2 in distilled H_2O (A) and CH_4O (B), with a flow rate of 0.4 mL/min. Throughout the analysis, the oven temperature remained steady at 50°C, and each injection volume was set at 10 µL. Supplementary operational parameters encompassed collision-induced dissociation (CID) gas pressure set at 230 kPa, a nitrogen (N_2) drying gas flow rate of 10 L/min, nebulizer gas flow rate of 3 L/min, heating gas flow rate of 10 L/min, desolvation line temperature maintained at 250°C, interface temperature set to 300°C, block heater temperature of 400°C, capillary voltage adjusted to 4.5 kV, and dwell time ranging from 1 to 33 ms.

A total of 114 insecticides, acaricides, nematicides, and their metabolites were screened. Optimization parameters for 111 of these compounds were meticulously provided by Balkan & Yılmaz (2022a), whereas supplementary optimization parameters concerning three additional agents are delineated in Table 1. LC-MS/MS optimization employed multiple reaction monitoring (MRM).

Table 1. Optimization of LC-MS/MS parameters of 3 pesticides in the MRM mode

Analyte	Type of pesticide*	Substance group*	Molecular formula*	Retention time (min)	Ion mode	Precursor ion (m/z)	Product ion (m/z)	Dwell time (msec)	Q1 Pre Bias (V)	CE (V)	Q3 Pre Bias (V)
Bifenazate	Insecticide, Acaricide	Carbazate	$C_{17}H_{20}N_2O_3$	8.437	pos	301.10	170.0	3.0	-15.0	-14.0	-18.0
						301.10	152.1	3.0	-15.0	-38.0	-29.0
						301.10	198.1	3.0	-15.0	-6.0	-20.0
Etoxazole	Acaricide	Diphenyl oxazoline	$C_{21}H_{23}F_2NO_2$	10.753	pos	360.10	141.0	1.0	-27.0	-27.0	-25.0
						360.10	304.1	1.0	-27.0	-17.0	-20.0
						360.10	57.0	1.0	-27.0	-28.0	-22.0
Spiromesifen	Insecticide, Acaricide	Tetronic acid	$C_{23}H_{30}O_4$	10.679	pos	371.20	273.2	2.0	-18.0	-10.0	-28.0
						371.20	255.2	2.0	-25.0	-23.0	-26.0

*IUPAC Pesticides Properties DataBase (IUPAC, 2024).

Method verification

To assess recovery, 15 g of pesticide-free fig samples were spiked with mixed pesticide solutions at concentrations of 0.01 and 0.05 mg/kg, each replicated five times. This experiment was conducted weekly for five consecutive weeks with the participation of two analysts.

The analytical methods were verified following internationally recognized guidelines (EURACHEM, 2014; SANTE, 2021). Key parameters assessed included limit of detection and quantification (LOD / LOQ), linearity, recovery efficiency, precision (both repeatability; RSDr and within-laboratory reproducibility; RSDwR), measurement uncertainty (U'). The expanded U' was computed for all insecticides using Approach 2, which involves estimating a generic measurement uncertainty based on proficiency test data (PT), as recommended in the SANTE Guideline (SANTE, 2021). Further are provided in the study by Balkan & Yılmaz (2022b).

Pesticide residues in figs

One hundred and fourteen pesticides were analyzed in 92 fresh fig and 50 dried fig samples using LC-MS/MS. The detection of pesticides in these samples was confirmed based on retention time and ion ratio, as defined in the identification criteria outlined in the SANTE guidelines (SANTE, 2021).

To evaluate the potential health risks, we compared the estimated dietary exposure (per unit of body weight, bw) to established toxicological benchmarks such as the acute reference dose (ARfD, mg kg bw^{-1} d^{-1}) and acceptable daily intake (ADI, mg kg bw^{-1} d^{-1}). The acute/short-term consumer health risk (aHI) was

assessed using the estimated short-term intake (ESTI, mg kg^{-1} d^{-1}) divided by the ARfD. Conversely, the chronic/long-term consumer health risk (chronic hazard index, cHI) was determined by comparing the estimated daily intake (EDI, mg kg^{-1} d^{-1}) to the ADI (EFSA, 2015). Following equations were used for these calculations (Liu et al.,2016);

$$ESTI = \text{the highest residue level} \times \text{food consumption} / bw \quad (1)$$

$$aHI = ESTI / ARfD \quad (2)$$

$$EDI = \text{the mean residue level} \times \text{food consumption} / bw \quad (3)$$

$$cHI = EDI / ADI \quad (4)$$

The average body weight of an adult was taken as 73.7 kg (TUIK, 2023). Daily fig consumption for the general population in Türkiye was reported as 19 g per day (TUIK, 2022). In risk assessment, when the Hazard Index (HI) exceeds one, it indicates potential health risks from pesticide residues to consumers (Akoto et al., 2015; Soydan et al., 2021).

Result and Discussion

Method verification

The outcomes of the method verification studies pertaining to the identified pesticides were delineated in Table 2. Satisfactory linearities were observed with correlation coefficients (R^2) from 0.990 to 0.998. The LOD and LOQ values of the analytical method were calculated in accordance with the Eurachem Guide (EURACHEM, 2014). Blank fig samples were spiked with an insecticide mixture solution at the level of 10 µg/kg and 10 replicate analyses were performed. The LOQ were found to be below the maximum residue limits (MRLs) established by the EU for figs. With the two-level spiking, the recovery rate of 70-120%, and repeatability (RSDr) and intra-laboratory reproducibility (RSDwR) ≤ 20% for insecticides, were found. Furthermore, expanded measurement uncertainties were below 50% for all insecticides. These findings underscore the efficacy of the QuEChERS method as a rapid and accurate technique for insecticide residue analysis in figs.

Table 2. Method verification parameters of detected active ingredients in fig samples

Active ingredient	LOD (µg/kg)	LOQ (µg/kg)	R^2	Repeatability (n=10)				Reproducibility (n=10)				U' %
				10 µg/kg		50 µg/kg		10 µg/kg		50 µg/kg		
				Rec.%	RSDr%	Rec.%	RSDr%	Rec.%	RSDwR%	Rec.%	RSDwR%	
Bifenazate	1.23	4.11	0.995	102.17	16.01	99.91	4.80	105.37	10.91	99.00	4.49	20.03
Etoxazole	1.68	5.60	0.998	84.93	3.75	105.34	2.01	74.55	4.32	95.28	2.94	15.58
Spiromesifen	2.09	6.97	0.990	113.26	16.78	107.17	8.72	104.02	14.03	108.86	6.41	27.04

Rec: Recovery; LOD: limit of detection; LOQ: limit of quantification; U': expanded measurement uncertainty.

Residue analyses in figs

Insecticide residue analysis was conducted on fig samples collected from the districts of Nazilli, Germencik, İncirliova, Köşk, and Bozdoğan in Aydın province. A total of 142 fig samples were analyzed. In the study, a total of 114 different insecticides were monitored, and no detectable residues were found in dried fig samples. However, insecticide residues exceeding the limit of quantification (LOQ) were detected in 27 out of 92 samples. The number of fresh fig samples exceeding the EU-MRLs was 16 (Table 3).

Table 3. Pesticides residues (mg/kg) detected in figs

Food commodity	Number of samples >LOQ and percentage (%)	Number of samples >MRL and percentage (%)	Pesticide	Frequency of detection	Pesticide residue range (mg kg^{-1})	Number of samples >MRL	MRL* (mg kg^{-1})
Fig	27 (29.3)	16 (17.4)	Bifenazate	19	0.013-0.153	13	0.02
			Etoxazole	3	0.063-0.031	3	0.01
			Spiromesifen	11	0.010-0.011	-	0.02
Dried fig	-	-	-				

*EU-MRL.

During the period from August 2 to September 30, 2022, bifenazate received temporary approval from the Ministry of Agriculture and Forestry of the Republic of Türkiye for use in figs (Hasdemir, 2022). However, etoxazole and spiromesifen are not authorized for figs. Residue levels exceeding the EU-MRL were detected for bifenazate in 13 samples and for etoxazole in 3 samples (Table 3). These findings were evaluated in accordance with EU-MRL standards. These results were assessed based on EU-MRL. The occurrence of bifenazate residues above the MRL may be attributed to factors such as repeated applications, excessive dosages, or non-compliance with the time interval between the last pesticide application and harvest. Nevertheless, the use of unauthorized pesticides may also be attributed to producers' unawareness and a solution-focused approach.

The studies regarding pesticide residue in figs are quite limited. Tatlı (2006) investigated pesticide residues in 5 fresh and 10 dried fig samples produced in the Aegean Region and reported that no pesticide residues were found in the fresh and dried fig samples. Aydin & Ulvi (2019) determined only two insecticides, chlorpyriphos and malathion, in 2 of 14 dried fig samples. Soydan et al. (2021) investigated pesticide residues in 900 dried fig samples in their study in the Aegean Region. They reported that no residues above LOD were found in 846 of these figs. They found residues exceeding EU-MRLs in 38 samples, while 16 samples had residue levels equal to or below the MRLs. Their residue analysis identified various active ingredients, including α-cypermethrin, α-endosulfan, bromopropylate, carbendazim, chlorpyrifos, chlorpyrifos-methyl, cypermethrin, deltamethrin, dichlorvos, dimethoate, λ-cyhalothrin, lufenuron, malathion, metalaxyl m, methoxychlor, permethrin, and triadimefon. Additionally, they found one pesticide in 48 samples, two pesticides in three samples, and five or more pesticides in three samples. However, in our study, no pesticide residues above the limit of detection (LOD) were found in dried figs.

Health risk assessment

Pesticide hazard evaluations have attracted considerable consumer attention in recent years, particularly in Türkiye (Çatak & Tiryaki, 2020; Soydan et al., 2021; Balkan & Kara, 2022; Serbes & Tiryaki, 2023). Health risk analyses were performed for three pesticides (Table 4). The calculations revealed that both acute (aHI) and chronic (cHI) health risk values were below 1, indicating the absence of health risks.

Table 4. Health risk estimation of insecticides residues in figs in Türkiye

Insecticide	ADI* (mg kg bw^{-1} d^{-1})	ARfD* (mg kg bw^{-1} d^{-1})	ESTI (mg kg^{-1} d^{-1})	aHI	EDI (mg kg^{-1} d^{-1})	cHI
Bifenazate	0.01	0.1	3.98E-06	0.00398	1.16E-06	0.0116
Etoxazole	0.04	/	8.14E-06	/	3.95E-06	0.0099
Spiromesifen	0.03	2	2.86E-07	0.00001	2.60E-07	0.0009

* ADI and ARfD values are from the IUPAC Pesticides Properties DataBase (IUPAC, 2024).

Literature regarding health risk assessment in figs is extremely limited. Rosas-Sánchez et al. (2023) conducted a study and did not find any health risk associated with the insecticides cypermethrin and permethrin in figs in Mexico.

Conclusion

This study investigates the presence of insecticide, acaricide, nematicide, and their metabolite residues in figs sourced from local markets in Aydın, Türkiye, in 2022. Furthermore, it evaluates the health risk posed by these residues to consumers. The research highlights that implementing conscientious and sustainable agricultural practices to reduce pesticide residues in figs. This encompasses the promotion of integrated pest management (IPM) strategies and the exploration of alternative pest control methodologies. The study underscores the necessity of strict adherence to pesticide residue regulations and standards within the agricultural sector. It encourages the agricultural industry to prioritize consumer safety and comply with established maximum residue limits (MRLs) for pesticides. The findings highlight the importance of ongoing research and monitoring of pesticide residues in figs and other agricultural products. This supports the continuous improvement of agricultural practices and ensures the safety of food products for consumers. Overall, the research has implications for promoting consumer awareness, encouraging responsible agricultural practices, and fostering regulatory compliance within the fig industry in Aydın, Türkiye.

Acknowledgments

We are grateful to Tokat Gaziosmanpaşa University Scientific Research Projects Coordination Unit for financial support.

References

Akoto, O., S. Gavor, M. K. Appah & J. Apau, 2015. Estimation of human health risk associated with the consumption of pesticide-contaminated vegetables from Kumasi Ghana. Environmental Monitoring and Assessment, 187 (5): 1-9.

Akşit, T., F. Özsemerci & İ. Çakmak, 2003. Aydın ilinde incir ağaçlarında saptanan zararlı türler. Turkish Journal of Entomology, 27 (3): 181-189 (in Turkish with abstract in English).

Anonymous, 2023. Türk gıda kodeksi pestisitlerin maksimum kalıntı limitleri yönetmeliği. (Web page: https://www.resmigazete.gov.tr/eskiler/2021/09/20210927M1-1.htm) (Date accessed: November 2023) (in Turkish).

Anonymous, 2024. Plant Protection Products Database. (Web page: https://bku.tarimorman.gov.tr/Arama/Index?csrt=11430343323884281979) (Date accessed: September 2024) (in Turkish).

AOAC, 2007. AOAC Official Method: Pesticide residues in foods by acetonitrile extraction and partitioning with magnesium sulfate gas chromatography/mass spectrometry and liquid chromatography/tandem mass spectrometry. First Action 2007. (Web page: https://nucleus.iaea.org/sites/fcris/Shared%20Documents/SOP/AOAC_2007_01.pdf) (Date accessed: September 2024).

Aydin, S. & M. Ulvi, 2019. Residue levels of pesticides in nuts and risk assessment for consumers. Quality Assurance and Safety of Crops & Foods, 11 (6): 539-548.

Balkan T. & Ö. Yılmaz, 2022a. Investigation of insecticide residues in potato grown in Türkiye by LC-MS/MS and GC-MS and health risk assessment. Turkish Journal of Entomology, 46 (4): 481-500.

Balkan T. & Ö. Yılmaz, 2022b. Method validation, residue and risk assessment of 260 pesticides in some leafy vegetables using liquid chromatography coupled to tandem mass spectrometry. Food Chemistry, 384 (2022): 132516 (1-21).

Balkan, T. & K. Kara, 2022. Determination of pesticide residues and risk assessment in some vegetables grown in Tokat province. Plant Protection Bulletin, 62 (2): 26-35.

Çatak, H. & O. Tiryaki, 2020. Insecticide residue analyses in cucumbers sampled from Çanakkale open markets. Turkish Journal of Entomology, 44 (4): 449-460.

EC, 2002. European Commission: Commission Directive 2002/63/EC of 11 July 2002 establishing Community methods of sampling for the official control of pesticide residues in and on products of plant and animal origin and repealing Directive 79/700/EEC. Official Journal of the European Communities, L 187 (45): 30-43.

EC, 2005. European Commission: Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC Text with EEA relevance. Official Journal of the European Communities, L 70 (48): 1-16.

EFSA, 2015. European Food Safety Authority: Revisiting the International Estimate of Short-Term Intake (IESTI equations) used to estimate the acute exposure to pesticide residues via food. EFSA Supporting Publication, 12 (12): 1-81.

EU-MRL, 2022. European Union (EU-MRL) Pesticides Database: Pesticide Residues MRLs. Directorate General for Health & Consumers. (Web page: https://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/mrls/?event=search.pr) (Date accessed: November 2022).

EURACHEM, 2014. Eurachem Guide: The Fitness for Purpose of Analytical Methods – A Laboratory Guide to Method Validation and Related Topics, (2nd Ed. 2014). ISBN 978-91-87461-59-0. (Web page: https://www.eurachem.org/images/stories/Guides/pdf/MV_guide_2nd_ed_EN.pdf) (Date accessed: December 2022).

FAO, 2021. FAOSTAT, Crops and livestock products. (Web page: https://www.fao.org/faostat/en/#data/QCL) (Date accessed: September 2024).

Hasdemir, M., 2022. Agricultural Products Market, Fig. (Web page: https://arastirma.tarimorman.gov.tr/tepge/Belgeler/PDF%20Tar%C4%B1m%20%C3%9Cr%C3%BCnleri%20Piyasalar%C4%B1/2023-Temmuz%20Tar%C4%B1m%20%C3%9Cr%C3%BCnleri%20Raporu/%C4%B0NC%C4%B0R%20T%C3%9CP%20HAZ%C4%B0RAN%202023-TEPGE.pdf) (Date accessed: November 2023).

IUPAC, 2024. The PPDB-Pesticide properties database, international union of pure and applied chemistry. (Web page: http://sitem.herts.ac.uk/aeru/iupac/index.htm) (Date accessed: September 2024).

Liu, Y., D. Shen, S. Li, Z. Ni, M. Ding, C. Ye & F. Tang, 2016. Residue levels and risk assessment of pesticides in nuts of China. Chemosphere, 144 (2016): 645-651.

RASFF, 2024. Rapid Alert System for Food and Feed. (Web page: https://food.ec.europa.eu/safety/rasff-food-and-feed-safety-alerts_en) (Date accessed: September 2024).

Rosas-Sánchez, J., H. Saldarriaga-Noreña, L. A. Chávez-Almazán, M. A. Murillo-Tovar, I. I. Ramírez-Bustos, V. A. Leon-Hernández & E. Fernández-Herrera, 2023. Occurrence of pesticide residues in fig crops (*Ficus carica* L.), in Morelos, Mexico. Revista Internacional de Contaminación Ambiental, 39 (2023): 215-227.

SANTE, 2021. SANTE/11312/2021, Analytical quality control and method validation procedures for pesticide residues analysis in food and feed, 1-55. (Web page: https://food.ec.europa.eu/system/files/2022-02/pesticides_mrl_guidelines_wrkdoc_2021-11312.pdf) (Date accessed: September 2022).

Serbes, E. B. & O. Tiryaki, 2023. Determination of insecticide residues in “Bayramiç Beyazı” nectarines and their risk analysis for consumers. Turkish Journal of Entomology, 47 (1): 73-85.

Soydan, K. D., N. Turgut, M. Yalçın, C. Turgut & P. B. K. Karakuş, 2021. Evaluation of pesticide residues in fruits and vegetables from the Aegean region of Turkey and assessment of risk to consumers. Environmental Science and Pollution Research, 28 (22): 27511-27519.

Tatlı, Ö., 2006. Ege Bölgesine Özgü Bazı Yaş Meyve, Sebze ve Kurutulmuş Gıda Ürünlerinde Pestisit Kalıntı Düzeylerinin Tespiti. Çukurova Üniversitesi Fen Bilimleri Enstitüsü, Toprak Anabilim Dalı, (Unpublished) Yüksek Lisans Tezi, Adana, 120 s (in Turkish with abstract in English).

TGK-MRL, 2022. Republic of Turkey, ministry of food, agriculture and livestock, general directorate of food and control, department of plant protection products, MRL rates. (Web page: https://bku.tarimorman.gov.tr/MRLOrani/Index?csrt=2789820862056200932) (Date accessed: November 2022).

TUIK, 2022. Turkish Statistical Institute. Poultry production statistics. (Web page: https://biruni.tuik.gov.tr/medas/?kn=80&locale=en) (Date accessed: November 2022).

TUIK, 2023. Turkey Health Interview Survey. (Web page: https://data.tuik.gov.tr/Bulten/DownloadIstatistikselTablo?p=WEBW229PP/91tMV2m71fU6pRWq2F1ZD/lzOFFk0bNDi2rjAC8QDCRN62nr2M3n1K) (Date accessed: November 2023).