GIDA
THE JOURNAL OF FOOD
E-ISSN 1309-6273, ISSN 1300-3070

Research/Araştırma
GIDA (2023) 48 (5) 993-1003
doi: 10.15237/gida.GD23063

DETERMINATION OF PESTICIDE RESIDUES IN POMEGRANATES GROWN IN ANTALYA AND HEALTH RISK ASSESSMENT

Tarık Balkan*, Özlem Yılmaz
Department of Plant Protection, Faculty of Agriculture, Tokat Gaziosmanpaşa University, Tokat, Türkiye

Received /Geliş: 22.05.2023; *Accepted* / Kabul: 23.06.2023; *Published online* / Online baskı: 21.09.2023

Balkan, T., Yılmaz, Ö (2023). Determination of pesticide residues in pomegranates grown in Antalya and health risk assessment. GIDA (2023) 48 (5) 993-1003 doi: 10.15237/ gida.GD23063

Balkan, T., Yılmaz, Ö (2023). Antalya'da yetiştirilen narlarda pestisit kalıntılarının belirlenmesi ve sağlık risk değerlendirmesi. GIDA (2023) 48 (5) 993-1003 doi: 10.15237/ gida.GD23063

ABSTRACT
Pesticide residues were screened in samples collected from pomegranate orchards in Antalya, Türkiye, and the health risks of such residues for consumers were assessed in this study. Analytical method verification was conducted to determine 260 pesticide residues by liquid chromatography-tandem mass spectrometry (LC–MS/MS). A total of 54 pomegranate samples were analyzed using this method. Ten of pomegranate samples contained pesticide residues above European Union Maximum Residue Limits (EU-MRLs). Both buprofezin and tebuconazole were detected in two of these samples, acetamiprid in three, tebuconazole in two, deltamethrin in two, and chlorpyrifos in one. In the risk assessment, deltamethrin has the potential for chronic toxicity for consumers, and chlorpyrifos shows both acute and chronic toxicity risks.
Keywords: Acute risk, Chronic risk, LC-MS/MS, Method verification, QuEChERS

ANTALYA'DA YETİŞTİRİLEN NARLARDA PESTİSİT KALINTILARININ BELİRLENMESİ VE SAĞLIK RİSK DEĞERLENDİRMESİ

ÖZ
Antalya ili nar bahçelerinden toplanan numunelerde pestisit kalıntıları taranmış ve bu kalıntıların tüketiciler için oluşturduğu sağlık riskleri değerlendirilmiştir. Sıvı kromatografi-tandem kütle spektrometresi (LC–MS/MS) ile 260 pestisit kalıntısının belirlenmesi için analitik metot doğrulaması yapılarak, Antalya ilinden toplanan 54 nar numunesi analiz edilmiştir. 10 nar numunesinde Avrupa Birliği Maksimum Kalıntı Limitlerinin (AB-MRL) aştığı belirlenmiştir. Bunlarda ikisinde buprofezin ve tebuconazole, üçünde acetamiprid, ikisinde tebuconazole, ikisinde deltamethrin ve birinde ise chlorpyrifos tespit edilmiştir. Tespit edilen pestisitlere yönelik risk değerlendirmesinde deltamethrin kalıntı değerlerinin tüketiciler için kronik toksisite potansiyeline sahip olduğu, chlorpyrifos kalıntı değerinin ise hem akut hem kronik toksisite riski gösterdiği belirlenmiştir.
Keywords: Akut risk, Kronik risk, LC-MS/MS, Metot doğrulama, QuEChERS

* *Corresponding author*/ Yazışmalardan sorumlu yazar
✉: tarik.balkan@gop.edu.tr ☏: (+90) 356 252 1616-1464 / (+90) 356 252 14 88

Tarık Balkan; ORCID no: 0000-0003-4756-4842
Özlem Yılmaz; ORCID no: 0000-0001-8564-120X

INTRODUCTION

Pomegranate (*Punica granatum* L.) has high nutritional content and medicinal properties. It provides numerous health benefits such as boosting immunity, regulating blood circulation, aiding digestion, and contributing to diabetes management (Naik et al. 2022).

In 2019, global pomegranate production was estimated to be around 4 million metric tons worth 8 million USD. The highest production occurs in Iran, India, China, Turkey, and the USA (Kahramanoğlu, 2019; Venkitasamy, 2019). Pomegranate is an important product for the Turkish economy and besides its contribution to human nutrition and various alternative uses (Özalp and Yılmaz, 2013). According to the 2021 report of the Turkish Statistical Institute, approximately one-fourth of pomegranate production in Turkey comes from Antalya (TSI, 2021).

Many pests and diseases have been reported in the pomegranate orchards of Antalya province. Among them, the most common pests are *Frankliniella occidentalis* Perg., *Nezara viridula* (L.), *Siphoninus phillyreae* (Haliday), *Planococcus citri* Risco., *Ectomyelois ceratoniae* (Zell.), *Ceratitis capitata* (Wiedemann), and the most common diseases are *Alternaria alternata* (Fr.) Keissler, *Botrytis cinerea* Pers., and *Penicillium* spp. (Şahin, 2013; Ilgın, 2016). These pests and diseases cause yield and quality losses before and after harvest. Therefore, growers frequently use pesticides to prevent yield loss. According to the 2020 TUIK data, Antalya is the city in Turkey where pesticides are used the most, with 4.349.658 kg-lt (Özercan and Taşçı, 2022). These pesticides can accumulate on/in fruits, posing food safety problems (Ryberg et al., 2018) and threatening environmental health and natural balance. Between January 2020 and October 2022, 32 notifications of pesticide residues in pomegranates exported from Turkey were reported by RASFF, 22 of which were classified as having a "serious" risk level. Kuchheuser and Birringer (2022) stated that the European Commission may need additional measures due to the increasing trend in notifications regarding pomegranates imported from Turkey. Furthermore, the limited number of residue studies on pomegranates in Turkey (Bakırcı et al., 2014; Dinçay et al., 2017; Gormez et al., 2021; Soydan et al., 2021) increases the necessity for residue monitoring studies.

Monitoring is an integral part of Good agricultural practices (GAPs). Therefore, testing samples for multiple pesticides at low levels, such as 0.01 mg/kg, is essential to ensure food safety. Electrospray ionization (ESI) liquid chromatography/tandem mass spectrometry (LC-MS/MS) has become the method of choice for pesticide residue analysis worldwide due to its high sensitivity, selectivity, wide scope, and ease of use (Ortelli et al, 2004; Banerjee et al., 2008).

In this study, the aims were (1) to report the verification results of multiple residue detection of 260 different pesticides in pomegranate using the QuEChERS method and LC/MS/MS (2) to investigate the pesticide residues of the samples collected from pomegranate orchards in Antalya (3) to evaluate the risk on human health.

MATERIALS AND METHOD

Sample collection and storage

The samples were collected randomly from the pomegranate orchards in Antalya, the region with the highest production in Turkey. Pomegranate samples were collected approximately 2 kg (at least five units) (EC, 2002). The samples were transported immediately to the laboratory and stored at -20 ºC.

Reagents and chemicals

Pesticide reference standards were supplied by Dr. Ehrenstorfer GmbH (Augsburg, Germany). Acetonitrile (ACN) was bought by Honeywell (North Carolina, USA). Methanol (MeOH), magnesium sulfate anhydrous ($MgSO_4$), ammonium formate (NH_4CO_2H), sodium acetate anhydrous (NaOAc), and acetic acid (AcOH) were procured from Merck (Darmstadt, Germany). Water was purified by MP Minipure Dest Up system (Ankara, Turkey). The QuEChERS products were purchased from Restek (Bellefonte, USA).

Chromatographic analysis
The analyses were conducted on Shimadzu UHPLC Nexera™ X2, and LCMS™-8050 triple quadrupole mass spectrometer with an electrospray ionization (ESI). The LC-MS/MS conditions were listed in Table 1.

Table 1. Operating conditions and gradient program

LC Conditions (Nexera X2)		MS Conditions (LCMS-8050)	
Column	Inertsil (ODS-4), C18 column (2.1 mm x 150 mm, 3 µm)	Ionization mode	ESI (Positive / Negative)
Oven temp.	40 °C	Desolvation line temp	250 ºC
Solvent A	5 mM/L ammonium formate/deionized water	Interface temp.	300 ºC
Solvent B	5 mM/L ammonium formate/methanol	Block heater temp.	400 ºC
Gradient	5%B. (0 min) - 60%B. (3 min) - 70%B. (4 min) - 80%B. (6 min) - 95%B. (7 - 8.50 min) - 5%B. (8.51-15 min)	Nebulizer gas flow	2.9 L/min.
Flow rate	0.4 mL/min	Drying gas flow	10.0 L/min.
Injection vol.	10 µL	Heating gas flow	15.0 L/min.
Rinse solution	R0: 50% methanol	Dwell time	1-33 msec

Sample extraction and clean-up
The official QuEChERS AOAC Method 2007.01 was used for the extraction and clean-up procedures (Lehotay, 2007). The following QuEChERS steps are illustrated in Figure 1. Each of the samples was analyzed in triplicates with LC-MS/MS.

For recovery studies, approximately 1 kg of pomegranate sample was homogenized with a blender and 15 g of the homogenized sample was weighed into a 50 ml Falcon tube. Then, 150 µl of pesticide mixture was added to 15 g of sample and vortexed for 60 seconds (Polat and Tiryaki, 2019; Dülger and Tiryaki, 2021). The mixture was left to stand for 15 minutes to allow the pesticides to interact with the matrix. Subsequently, the steps illustrated in Figure 1 were followed.

Calculation of risk assessment
In assessing the acute and chronic risk of pesticide residues, the estimated dietary exposure was compared to toxicological values known as acute reference dose (ARfD, mg kg^{-1} bw day^{-1}) and acceptable daily intake (ADI, mg kg^{-1} bw day^{-1}). The short-term consumer health risk (acute hazard index, aHI) was calculated by dividing the estimated short-term intake (ESTI, mg kg^{-1} day^{-1}) by the acute reference. On the other hand, the long-term consumer health risk (chronic hazard index, cHI) was calculated by dividing the estimated daily intake (EDI, mg kg^{-1} day^{-1}) by the acceptable daily intake. The relevant formulas used for these calculations were as follows (Liu et al., 2016);

$$\text{ESTI} = (\text{high residue level x food consumption})/\text{body weight} \quad (1)$$

$$\text{aHI} = \text{ESTI}/\text{ARfD x } 100 \quad (2)$$

$$\text{EDI} = (\text{mean residue level x food consumption})/\text{body weight} \quad (3)$$

$$\text{cHI} = \text{EDI}/\text{ADI x } 100 \quad (4)$$

The food is considered a risk to the consumers when the health risk index >1. The food is considered acceptable when the index is <1 (Darko and Akoto, 2008, Soydan et al., 2021; Balkan and Kara, 2022). The average body weight of an adult was considered 73.5 kg (TSI 2019), and daily consumption of pomegranate for the general population in Turkey were used as 0.01 $kg^{-1}day^{-1}$ (TSI, 2021).

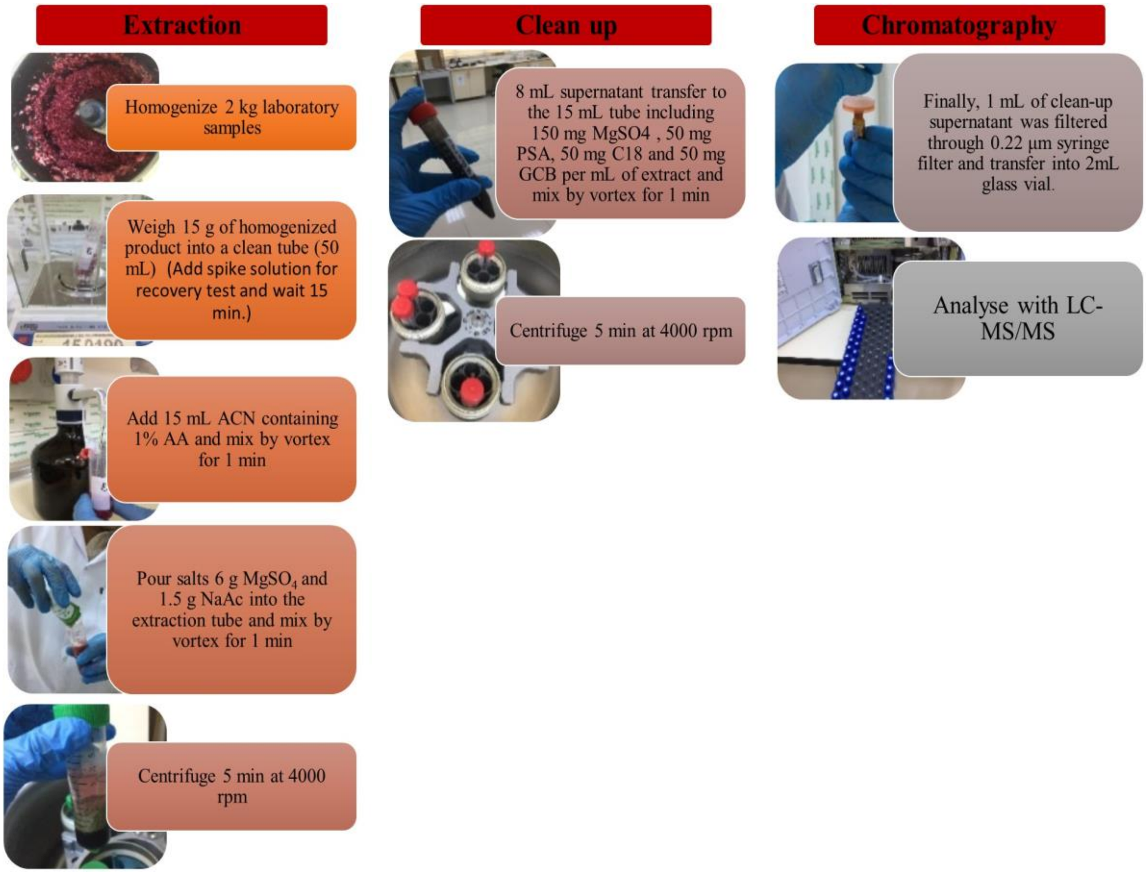

Figure 1. Analytical steps of the QuEChERS-AOAC Official Method 2007.01

RESULTS AND DISCUSSION

Method verification

In our previous study, we reported the results of the method validation analysis of 260 pesticides determined by LC-MS/MS in some leafy vegetables (Balkan and Yılmaz, 2022). This method has been verified to determine pesticide residues in pomegranates. The matrix calibration curves and calibration equations of 260 pesticides were linear ($R^2 \geq 0.990$) in the 5-200 μg mL^{-1} calibration range. The correlation coefficient (R^2) over 0.990 is an important criterion of linearity (Tiryaki et al., 2008). Calibration curves of boscalid and pyraclostrobin, which had concentrations lower than the MRL, and acetamiprid, buprofezin, chlorpyrifos, deltamethrin, and tebuconazole, which had residue concentrations higher than the MRL, are shown in Figure 2.

Detection and quantification limits

The LOD and LOQ values of pesticides detected were found to be lower than MRL values determined by the European Union for pomegranate (EC, 2022). The LODs ranged from 0.75 to 2.67 μg kg^{-1}, while LOQ values were between 2.50 and 8.90 μg kg^{-1}. The LOD and LOQ values of acetamiprid, boscalid, buprofezin, chlorpyrifos, deltamethrin, pyraclostrobin, and tebuconazole were 2.19-7.30, 0.84-2.80, 0.75-2.50, 0.87-2.90, 2.67-8.90, 1.68-5.60 and 2.49-8.30 μg kg^{-1}, respectively (Table 2).

Precision and accuracy

Precision and accuracy of the method is evaluated by repeatability (%RSD) and recovery (%Q) (SANTE, 2021). The recovery tests were carried out using five replicates at three fortification levels of 10, 50, and 100 μg kg^{-1}, respectively. The recovery rates of acetamiprid, boscalid, buprofezin, chlorpyrifos, deltamethrin, pyraclostrobin, and tebuconazole are given in Table 2. The results obtained for the other 253 pesticides were within the values stated by SANTE recovery limits (70% ≤ Q ≤ 120%) and repeatability (≤ 20%) (SANTE, 2021).

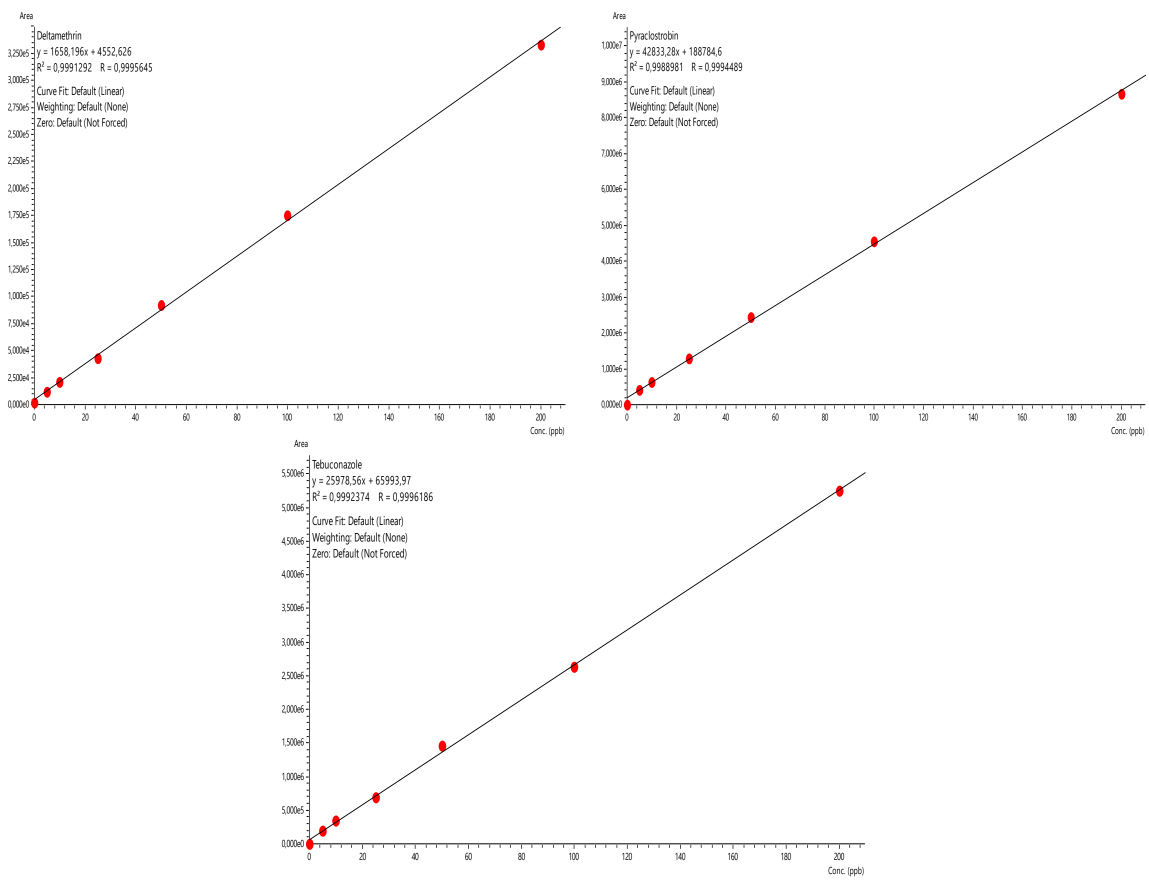

Figure 2. Calibration curves for seven pesticides in matrix-matched calibration

The findings regarding the recovery rates were in line with the method validation parameters for pesticide residue analysis (SANTE, 2021). The accuracy values, which express the closeness of the measured values to the actual values are given in Table 2. The findings indicate that QuEChERS provides effective recovery rates for 260 pesticides. As a result, the validated method can be considered a rapid and accurate approach for analyzing pomegranate residues (Tiryaki, 2016).

Pesticide residues

The study investigated 260 pesticides from various groups, including insecticides, acaricides, nematicides, fungicides, herbicides, plant growth regulators, and some metabolites. LC-MS/MS was used for the analysis of these pesticides in pomegranate samples. Out of the 16 samples analyzed, seven different pesticides were detected. On the other hand, in 38 of the samples, no pesticide active ingredient was found. The residue levels of the detected pesticides ranged between 11.06 and 144.44 μg kg^{-1}. The results and frequency of the pesticides were presented in Table 3.

Table 2. QuEChERS-AOAC method verification data

Pesticide	Linear regression equation	R^2	LOD (μg kg⁻¹)	LOQ (μg kg⁻¹)	Concentration (μg kg⁻¹) Spiked	Concentration (μg kg⁻¹) Measured	Recovery %	RSD %
Acetamiprid	Y=133556.6x + 690071.0	0.998	2.19	7.30	10	10.12	101.12	8.91
					50	53.89	107.78	10.21
					100	111.88	111.88	9.13
Boscalid	Y=14332.22x + 109937.2	0.996	0.84	2.80	10	11.05	110.50	7.57
					50	56.85	113.70	11.53
					100	111.74	111.74	12.07
Buprofezin	Y=61556.09x + 27121.12	0.999	0.75	2.50	10	9.11	91.10	8.68
					50	57.22	114.44	13.35
					100	107.50	107.50	8.37
Chlorpyrifos	Y=14913.12x + 52824.51	0.999	0.87	2.90	10	9.55	95.50	11.31
					50	57.97	115.94	12.39
					100	115.49	115.49	7.17
Deltamethrin	Y=1658.196x + 4552.626	0.999	2.67	8.90	10	11.68	116.80	12.93
					50	53.70	107.40	10.15
					100	100.74	100.74	10.52
Pyraclostrobin	Y=42833.28x + 188784.6	0.998	1.68	5.60	10	11.47	114.70	11.93
					50	55.36	110.72	13.34
					100	115.22	115.22	6.32
Tebuconazole	Y=25978x+ 65993.97	0.999	2.49	8.30	10	11.39	113.90	11.42
					50	55.31	110.62	11.28
					100	108.03	108.03	6.86

Table 3. Pesticide residue levels and frequencies

Food commodity	Number of sample >LOQ and percentage, (%)	Number of sample >MRL and percentage, (%)	Pesticide	Frequency of detection	Pesticide residue [a] (mg kg⁻¹)	Number of sample >MRL	MRL[b] (mg kg⁻¹)
Pomegranate	16 (29.6)	10 (18.5)	Acetamiprid	3	0.013- 0.014- 0.018	3	0.01
			Boscalid	7	0.018-0.088	-	2
			Buprofezin	2	0.016- 0.024	2	0.01
			Chlorpyrifos	1	0.102	1	0.01
			Deltamethrin	2	0.028- 0.144	2	0.01
			Pyraclostrobin	1	0.014	-	0.02
			Tebuconazole	5	0.011- 0.075	4	0.02

[a] Mean of three analytical portions, [b] EU pesticide database (European Commission, 2022)

The results were evaluated according to EU-MRL. Residue values from 10 out of 16 pomegranate samples (18.5%) exceeded the MRL. Concentrations of acetamiprid, buprofezin, chlorpyrifos, deltamethrin, and tebuconazole were higher than the MRL. Additionally, six samples contained two different pesticides.

In Turkey, acetamiprid is licensed for use against *Siphoninus phillyreae, Planococcus citri, Aphis punicae*. The acetamiprid residue level was found to be higher than the MRL values in 3 samples. Buprofezin is licensed in Turkey for control against *Alternaria alternata*. The residue of buprofezin exceeded the MRL in 2 samples. The use of chlorpyrifos was terminated in May 2020 and was banned in Turkey. According to the previous license information, it has been used against many pests, especially sucking insects in orchards. Interestingly, the chlorpyrifos residue concentration exceeded the EU-MRL by 102 times in one sample.

Deltamethrin is licensed in Turkey for control against *Ceratitis capitata*. The deltamethrin residue level in 2 samples were higher than the MRL. Tebuconazole is licensed in Turkey for against control *Phytophthora* spp. The tebuconazole residue level in 4 samples were higher than the MRL. Boscalid and pyraclostrobin is licensed in Turkey for control against *Alternaria alternata*. These licensed active ingredients are sold as mixed preparations containing 25.2% boscalid and 12.8% pyraclostrobin. The boscalid residue in 7 samples and pyraclostrobin residue in 1 sample were lower than the MRL.

Dinçay et al. (2017) found malathion in 17.1% and spinosad in 9.1% of 187 pomegranate samples collected in 2013, while malathion was found in 14.3% and spinosad in 15.4% of 91 pomegranate samples collected in 2014, Turkey. They stated that the active ingredients in these samples did not exceed the MRL. Savant et al. (2010) detected chlorpyrifos residues lower than the MRL in a pomegranate sample from India. The residues of imidacloprid were found below the specified MRL in two fresh fruit samples collected from retail markets in India (Utture et al., 2012). In a study conducted in India, acephate thiamethoxam, imidacloprid, carbendazim, tebuconazole, difenoconazole, profenofos, quinalphos, novaluron, thiophanate methyl, and acetamiprid were found lower than MRL in whole pomegranate samples from the markets (Naik et al. 2022). Soydan et al. (2021) detected pesticide residues higher than MRL in 7 of 157 pomegranate samples. In this study, pesticide residues were higher than MRL in 10 out of 54 pomegranate samples.

Uçan et al. (2009), Ersoy et al. (2011), Bakırcı et al. (2014), and Ersoy (2019) conducted separate investigations on pesticide residues in pomegranate samples. In their respective studies, they found no detectable levels pesticides in the samples. In this study, no detectable pesticide residue was found in 38 pomegranate samples, while quantifiable pesticide residues were found in 16 samples.

Health risk assessment

The risk analysis was conducted for seven detected pesticides, and the results were presented in Table 4.

Table 4. The results of long-term and short-term risk assessments

Food commodity	Pesticide	ADI* (mg kg^{-1} bw day^{-1})	ARfD* (mg kg^{-1} bw day^{-1})	Long-term risk		Short-term risk	
				EDI (mg kg^{-1}day^{-1})	cHI	ESTI (mg kg^{-1}day^{-1})	aHI
Pomegranate	Acetamiprid	0.025	0.025	1.71E-05	0.07	2.08E-05	0.08
	Boscalid	0.04	/	4.85E-05	0.12	9.76E-05	/
	Buprofezin	0.01	0.05	2.30E-05	0.23	2.75E-05	0.05
	Chlorpyrifos	0.001	0.005	1.13E-04	11.32	1.13E-04	2.26
	Deltamethrin	0.01	0.025	9.50E-05	0.95	1.59E-04	0.63
	Pyraclostrobin	0.03	0.03	1.56E-05	0.05	1.56E-05	0.05
	Tebuconazole	0.03	0.03	5.14E-05	0.17	8.28E-05	0.28

*ARfD and ADI were adopted from IUPAC pesticides properties database (IUPAC, 2022).
The symbol of "/" represented that there was no authorized value for ARfD. and the corresponding risk index could not be computed.

The short-term risk assessment revealed that chlorpyrifos level poses a high risk with a value of 2.2631, while all other aHI values were less than 1, indicating a negligible acute risk. Similarly, the long-term risk assessment revealed that chlorpyrifos level possesses high risk with a value of 11.3155, while all other cHI values were less than 1, indicating a negligible acute risk. However, the cHI value of deltamethrin (0.9501) is quite close to the limit value. These findings suggest that the acute and chronic risks associated with pesticide exposure through the consumption of pomegranate should be taken into consideration.

In the literature review, a study conducted in Turkey examined the health risks arising from pesticide residues in pomegranate. The hazard quotient values reported in the study conducted by Soydan et al. (2021) indicated that pomegranate consumption does not pose a public health problem. Similarly, other studies conducted by Saleh et al. (2020), Matadha et al. (2021), and Tripathy et al. (2022) also reported that there is no significant potential human risk to consumers from pesticide residues in pomegranate.

CONCLUSIONS

This study aimed to investigate pesticide residues in pomegranates produced in Antalya province and assess the related health risks. Among the 54 samples analyzed, seven different pesticides were detected in 16 samples. The residue levels of 5 pesticides exceeded the MRLs. Our study results indicate that some negative situations posing potential risks to human health may be encountered. To prevent such scenarios, raising awareness among producers and plant protection product sellers about the impacts of pesticides on the environment and human health is crucial. In addition, it would be appropriate to encourage producers to implement good agricultural practices (GAPs) and integrated pesticide management (IPM).

In conclusion, conducting more comprehensive research on pesticides and performing routine residue analyses are essential to ensure food safety and protect public health. In addition, increasing the number of pesticides investigated in this type of analysis is important to obtain more comprehensive results.

CONFLICT OF INTEREST

Authors declare no conflict of interest.

AUTHORS' CONTRIBUTIONS

TB made significant contributions to the design and execution of the study. ÖY contributed to conduction of research. TB and ÖY performed the experiments. TB and ÖY calculated, analysed and interpreted data. TB attented in designing and writing the manuscript. TB gave final confirmation for the submission of revised version. All authors read, approved final manuscript.

REFERENCES

Bakırcı, G.T., Acay, D.B.Y., Bakırcı, F., Ötleş, S. (2014). Pesticide residues in fruits and vegetables from the Aegean region, Turkey. *Food Chemistry*, 160; 379-392. Https://doi.org/10.1016/ j.foodchem.2014.02.051.

Banerjee, K., Oulkar, D.P., Patil, S.B., Patil, S.H., Dasgupta, S., Savant, R., Adsule, P.G. (2008). Single-laboratory validation and uncertainty analysis of 82 pesticides determined in pomegranate, apple, and orange by ethyl acetate extraction and liquid chromatography/tandem mass spectrometry. *Journal of AOAC International*, 91(6); 1435–1445.

Balkan, T., Kara, K. (2022). Determination of pesticide residues and risk assessment in some vegetables grown in Tokat province. *Plant Protection Bulletin*, 62(2); 26-35, Https://doi.org/10.16955/bitkorb.1053952.

Balkan, T., Yılmaz, Ö. (2022). Method validation, residue and risk assessment of 260 pesticides in some leafy vegetables using liquid chromatography coupled to tandem mass spectrometry. *Food Chemistry*, 384; 132516, Https://doi.org/10.1016/j.foodchem.2022.132516.

Darko, G., Akoto, O. (2008). Dietary intake of organophosphorus pesticide residues through vegetables from Kumasi, Ghana. *Food and*

Chemical Toxicology, 46(12); 3703-3706, Https://doi.org/10.1016/j.fct.2008.09.049.

Dinçay, O., Civelek, H. S., Görmez, E. (2017). Residue analysis of insecticides used for control of Mediterranean Fruit Fly [Ceratitis capitata (Wiedemann) (Diptera: Tephritidae)] in Izmir's satsuma (mandarin) and Antalya's pomegranates. *Ege Üniversitesi Ziraat Fakültesi Dergisi*, 54(2); 231-238 (in Turkish with English abstract), Https://doi.org/10.20289/zfdergi.387346.

Dülger, H., Tiryaki, O. (2021). Investigation of pesticide residues in peach and nectarine sampled from Çanakkale, Turkey, and consumer dietary risk assessment. *Environmental monitoring and assessment*, 193; 561, Https://doi.org/10.1007/s10661-021-09349-8.

Ersoy N., Tatlı Ö., Özcan S., Evcil E., Coşkun L.Ş., Erdoğan E., 2011. Pesticide residues in some tropical and subtropical fruit species. *Selçuk Tarım ve Gıda Bilimleri Dergisi*, 25(2); 81-88, (in Turkish with English abstract).

Ersoy, N. (2019). Good Agricultural Practices (GAPs) in 'Hicaznar' Pomegranate (Punica granatum L.) Cultivar. *Mehmet Akif Ersoy Üniversitesi Fen Bilimleri Enstitüsü Dergisi*, 10(2); 198-209. Https://doi.org/10.29048/makufebed.637515.

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, 2, 30–43.

EC, 2022. European Commission, European Union (EU) pesticides database. Pesticide Residues MRLs. Directorate General for Health & Consumers. https://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/start/screen/mrls Accessed September 10, 2022.

Ilgın, T. (2016). Antalya ilinde nar üretim alanlarında ve depolanan meyvelerde görülen fungal hastalık etmenlerinin belirlenmesi. Süleyman Demirel Üniversitesi Fen Bilimleri Enstitüsü Bitki Koruma Anabilim Dalı Yüksek Lisans Tezi, Isparta, Türkiye, 68s.

Gormez, E., Golge, O., Kabak, B. (2021). Quantification of fosetyl-aluminium/phosphonic acid and other highly polar residues in pomegranates using Quick Polar Pesticides method involving liquid chromatography-tandem mass spectrometry measurement. *Journal of chromatography. A*, 1642; 462038, https://doi.org/10.1016/j.chroma.2021.462038.

IUPAC 2022. The PPDB - Pesticide Properties Database International Union of Pure and Applied Chemistry. http://sitem.herts.ac.uk/aeru/iupac/index.htm (Accessed date: October 7, 2022).

Kahramanoglu, İ., (2019). Trends in pomegranate sector: production, postharvest handling and marketing. *International Journal of Agriculture Forestry and Life Sciences*, 3(2); 239-246.

Kuchheuser, P., Birringer, M. (2022). Evaluation of specific import provisions for food products from third countries based on an analysis of RASFF notifications on pesticide residues. *Food Control*, 133;108581, Https://doi.org/10.016/j.foodcont.2021.108581.

Lehotay S.J., 2007. Determination of pesticide residues in foods by acetonitrile extraction and partitioning with magnesium sulfate: Collaborative Study. *Journal of AOAC International*, 90(2); 485–520.

Liu, Y., Shen, D., Li, S., Ni, Z., Ding, M., Ye, C., Tang, F. (2016). Residue levels and risk assessment of pesticides in nuts of China. *Chemosphere*, 144; 645-651, Https://doi.org/10.1016/j.chemosphere.2015.09.008.

Matadha, N.Y., Mohapatra, S., Siddamallaiah, L. (2021). Distribution of fluopyram and tebuconazole in pomegranate tissues and their risk assessment. *Food chemistry*, 358; 129909, Https://doi.org/10.1016/j.foodchem.2021.129909.

Naik, R.H., Pallavi, M.S., Nandini, Shwetha, A., Bheemanna, M., Nidoni, R.U. (2022). Simultaneous determination of pesticide residues in pomegranate whole fruit and arils using LC-

MS/MS. *Food Chemistry*, 387; 13286, Https://doi.org/10.1016/j.foodchem.2022.132865.

Ortelli, D., Edder, P., Corvi C. (2004). Multiresidue analysis of 74 pesticides in fruits and vegetables by liquid chromatography–electrospray–tandem mass spectrometry. Analytica Chimica Acta, 520; 33–45. Https://doi.org/10.1016/j.aca.2004.03.037.

Özalp, A., Yılmaz, İ. (2013). Input usage, profitability and productivity analysis of pomegranate production in Antalya province. *Akdeniz University Journal of the Faculty of Agriculture*, 26(1); 9-26, (in Turkish with English abstract).

Özercan, B. Taşcı, R. (2022). Investigation of Pesticide Use in Türkiye in Terms of Provinces, Regions and Pesticide Groups. *Ziraat Mühendisliği*, (375); 75-88, (in Turkish with English abstract), Https://doi.org/10.33724/zm.1120599.

Polat, B., Tiryaki, O. (2019). Determination of some pesticide residues in conventional-grown and IPM-grown tomato by using QuEChERS method. *Journal of Environmental Science and Health, Part B*, 54(2); 112-117, Https://doi.org/10.1080/03601234.2018.1531663.

Ryberg, M.W., Rosenbaum R.K., Mosqueron, L., Fantke, P. (2018). Addressing bystander exposure to agricultural pesticides in life cycle impact assessment. *Chemosphere*, 197; 541–549, Https://doi.org/10.1016/j.chemosphere.2018.01.088.

Saleh, I.A., Khorshed, M.A., Eshmawy, M.R., Kandil, M.A. (2020). Pesticide residues in horticultural crops from domestic markets of giza, egypt: occurrence and risk assessment. *Plant Archives*, 20(1); 3862-3869.

SANTE, 2021. SANTE/11312/2021 Analytical quality control and method validation procedures for pesticide residues analysis in food and feed. 1–55. https://www.accredia.it/app/uploads/2021/02/SANTE_11312_2021.pdf (Accessed date: October 7, 2022).

Savant, R.H., Banerjee, K., Utture, S.C., Patil, S.H., Dasgupta, S., Ghaste, M.S., Adsule, P.G. (2010). Multiresidue analysis of 50 pesticides in grape, pomegranate, and mango by gas chromatography-ion trap mass spectrometry. *Journal Agricultural Food Chemistry*, 58; 1447–1454, Https://doi.org/10.1021/jf903398f.

Soydan, K. D., Turgut, N., Yalçın, M., Turgut, C., Karakuş, P. B. K. (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; 27511–27519. Https://doi.org/10.1007/s11356-021-12580-y.

Şahin, Ö. (2013). Antalya ili nar alanlarında görülen zararlılar ve doğal düşmanları. Süleyman Demirel Üniversitesi Fen Bilimleri Enstitüsü Bitki Koruma Anabilim Dalı Yüksek Lisans Tezi, Isparta, Türkiye, 41s.

Tiryaki, O. (2016). Validation of QuEChERS method for the determination of some pesticide residuesin two apple varieties. *Journal of Environmental Science and Health, Part B*, 51; 722-729. Https://doi.org/10.1080/03601234.2016.1191922.

Tiryaki, O., Baysoyu, D., Seçer, E., Aydın G. (2008). Testing the stability of pesticides during sample processing for the chlorpyrifos and malathion residue analysis in cucumber including matrix effects. *Bulletin of Environmental Contamination and Toxicology*, 80(1); 38-43.

Tripathy, V., Sharma, K.K., Mohapatra, S. et al. 2022. Persistence evaluation of fluopyram + tebuconazole residues on mango and pomegranate and their risk assessment. *Environmental Science and Pollution Research* 29; 33180–33190, Https://doi.org/10.1007/s11356-021-17993-3.

TSI, 2019. Turkey Health Interview Survey. https://data.tuik.gov.tr/Bulten/DownloadIstatistikselTablo?p=WEBW229PP/91tMV2m71fU6pRWq2F1ZD/lzOFFk0bNDi2rjAC8QDCRN62nr2M3n1K (Accessed date: September 10, 2022).

TSI, 2021. Turkish Statical Institute. https://biruni.tuik.gov.tr/medas/?kn=92&locale=tr (Accessed date: September 10, 2022)

Uçan, H. N., Dursun S., Gür K., Aktümsek A. (2009). Organochlorine Pesticide Residue

Analyses in Some Fruit Samples Collected from Konya City Supermarkets. *Asian Journal of Chemistry,* 21(6); 4843-4855.

Utture S.C., Banerjee, K., Kolekar, S.S., Dasgupta, S., Oulkar, D., Patil, S. H., Wagh, S.S., Adsule, P.G., Anuse, M.A. (2012). Food safety evaluation of buprofezin, dimethoate and imidacloprid residues in pomegranate. *Food Chemistry*, 131; 787-795, Https://doi.org/10.1016/j.foodchem.2011.09.044.

Venkitasamy, C., Zhao, L., Zhang, R., Pan Z. (2019). Chapter 8 – Pomegranate. *Integrated Processing Technologies for Food and Agricultural By-Products*, Academic Press, Pages 181-216. https://doi.org/10.1016/B978-0-12-814138-0.00008-3.