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Determination of pesticide residues and risk assessment in some vegetables grown in Tokat province

Tokat ilinde üretilen bazı sebzelerde pestisit kalıntılarının tespiti ve risk değerlendirmesi

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A R T I C L E I N F O

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A B S T R A C T

This study was carried out to determine the pesticide residue levels and health risk assessments in tomato, pepper, and cucumber grown in Tokat province of Turkey. A residue analytical method was verified to determine 260 pesticides by using a liquid chromatography-tandem mass spectrometry (LC-MS/MS). Pesticide solutions at 10 and 50 μg kg^{-1} doses were fortified with the pesticide-free cucumber matrix for verification of the method. The linearities (R^2), the limit of detection (LODs), the limit of quantification (LOQs), and mean recovery values of the pesticides ranged between 0.990-0.999, 0.71-2.96 μg kg^{-1}, 2.36-9.86 μg kg^{-1} and 77.25-117.61%, respectively. Pesticide residue levels of 28 vegetable samples collected from Tokat province were determined using the verified method. Seventeen different pesticides were detected in 16 samples, and pesticide concentrations in 3 samples were higher than the allowable limits of the European Union Maximum Residue Limits (EU-MRL). The pyridaben level in 1 pepper sample, metrafenone in 2 cucumber samples were higher than the threshold values stated by the EU-MRL. The results of health risk assessments indicated that famoxadone and pyridaben have chronic toxicity potential for consumers.

INTRODUCTION

Vegetables are an important part of the human diet as they provide essential specific nutrients for the human body (Keikotlhaile and Spanoghe 2011). Their consumption is recommended not only to prevent avitaminosis, but also to reduce the incidence of important diseases such as cancer, cardiovascular diseases, and obesity. Rapid urbanization and increase in human population caused changes in lifestyle and eating habits, and the food demands of the consumers have diversified. These approaches have strongly impressed food production and consumption; therefore, the producers have more carefully adopted plant protection measures to control pests. Pesticides are commonly used in pest management due to their rapid action, easy use, and affordability. However, misuse or unconscious use of pesticides may damage plants, the environment, wildlife, and public health. Because pesticides used in agricultural production often lead to residues. Contamination of crops, especially fruits and vegetables, is one of the current important issues. The majority of consumers are not familiar with pesticide residue in the food; therefore, monitoring the residue levels of pesticides in food is vital for human health.

Detailed studies on pesticide residue levels in vegetables have been reported in Turkey. Bakırcı et al. (2014), Balkan and Kara (2019), Çatak and Tiryaki (2020), Çiftçi (2019), Durmuşoğlu (2002), Ersoy et al. (2011), Golge and Kabak (2018) reported residue concentrations over MRL threshold in cucumber, tomato, and pepper samples. In contrast, the residue concentration in vegetables reported by Hepsağ (2019), Kaya and Tuna (2019), Polat and Tiryaki (2018), Zengin and Karaca (2017) were lower than the MRL threshold values.

Exposure to pesticides can cause health problems, such as nausea and headaches in a shorth term. In addition, neurotoxicity, cytogenetic damage, infertility, and endocrine system problems may occur chronically (Baldi et al. 2001), and leukemia, non-Hodgkin lymphoma, brain, bone, breast, ovarian, prostate, testicular, and liver cancers in the long term (Cantor et al. 1992). Therefore, health risk assessment is very important for pesticide residues. Although studies on detection of pesticide residues for important vegetables in Turkey have increased, the studies on health risk assessment are not at the desired level. Gölge and Kabak (2018) determined no health risk in tomato, Soydan et al. (2021) stated no health risk in tomato and pepper. Çatak and Tiryaki (2020) noted no risk of chronic exposure in cucumber.

The increase in living standards raised the awareness on pesticide residues in agricultural products. In addition, ensuring safe pesticide residue level is important in promoting agricultural product export of Turkey. Increasing residue studies is essential for both local agricultural development and raising awareness for consumers. This study aimed to determine the pesticide residue levels and the health risk assessments in tomato, pepper, and cucumber grown in Tokat province, which has a high agricultural potential.

MATERIALS AND METHODS

Sample collection and storage

Samples were collected in accordance with the Commission Directive 2002/63/EC on sampling for the official control of pesticide residues in and on products of plant and animal origin (EC 2002). Ten cucumbers, 8 tomatoes, and 10 peppers were collected randomly in Tokat province, Turkey. Collected samples were transported immediately to the laboratory and stored at -18 °C.

Reagents and chemicals

Pesticide reference standards were purchased from Dr. Ehrenstorfer (Augsburg, Germany). Acetonitrile (MeCN > 99% purity), methanol (MeOH > 99% purity), anhydrous magnesium sulfate ($MgSO_4$ ≥ 99% purity), ammonium formate (CH_5NO_2 ≥ 99% purity), sodium acetate (NaOAc ≥ 99% purity), and acetic acid (AcOH > 99% purity) were supplied by Merck. Primary-secondary amine (PSA) was obtained from Supelco analytical.

Chromatographic analysis

This study was carried out using an LC-MS 8050 model (Shimadzu®) equipped with a UPLC: LC-30AD pump x 2, SIL-20A autosampler, a DGU-20A3R degasser, a CTO-20ACV column oven, and a triple quadrupole MS/MS detector. The LC column was inertsil (ODS IV) C18 column (2.1 mm x 150 mm, 3 µm particle size) of GL Sciences Inc (Tokyo, JAPAN). Chromatographic separation was performed using a gradient elution program with eluent A consisting of distilled H_2O + 5 mM ammonium formate and eluent B consisting of methanol + 5 mM ammonium formate. The analysis started with 5% eluent B, which was increased linearly to 60% in 3 min, 70% in 4 min, 80% in 6 min, and 95% in 7 min. The gradient elution was started with 5% of B (held 1 min), then increased linearly to reach 95% of B in 4 min (held 2 min), and decreased to the initial stage (5% of B) at 6 min, and kept until 9 min. The flow rate was 0.40 mL min^{-1}, and the injection volume was set to 10 µL. The column and autosampler temperatures were set to 35 °C and 4 °C, respectively. For MS/MS detection, the electrospray ionization (ESI) interface was used for positive polarity with the following: 3 kV of capillary voltage, 3V of extractor voltage, 350 °C of heat block temperature, 250 °C of desolvation line (DL) temperature, nitrogen (N_2) as nebulizer gas of 2.9 L min^{-1} and drying gas of 10 L min^{-1}. Nitrogen gas of 99% purity produced by a Peak Scientific nitrogen generator (Billerica, MA, USA) was used in the ESI source and the collision cell. Collision-induced dissociation (CID) gas was argon (Ar, 99.999%) of 230 kpa with a flow rate of 0.15 mL min^{-1}. All parameters of the instrument were controlled using LabSolution® software (version 4.91) (Balkan and Yılmaz 2022).

Sample extraction and clean up

The official QuEChERS AOAC Method 2007.01 was used for the extraction and clean-up procedures (Lehotay 2007). The steps in Figure 1 were followed for the QuEChERS process. Each sample was analyzed in triplicate with LC-MS/MS.

Calculation of risk assessment

In assessing the acute and chronic risk of pesticide residues; 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 acute/short-term consumer health risk (aHI) was calculated based on the estimated short-term intake (ESTI, mg kg^{-1} day^{-1})

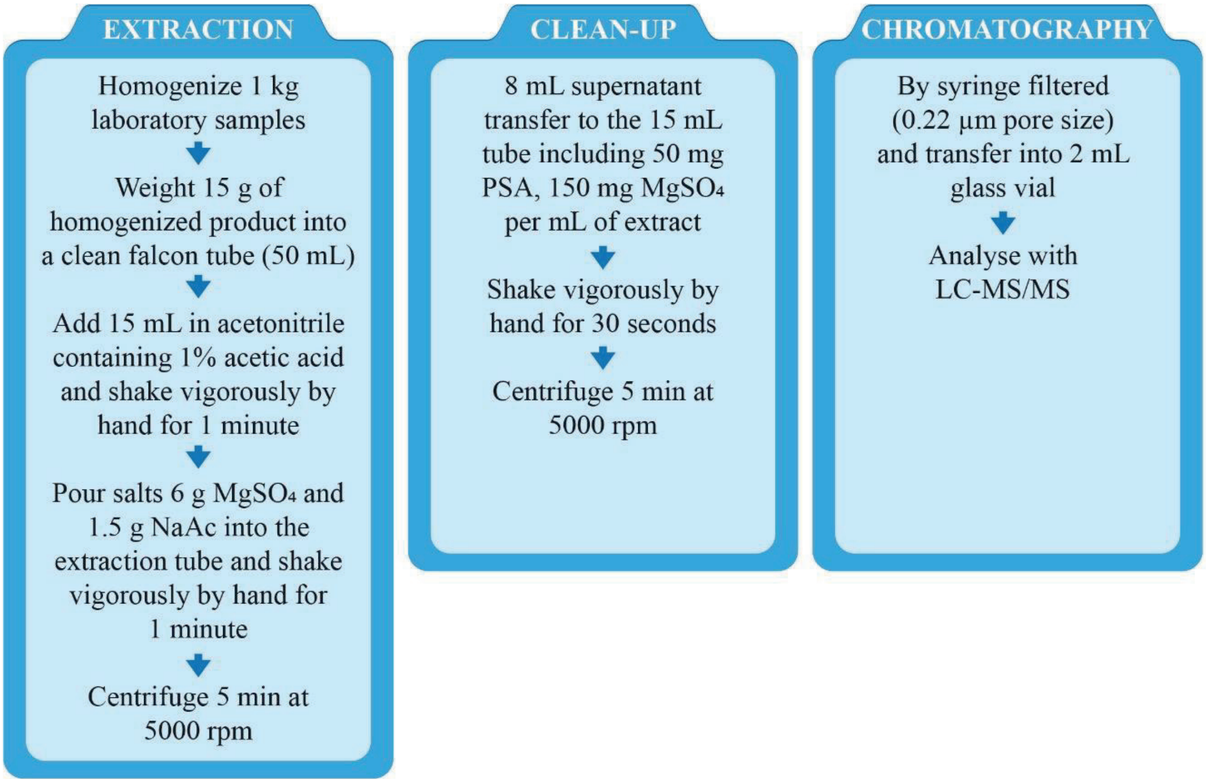

Figure 1. Analytical steps of the QuEChERS-AOAC Official Method 2007.01

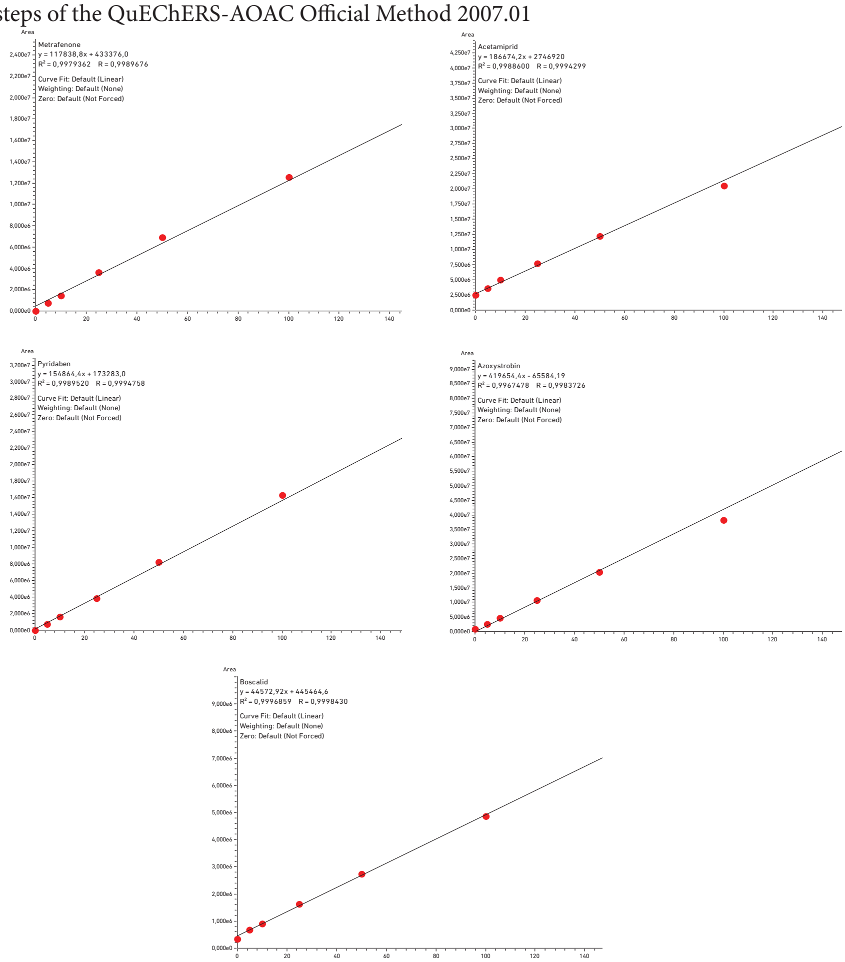

Figure 2. Calibration curves for five compounds in matrix-matched calibration

and the acute reference dose (ARfD). The chronic/long-term consumer health risk (chronic hazard index, cHI) was calculated based on the estimated daily intake (EDI, mg kg^{-1} day^{-1}) and the acceptable daily intake (ADI). The relevant formulas were as follows (Liu et al. 2016);

ESTI=(high residue level x food consumption)/body weight (1)

aHI=ESTI/ARfD x 100 (2)

EDI=(mean residue level x food consumption)/body weight (3)

cHI=EDI/ADI x 100 (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). The average body weight of an adult was considered 73.5 kg. (TÜİK, 2019). Daily consumption of cucumber, tomato, and pepper for the general population in Turkey were used as 0.05, 0.31, and 0.07 kg^{-1}day^{-1}, respectively (TÜİK 2021).

RESULTS AND DISCUSSION

Method verification

The matrix calibration curves and calibration equations of 260 pesticides in the LC-MS/MS system were linear ($R^2 \geq 0.990$) in the calibration range of 5–200 µg mL^{-1}. Correlation coefficient (R^2) over 0.990 is an important criteria of linearity (Tiryaki et al. 2008). Calibration curves of metrafenone, pyridaben of which the concentration was over MRL, and acetamiprid, azoxystrobin, boscalid of which the residue concentration was lower than the MRL, were shown Figure 2.

Detection and quantification limit

The LOD and LOQ values in all target analytes of the representative cucumber matrix were extremely low (EC 2019). The LOD values ranged from 0.71 to 2.96 µg kg^{-1}, while LOQs were between 2.36 and 9.86 µg kg^{-1}. The lowest LOD and LOQ values were recorded in the methomyl, while the highest value was determined in the fenthion active substance. The LOD and LOQ values of acetamiprid, azoxystrobin, boscalid, metrafenone, and pyridaben were 0.99-3.32, 2.35-7.84, 1.98-6.61, 1.57-5.22, and 1.74-5.79 µg kg^{-1}, respectively (Figure 2). The values for all pesticides were lower than the MRL values determined by the European Union for pepper, tomato, and cucumber.

Precision and accuracy

Method of precision and accuracy is evaluated by repeatability (%RSD) and recovery (%Q) (EC 2019, Magnusson and Örnemark 2014, TURKAK 2019). The recovery tests were carried out using five replicates at two fortification levels of 10 and 50 µg kg^{-1}, respectively. The recovery rates of acetamiprid, azoxystrobin, boscalid, metrafenone, and pyridaben are given in Table 1. Figures obtained for the other 255 pesticides were within the values stated by SANTE recovery limits (70% ≤ Q ≤ 120%) and repeatability (≤ 20%) (EC 2019).

Current findings on recovery rates are consistent with method validation parameters for pesticide residue analysis (EC 2019, Magnusson and Örnemark 2014). The accuracy values, which are expressed as the closeness of the measured values to the actual values (Tiryaki 2016), are given in Table 1. The results showed that QuEChERS provides efficient recovery rates for 260 pesticides. Therefore, the current analytical method could offer a fast and accurate method for residue analysis in the studied matrices.

Residues of samples

Two hundred sixty pesticides belonging to different groups used in Turkey were discussed in the study. Analysis of 260 pesticides was carried out using an LC-MS/MS device. The LOD, LOQ, and EU-MRL values of 17 pesticides detected are given in Table 2.

Table 1. QuEChERS method verification data

Analyte	Concentration (µg kg^{-1})		Recovery % (As a tool for trueness)	RSD % (As a tool for precision)
	Spiked	Measured[a]		
Acetamiprid	10	11.18	111.83	7.41
	50	56.39	112.78	4.05
Azoxystrobin	10	10.48	104.78	10.48
	50	51.92	103.83	7.20
Boscalid	10	10.57	105.67	6.50
	50	54.08	108.15	2.69
Metrafenone	10	7.94	79.40	5.15
	50	53.25	106.49	3.45
Pyridaben	10	10.56	105.60	6.90
	50	49.05	98.09	5.03

[a] Mean of three analytical portions

A total of 28 samples were analyzed. The residue levels were between 7.93 and 1501.30 µg kg^{-1}. The results and frequency of pesticides are presented in Table 3.

Acetamiprid, azoxystrobin, and boscalid were the active substances detected in all three vegetables (Table 3). The concentration of acetamiprid, azoxystrobin, and boscalid were between 9.90- 51.53, 11.46- 56.69, and 16.39-664.88 µg kg^{1}, respectively.

Bifenazate and kresoxim-methyl were detected in pepper and cucumber, pyridaben in pepper and tomato, penconazole in tomato and cucumber, pyrimethanil in pepper, famoxadone in tomato and clofentezine, fluopyram, hexythiazox, metrafenone, pirimicarb+pirimicarb-desmethy, tebuconazole and thiacloprid in cucumber. The

Table 2. LOQ and EU-MRL values

Analyte	LOD µg kg^{-1}	LOQ µg kg^{-1}	EU-MRL µg kg^{-1}			ADI*(mg kg^{-1} bw day^{-1})	ARfD* (mg kg^{-1} bw day^{-1})
			pepper	tomato	cucumber		
Acetamiprid	0.99	3.32	0.3	0.5	0.3	0.025	0.025
Azoxystrobin	2.35	7.84	3	3	1	0.2	/
Boscalid	1.98	6.61	3	3	4	0.04	/
Bifenazate	1.61	5.36	3		0.5	0.01	/
Clofentezine	2.24	7.45			0.2	0.02	/
Famoxadone	2.02	6.73		2		0.012	0.2
Fluopyram	1.92	6.39			0.5	0.012	0.5
Hexythiazox	2.30	7.66			0.5	0.03	/
Kresoxim-methyl	2.26	7.52	0.8		0.5	0.4	/
Metrafenone	1.57	5.22			0.5	0.25	/
Penconazole	2.31	7.72		0.1	0.06	0.03	0.5
Pirimicarb+ Pirimicarb-desmethy	1.41 1.08	4.70 3.59			1	0.035	0.1
Pyridaben	1.74	5.79	0.3	0.15		0.01	0.05
Pyrimethanil	2.26	7.52	2			0.17	/
Tebuconazole	2.21	7.38			0.6	0.03	0.03
Tetraconazole	1.84	6.13			0.2	0.004	0.05
Thiacloprid	1.60	5.32			0.5	0.01	0.03

*ARfD and ADI were adopted from IUPAC pesticides properties database (IUPAC 2022)
The symbol of "/" represented that there was no authorized value for ARfD

results were evaluated according to the threshold values stated by the EU-MRL. The residual values from 1 (10%) of pepper and 2 (20%) of cucumber samples were over MRL. Metrafenone and pyridaben concentrations were higher than the MRL values.

Metrafenone is used against powdery mildew, and pyridaben is used against plant-feeding mites and whitefly. Eleven samples contained more than one pesticide, and 8 of them contained more than three pesticide active ingredients. These samples (10.7%) had pesticide residues above EU-MRL values. The residue level in tomato samples was not higher than the EU-MRL values. Similarly, Szpyrka et al. (2015) reported that the residual values detected in 40 cucumbers and 42 tomato samples collected in southeast Poland were below the MRL values. Adeniyi et al. (2016) reported that the residual values detected in 6 tomato samples in Louisiana, USA was below the MRL values. Zengin and Karaca (2017), Polat and Tiryaki (2018), and Hepsağ (2019) indicated that the residue levels in tomato samples were collected from open and greenhouse tomato growing areas in Uşak, Çanakkale, and the Mediterranean region, respectively were below the MRL. The residue values in tomato and cucumber samples from İzmir province were below the MRL, while no pesticides were detected in pepper samples (Kaya and Tuna 2019). Algharibeh and Al Fararjeh (2019) reported that the residual values detected in 40 tomato samples were below the MRL, while pesticide content in 17 of 32 pepper samples in Jordan was above the MRL. Velioglu et al. (2019) stated that the residual values in tomato samples taken from Tekirdag province, Turkey were close to the EU-MRL values. Gölge and Kabak (2018) determined that the pesticide content of 5 tomato samples from Mersin and Antalya provinces was higher than the MRL value. Loughlin et al. (2018) reported that the residue level in 2 of 10 tomato samples and 7 of 23 pepper samples in Argentina were above the MRL. Elmastaş (2018) determined that the residual values of 2 tomato samples from Diyarbakır province, Turkey were above the MRL, and the residue level in cucumber samples was lower than the MRL values. Salamzadeh et al. (2018) reported that the residue levels in 4 of 150 tomato samples in Iran were above the MRL. Çiftçi (2019) detected residues in 10 tomato samples and 14 pepper samples from Çanakkale province, Turkey higher than the maximum residue limits (MRL) specified in the Turkish Food Codex (TGK 2021). Hu et al. (2020) reported that the residual values detected in 22 dried cucumbers and 40 pepper samples in Jilin, China were below the MRL, while the residue level in 3 of 31 tomato samples were above the MRL. Yi et al. (2020) reported that the residue values in 638 cucumber samples in Korea were below the MRL, and 1 of 149 tomato and 638 pepper samples had residue above the MRL. Osaili et al. (2020) reported that the residue levels in 87 of 233 cucumber samples, 41 of 205 tomato samples, and 130 of 316 pepper samples in the United Arab Emirates were above the MRL value. Ramadan et al. (2020) indicated that detectable pesticide residues in 44 samples (20.9%) were above MRLs, and residue level in 145 samples (68.7%)

Table 3. Pesticide residue levels and frequencies

Matrix	No. of samples detectable residues (%)	No. of samples >MRL (%)	Pesticides	Frequency of detection	Pesticide residue (µg kg^{-1})	No. of samples >MRL
Pepper	6 (60)	1 (10)	Acetamiprid	3	14.58- 27.33- 28.83	
			Azoxystrobin	1	11.46	
			Bifenazate	1	21.23	
			Boscalid	2	16.39- 664.88	
			Kresoxim methyl	1	53.35	
			Pyridaben	4	11.06- 37.61- 44.18- 684.36	1
			Pyrimethanil	1	60.07	
Tomato	3 (37.5)	-	Acetamiprid	1	21.74	
			Azoxystrobin	1	34.17	
			Boscalid	1	37.21	
			Famoxadone	1	58.99	
			Penconazole	1	10.87	
			Pyridaben	2	13.73- 40.43	
Cucumber	7 (70)	2 (20)	Acetamiprid	2	9.90- 51.53	
			Azoxystrobin	3	17.36- 35.45- 56.69	
			Bifenazate	2	18.71- 28.21	
			Boscalid	1	522.95	
			Clofentezine	2	25.95- 67.54	
			Fluopyram	3	18.20- 37.02- 49.35	
			Hexythiazox	1	8.47	
			Kresoxim-methyl	2	107.30- 419.48	
			Metrafenone	2	1412.11- 1501.30	2
			Penconazole	1	12.76	
			Pirimicarb+ Pirimicarb-desmethy	1	65.73	
			Tebuconazole	2	7.93- 22.70	
			Tetraconazole	2	45.37-60.19	
			Thiacloprid	1	7.68	

was lower than MRLs. The MRL values in chili pepper (14 samples) and cucumber (10 samples) were high. Soydan et al. (2021) found the residual values determined in peppers grown in the Aegean region lower than the MRL. The residue values in 2 cucumber samples were above the MRL. Çatak and Tiryaki (2020) determined the MRL value of 1 cucumber sample collected from Çanakkale open markets was above the MRL.

Residues of samples

Risk analysis was carried out for 27 pesticides and the results are given in Table 4.

The short-term risk assessment revealed that pyridaben level possesses high risk with a value of 1.3139, while all other aHI values were less than 1 which indicates a negligible acute risk. In the long-term risk assessment, the chronic risk index (cHI) values were considerably higher than the aHI values. The results indicate that the chronic risk from pesticide exposure through the consumption of peppers, tomatoes, and cucumbers should be considered. The risk assessment considering the pesticide exposures in tomato and cucumber vegetables showed that the pesticide residues would not pose a risk for the Kazakh people (Lozowicka et al. 2015). However, the estimated dietary pesticide exposures considered only tomato and cucumber exposures. Yi et al. (2020) stated that pesticide residues may not be considered a serious public health problem in Korea. Theoretical maximum daily intake evaluation of Çatak and Tiryaki (2020) showed that the pesticides in cucumber did not pose a risk of chronic exposure. Soydan et al. (2021) reported that pesticide residue levels detected cannot be considered a serious public health problem. Darko and Akoto (2008)

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

Matrix	Pesticide	Long-term risk EDI (mg $kg^{-1}day^{-1}$)	cHI	Short-term risk ESTI (mg $kg^{-1}day^{-1}$)	aHI
Pepper	Acetamiprid	2.26211E-05	0.0905	2.76751E-05	0,1107
	Azoxystrobin	1.0994E-05	0.0055	1.10009E-05	/
	Bifenazate	2.03667E-05	0.2037	2.03795E-05	/
	Boscalid	0.000326783	0.8170	0.000638245	/
	Kresoxim-methyl	5.11805E-05	0.0128	5.12128E-05	/
	Pyridaben	0.000186399	1.8640	0.000656944	1.3139
	Pyrimethanil	5.76272E-05	0.4802	5.76636E-05	/
Tomato	Acetamiprid	9.25963E-05	0.3704	2.08691E-05	0.0835
	Azoxystrobin	0.000145539	0.0728	3.28011E-05	/
	Boscalid	0.000158487	0.3962	3.57194E-05	/
	Famoxadone	0.000251254	2.0938	5.66268E-05	0.0283
	Penconazole	4.62981E-05	0.1543	1.04345E-05	0.0021
	Pyridaben	0.000115341	1.1534	3.88104E-05	0.0776
Cucumber	Acetamiprid	2.13811E-05	0.0855	4.94657E-05	0.1979
	Azoxystrobin	2.54122E-05	0.0127	5.4419E-05	0.1814
	Bifenazate	1.63334E-05	0.1633	2.70799E-05	/
	Boscalid	0.000364091	0.9102	0.000502	/
	Clofentezine	3.25415E-05	0.1627	6.48343E-05	/
	Fluopyram	2.42634E-05	0.2022	4.7373E-05	0.0095
	Hexythiazox	6.085E-06	0.0203	8.38987E-06	/
	Kresoxim-methyl	0.000183379	0.0458	0.000402675	/
	Metrafenone	0.001014191	0.4057	0.001441157	/
	Penconazole	8.88383E-06	0.0296	1.22488E-05	0.0024
	Pirimicarb+ Pirimicarb-desmethy	4.57629E-05	0.1308	6.30968E-05	0.0631
	Tebuconazole	1.06592E-05	0.0355	2.17906E-05	0.0726
	Tetraconazole	3.67467E-05	0.9187	5.77788E-05	0.1156
	Thiacloprid	5.34701E-06	0.0535	7.37234E-06	0.0246

The symbol of "/" represented that there was no authorized value for ARfD, and the corresponding risk index could not be computed.

reported that some health risks may occur due to the OP residues detected in tomatoes and eggplants collected from a market in Ghana Kumasi. Bolor et al. (2018) stated that the residues on vegetables consumed by children may pose both carcinogenic and non-carcinogenic health risks, while the residue level detected may not pose health risks for adults. Kumari and John (2019) reported that methyl parathion and triazophos residues detected in fruit and vegetable samples collected from the West Indian Himalayan region pose a potential threat to human health, especially in children. Odewale et al. (2021) reported that α-HCH and γ-HCH in tomato and watermelon samples may pose carcinogenic health risks in child consumers, and α-HCH in adult consumers. Odewale et al. (2021) reported that the consumption of tomatoes and watermelon containing α-HCH and γ-HCH may pose carcinogenic health risks in child consumers, and α-HCH in adult consumers.

This study was conducted to reveal the pesticide residues in tomatoes, cucumbers, and peppers produced in Tokat province, and the health risk assessments related to pesticide residues. Seventeen different pesticides were detected in 16 of 28 samples. The residue content of 2 pesticides exceeded the MRL levels. Risk analysis was conducted for 27 pesticides in the study. The short-term risk assessment revealed that the highest risk was in pyridaben with a 1.3139 aHI value, and aHI values for other active substances were at a negligible level. The long-term risk assessment showed that cHI values were much higher than the aHI values. The results indicated that the chronic risk arising from pesticide exposure in tomatoes and cucumbers should be taken into account. Potential risks are possible due to prolonged dietary exposure; therefore, reside level should constantly be monitored. Some precautions should be taken into account to minimize such risks. The precautions suggested can be listed as follows. Adopting IPM and GAP approaches to producers is important to reduce both pesticide residue and health risks. Pest control should be carried out regularly by experts, and appropriate doses and application time between the last spraying and harvest should be determined. Farmers should be trained and precautions should be taken regarding the sale of pesticides. Manufacturers should be informed of the consequences of incorrect pesticide use. The use of banned preparations should be strictly avoided. The use of highly toxic pesticides should be limited. Agricultural products should be marketed after the residue analysis. Products with residue problems must be destroyed, and dealers who advise these manufacturers should be punished. To monitor appropriate measures and thus minimize the associated health risks, a national monitoring program and database should be established for locally consumed agricultural products and processed food. Agricultural production cannot be carried out without using pesticides. However, pesticide residue levels should be kept at values below the MRL. Compounds used in agricultural activities have a high tendency to accumulate in human tissues as biomass. Since vegetables are widely consumed, pesticide residues in vegetables may cause serious health problems. Overall, the findings of the study are important as a reference point for future research.

ÖZET

Bu çalışma, Tokat ve çevresinde üretilen domates, biber ve hıyarda pestisit kalıntı düzeylerini tespit etmek ve bu ürünler için sağlık risk değerlendirmelerini belirlemek amacıyla yapılmıştır. Sıvı kromatografi-tandem kütle spektrometresi (LC–MS/MS) ile bu sebzelerdeki 260 pestisit kalıntısının belirlenmesi için analitik metot doğrulaması yapılmıştır. Bunun için, pestisit içermeyen salatalık matrisine 10 ve 50 µg kg^{-1} seviyelerinde pestisit çözeltisi eklenmiştir. Pestisitlerin doğrusallıkları (R^2), tayin limitleri (LOD), tespit limitleri (LOQ) ve ortalama geri kazanımları sırasıyla 0.990-0.999, 0.71-2.96 µg kg^{-1}, 2.36-9.86 µg kg^{-1} ve %77.25-117.61 arasında bulunmuştur. Bu yöntemle, Tokat (Türkiye) ilinden toplanan 28 sebze örneği analiz edilmiştir. 16 örnekte 17 farklı pestisit tespit edilmiş ve bunların 3'ünde pestisit konsantrasyonları Avrupa Birliği Maksimum Kalıntı Limitlerinden (AB-MRL) yüksek bulunmuştur. 1 biber örneğinde pyridaben ve 2 hıyar örneğinde metrafenone için AB-MRL değerleri aşılmıştır. Örneklerde tespit edilen pestisitlere yönelik risk değerlendirmesinde famoxadone ve pyridaben'in tüketiciler için kronik toksisite potansiyeline sahip olduğu kanaatine varılmıştır.

Anahtar kelimeler: akut risk, kronik risk, LC-MS/MS, metot doğrulama, QuEChERS

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