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ARAŞTIRMA MAKALESİ **RESEARCH ARTICLE**

Pesticide Residues in Table Grapes and Health Risk Assessment

Sofralık Üzümlerde Pestisit Kalıntıları ve Sağlık Risk Değerlendirmesi

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Abstract

Pesticides are widely used in grape production to control pests and diseases, protect yield, and maintain commercial quality. This study investigated pesticide residues in fresh table grapes collected from Salihli, Alaşehir, and Sarıgöl (Manisa Province, Türkiye). A multiresidue QuEChERS-LC–MS/MS method was applied, covering 260 active substances across different pesticide classes (insecticides, fungicides, herbicides, acaricides, and nematicides). In total, 54 grape samples were analyzed. At least one pesticide residue was detected in every sample. Two different pesticides were found in 5 samples, while three or more pesticides were identified in 43 samples, indicating frequent co-occurrence of multiple residues. Overall, 32 different active substances were detected. The insecticides identified included acetamiprid, buprofezin, chlorantraniliprole, lambda-cyhalothrin, deltamethrin, flubendiamide, indoxacarb, methoxyfenozide, and tebufenozide. The fungicides identified included ametoctradin, azoxystrobin, boscalid, bupirimate, cymoxanil, cyprodinil, difenoconazole, dimethomorph, famoxadone, fluopicolide, fluopyram, kresoxim-methyl, mandipropamid, metalaxyl-M, metrafenone, myclobutanil, penconazole, pyrimethanil, spiroxamine, tetraconazole, tebuconazole, triadimenol, and trifloxystrobin. Residue concentrations were evaluated against the European Union maximum residue limits (EU-MRLs). Among 301 quantified pesticide-sample combinations, 294 results complied with EU-MRLs, while seven exceeded the corresponding legal limits. Pyrimethanil, boscalid, and cyprodinil were the most frequently detected compounds. Except for bupirimate (a fungicide), all exceedances involved insecticides, specifically acetamiprid, buprofezin, deltamethrin, and methoxyfenozide. The dietary risk assessment revealed that acetamiprid contributed the highest acute exposure, while bupirimate and famoxadone showed relatively higher chronic exposure values. These results emphasize the importance of continuous pesticide residue monitoring in table grapes, ensuring the use of only authorized plant protection products and maintaining residue levels within regulatory limits to safeguard consumer health and environmental sustainability.

Keywords: LC-MS/MS, Multiresidual analysis, Pesticide residue, Sultani seedless grape, QuEChERS

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Öz

Pestisitler, bağcılıkta zararlılar ve hastalıklarla mücadelede, ürün verimini ve ticari kalitesini korumak amacıyla yaygın olarak kullanılmaktadır. Bu çalışmada, Türkiye'nin Manisa iline bağlı Salihli, Alaşehir ve Sarıgöl ilçelerinden toplanan taze sofralık üzüm örneklerinde pestisit kalıntılarının belirlenmesi amaçlanmıştır. Farklı kimyasal sınıflardaki (insektisit, fungisit, herbisit, akarisit ve nematisit) toplam 260 aktif maddeyi kapsayan çoklu kalıntı QuEChERS–LC–MS/MS yöntemi uygulanmıştır. Toplam 54 üzüm örneği analiz edilmiştir. Tüm örneklerde en az bir pestisit kalıntısı tespit edilmiştir. Beş örnekte iki farklı pestisit, kırk üç örnekte ise üç veya daha fazla farklı pestisit kalıntısı belirlenmiş olup, birden fazla kalıntının birlikte bulunması yaygındır. Genel olarak 32 farklı aktif madde tanımlanmıştır. Tespit edilen insektisitler; acetamiprid, buprofezin, chlorantraniliprole, lambda-cyhalothrin, deltamethrin, flubendiamide, indoxacarb, methoxyfenozide ve tebufenozide; fungisitler ise ametoctradin, azoxystrobin, boscalid, bupirimate, cymoxanil, cyprodinil, difenoconazole, dimethomorph, famoxadone, fluopicolide, fluopyram, kresoxim-methyl, mandipropamid, metalaxyl-M, metrafenone, myclobutanil, penconazole, pyrimethanil, spiroxamine, tetraconazole, tebuconazole, triadimenol ve trifloxystrobin olarak belirlenmiştir. Kalıntı konsantrasyonları Avrupa Birliği maksimum kalıntı limitleri (EU-MRLs) ile karşılaştırılmıştır. Analiz edilen 301 pestisit-örnek kombinasyonundan 294'ü yasal sınırlar içinde kalırken, yedisi MRL değerini aşmıştır. En sık tespit edilen pestisitler pyrimethanil, boscalid ve cyprodinil'dir. Bupirimate (fungisit) dışındaki tüm aşımlar insektisit grubundaki bileşiklerle (acetamiprid, buprofezin, deltamethrin ve methoxyfenozide) ilişkilidir. Yapılan diyet risk değerlendirmesi sonucunda, akut maruziyet açısından en yüksek katkı acetamiprid tarafından sağlanırken, kronik maruziyette bupirimate ve famoxadone diğer pestisitlere göre daha yüksek değerler göstermiştir. Elde edilen bulgular, sofralık üzüm üretiminde pestisit kalıntılarının düzenli olarak izlenmesinin, yalnızca ruhsatlı bitki koruma ürünlerinin kullanılmasının ve kalıntı düzeylerinin yasal sınırlar içinde tutulmasının hem tüketici sağlığının korunması hem de çevresel sürdürülebilirlik açısından büyük önem taşıdığını ortaya koymaktadır.

Anahtar Kelimeler: LC-MS/MS, Çoklu kalıntı analizi, Pestisit kalıntısı, Sultani çekirdeksiz üzüm, QuEChERS

1. Introduction

Grapes, belonging to the Vitis genus of the Vitaceae family, are known as one of the oldest cultivated fruit species in the world, with a history dating back to 3500 B.C. Grapes are among the fruit species with the most varieties compared to others, with over 15,000 varieties reported worldwide and more than 1,200 varieties in Anatolia (Kök et al. 2018; Güçer et al., 2021). The top grape-producing countries in the world are China, Italy, and France, while Türkiye produces approximately 4.17 million tons of grapes on 384,537 hectares. Türkiye ranks 5th in the world in terms of vineyard area and 6th in terms of production volume (FAO, 2024). There are numerous diseases and pests that limit production in viticulture areas. These include pests and diseases such as *Planococcus citri* (Risso, 1813) (Hemiptera; Pseudococcidae), *Empoasca vitis* (Gothe, 1875) (Hemiptera; Cicadellidae), *Rubiothrips vitis* (Priesner, 1933) (Thysanoptera; Thripidae), *Lobesia botrana* (Denis and Schiffermüller, 1775) (Lepidoptera; Tortricidae), *Tetranychus urticae* (Koch, 1836) (Acarina: Tetranychidae), *Erysiphe necator* (Schw.) (Grape Powdery Mildew), *Plasmopara viticola* (Grape Downy Mildew), *Botrytis cinerea* (Pers. Ex. Fr.), *Phomopsis viticola* (Sacc.), *Phellinus signiarius* (L.) Quél., and *Fomitiporia mediterranea* M. Fisch. (TAGEM, 2017).

Producers are increasingly turning to pesticides in pest control to cultivate high-quality and high-yield crops (Köycü et al. 2018). There is significant interest in pesticide mixtures in vineyard areas to save time, energy, and labor. As a result, both single and multiple pesticide residues can be found in the produced goods. Additionally, the number of available preparations used in vineyards is decreasing due to bans, and introducing new products to the market takes time. Therefore, the frequency of use of currently authorized pesticides is also increasing. Producers often prefer chemical control due to their easy availability, ease of application, and the rapid appearance of their effects. However, alongside these advantages, pesticides have undesirable consequences such as environmental pollution, resistance, and pesticide residues. In addition, the harmful effects of pesticide use on human health have become an alarming issue, raising severe concerns in society, particularly concerning food safety in recent years. Thus, monitoring pesticide residues has become essential.

EU countries have implemented the Rapid Alert System for Food and Feed (RASFF), which facilitates swift and coordinated responses to health threats arising from food or feed. Current residue issues are reported to RASFF members' contact points and forwarded to the European Commission to create a safety barrier, ensuring member states take necessary precautions or are informed (EC, 2025). In this context, an analysis of pesticide residue notifications related to grapes issued by RASFF between 2021 and 2024 indicates that acetamiprid, cypermethrin, dithiocarbamates, iprodione, lambda-cyhalothrin, metalaxyl, triadimenol, and pyriproxyfen have exceeded European Union maximum residue limits (MRLs) in Türkiye (RASFF, 2024). At this point, routine residue analyses and health risk assessments are critical. Several studies conducted in Türkiye have reported the presence of pesticide residues in grapes or raisins (Turgut et al., 2010, Turgut et al., 2011; Nalcı et al., 2018; Gazioğlu Şensoy et al., 2017; Golge and Kabak, 2018; Soydan et al., 2021; Kanbolat et al., 2023; Özbek et al., 2025). However, only a limited number of these studies have included a dietary risk assessment, and most relied on samples collected from retail markets rather than directly from commercial vineyards. The present research analyzed fresh table grapes collected directly from commercial vineyards in Manisa, one of the major grape production regions in Türkiye, using a validated LC–MS/MS multiresidue method in accordance with SANTE (2021) guidelines, assessed compliance with MRLs, and evaluated both acute and chronic dietary exposure risks. In this way, the study provides a more comprehensive food safety perspective and complements earlier national research.

2. Materials and Methods

2.1. Reagents and chemicals

Certified reference standards of all target pesticides were obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany). LC-grade acetonitrile (MeCN, >99 % purity) and methanol (MeOH, >99 % purity), as well as glacial acetic acid, were supplied by Merck (Darmstadt, Germany). Anhydrous magnesium sulfate ($MgSO_4$, ≥99 % purity) and anhydrous sodium acetate (CH_3COONa, ACS reagent grade, assay 99–101 %) were purchased from AFG Bioscience LLC (Northbrook, IL, USA). Ammonium formate (NH_4HCOO) used for mobile phase buffering was obtained from Sigma-Aldrich Chemie GmbH (Steinheim, Germany). Primary–secondary amine (PSA) sorbent (40 µm particle size) employed in the dispersive clean-up step was sourced from Supelco Analytical (Bellefonte, PA,

USA).

2.2. Sample collection and storage

Fresh table grape samples were collected in 2022 from three major production districts in Manisa Province (Salihli, Alaşehir, and Sarıgöl). The Sultani variety was sourced from Sarıgöl, while early and other varieties (Superyol, Thomson, Mevlana, Royal, Alfonso, Crimson, and Redglobe) were obtained from Alaşehir, Salihli, and Sarıgöl. Samples were collected from the large-size fresh produce category (grape clusters), with a minimum of 2 kg (at least five units) per sample and were transported to the laboratory under refrigerated conditions within 24 hours (EC, 2002). The samples were homogenized, and 15 g was weighed into a 50 ml Falcon tube, then stored at -20 ˚C until analysis.

2.3. Instrumentation

The analyses were carried out using a Shimadzu LCMS-8050 triple quadrupole mass spectrometer (Shimadzu Corporation, Kyoto, Japan) coupled to a Nexera X2 UHPLC system. The LC–MS/MS system consisted of two LC-30AD pumps, a SIL-20A autosampler, a DGU-20A3R degasser, and a CTO-20ACV column oven. Chromatographic separation was performed on a Purospher STAR RP-18 endcapped column (2.1 × 100 mm, 2 µm; Merck, Darmstadt, Germany) maintained at 40 °C. The mobile phases were (A) 5 mmol L^{-1} ammonium formate in distilled water and (B) 5 mmol L^{-1} ammonium formate in methanol. The gradient started at 5% B at 0.00 min, increased to 60% B at 3 min, 70% B at 4 min, 80% B at 6 min, and 95% B between 7 and 8.5 min, and then returned to 5% B for re-equilibration until 15 min. The flow rate was 0.4 mL min^{-1} and the injection volume was 10 µL.

The mass spectrometer was operated with an electrospray ionization (ESI) source in both positive and negative modes. Source/desolvation conditions were as follows: desolvation line temperature 250 °C, interface temperature 300 °C, and heat block temperature 400 °C. Multiple reaction monitoring (MRM) mode was used for quantification, and for each analyte, at least two transitions (quantifier and qualifier ions) were monitored. Data acquisition and quantification were performed using LabSolutions software (Shimadzu, Japan).

2.4. Method validation

Method validation and performance evaluation were carried out to ensure analytical quality control of the multi-residue procedure. Although QuEChERS is widely accepted in advanced pesticide residue laboratories, its performance must be demonstrated under local laboratory conditions before routine use (Dülger and Tiryaki, 2021). Therefore, the method was assessed in terms of linearity, sensitivity (limits of detection, LOD; and limits of quantification, LOQ), trueness/accuracy, precision (repeatability and within-laboratory reproducibility), and measurement uncertainty, following the guidance provided in the EU document "Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed" (SANTE, 2021).

Linearity was evaluated by building a seven-point calibration curve using matrix-matched standards prepared at 0 (blank), 5, 10, 25, 50, 100, and 200 µg kg^{-1}. Each level was injected in triplicate. Matrix-matched calibration was used to compensate for matrix effects and allow accurate quantification in grape samples.

The LOD and the LOQ were estimated to characterize the method's detection capability. For this purpose, pesticide-free matrix (grape) was fortified at 10 µg kg^{-1} with a mixed working standard solution and analyzed repeatedly (n = 10). The standard deviation (SD) of these replicate measurements was calculated. The LOD was defined as three times this SD, and the LOQ as ten times the SD (EURACHEM, 2014).

Accuracy (trueness) and precision were assessed by recovery studies. Pesticide-free grape samples were fortified at two concentration levels (10 and 50 µg kg^{-1}) and analyzed in five replicates at each level. Repeatability (RSDr) was evaluated under same-day conditions by two independent analysts. Within-laboratory reproducibility (RSDwR) was assessed by repeating the analyses over five consecutive weeks, again with two analysts, to represent routine laboratory conditions. Precision was expressed as relative standard deviation (RSD, %). Method performance was judged according to the criteria given in SANTE (2021), which require mean recoveries to fall within an acceptable range and precision (repeatability and within-laboratory reproducibility) to remain within predefined RSD limits. In addition, the expanded measurement uncertainty (U′) for each analyte was estimated following "Approach 2" described in SANTE (2021).

2.5. Sample preparation, extraction, and clean up

The extraction and clean-up procedures, based on the QuEChERS AOAC Method 2007.01, were performed according to the guidelines provided by Lehotay (2007). The procedural steps for the QuEChERS method are illustrated in Figure 1. LC–MS/MS analyses were performed in triplicate.

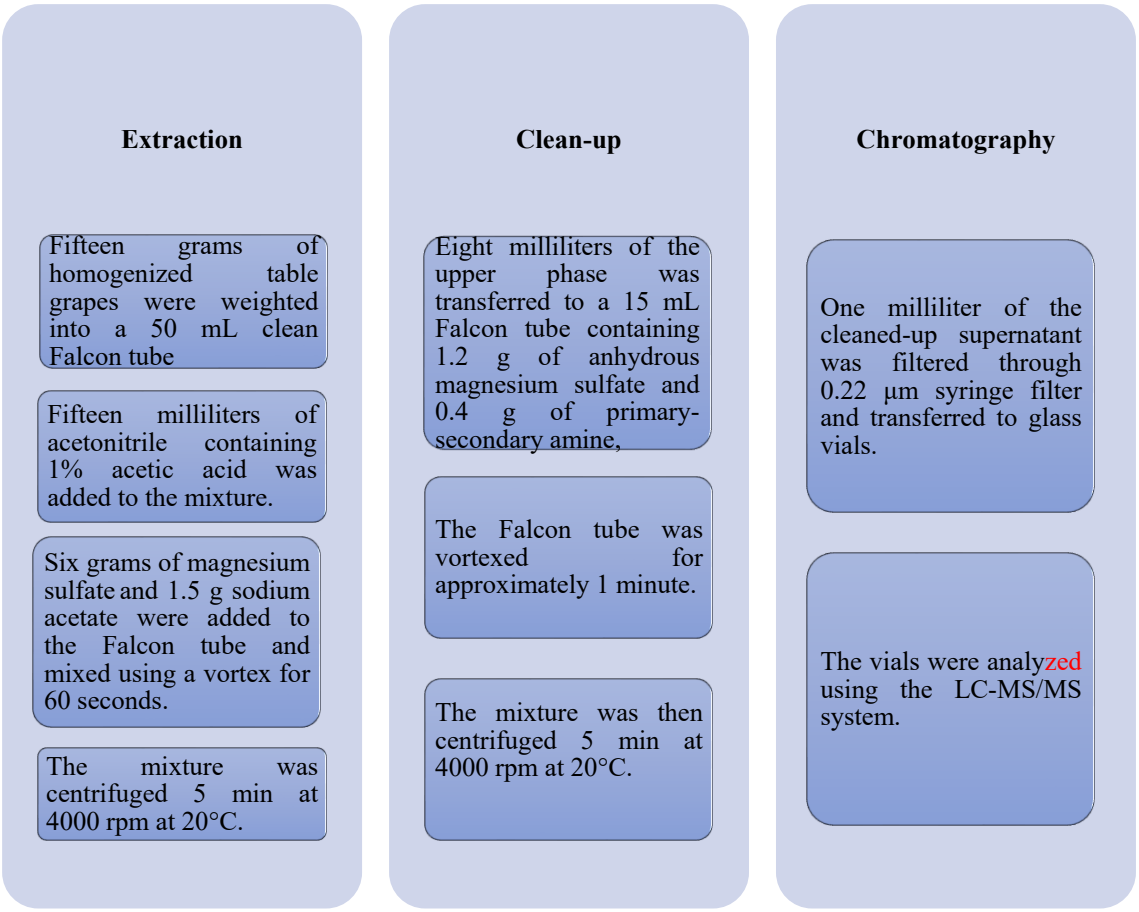

Figure 1. Analytical Steps of the QuEChERS-AOAC Official Method 2007.01. (Lehotay, 2007)

2.6. Pesticide residues in table grapes

A total of 54 fresh table grape samples were analyzed. To determine residues of pesticides with different chemical structures, a validated QuEChERS–LC–MS/MS method was applied following extraction and clean-up. For each sample, three analytical portions were prepared, and all measurements were carried out in triplicate. Identification of each pesticide was based on two independent criteria: (i) retention time (RT) matching that of the corresponding reference standard and (ii) compliance with the required ion ratio between the quantifier and qualifier transitions, in accordance with SANTE (2021).

The quantified residues were then compared with the MRLs established for table grapes in the European Union (EU-MRL, 2024). This evaluation was used to determine both legal compliance (MRL exceedances) and the occurrence of non-authorized active ingredients in grape samples.

2.7. Health risk assessment

The health risk assessment was carried out to evaluate both acute and chronic dietary exposure to pesticide residues. The methodology was adapted from EFSA (2015) and Liu et al. (2016), and calculations were performed in relation to toxicological reference values: the Acceptable Daily Intake (ADI, mg kg^{-1} bw day^{-1}) and the Acute Reference Dose (ARfD, mg kg^{-1} bw day^{-1}). For each pesticide, the estimated daily intake (EDI) and the estimated short-term intake (ESTI) were calculated using the mean (MR) and high residue (HR) concentrations, respectively. For the Turkish population, an average adult body weight (bw) of 73.7 kg was assumed (TUIK, 2024), along with a daily grape consumption (FC) rate of 0.058 kg per person (TUIK, 2023). The calculations were performed using the

following equations:

$$ESTI = \frac{\text{HR } x \text{ FC}}{\text{bw}} \quad \text{(Eq. 1)}$$

$$HQa = \left(\frac{\text{ESTI}}{\text{ARfD}}\right) x\ 100 \quad \text{(Eq. 2)}$$

$$EDI = \frac{\text{MR } x \text{ FC}}{\text{bw}} \quad \text{(Eq. 3)}$$

$$HQc = \left(\frac{\text{EDI}}{\text{ADI}}\right) x\ 100 \quad \text{(Eq. 4)}$$

Health risks were expressed as hazard quotients: the acute hazard quotient (HQa) for short-term exposure and the chronic hazard quotient (HQc) for long-term exposure. HQa and HQc values greater than 100% were therefore considered indicative of unacceptable health risk, while values below 100% were considered acceptable (Akoto et al., 2015; Soydan et al., 2021).

3. Results and Discussion

3.1. Method validation

Validation results for the analytical method applied to grape samples are summarized in *Table 1*. Calibration curves for all target pesticides showed good linearity, with coefficients of determination (R^2) ranging from 0.9902 to 0.9986. The LODs ranged from 0.78 to 2.99 µg kg^{-1}, while the LOQs were between 2.61 and 9.96 µg kg^{-1}. Apparent recoveries obtained during the validation study were between 78.70% and 112.96%, and method precision, expressed as relative standard deviation (RSD), varied from 2.22% to 13.68% (*Table 1*). In this study, all validated pesticides showed recovery values within 70–120% and precision values (RSD) below 20%, indicating that the method satisfied the performance requirements set out in SANTE (2021). The expanded measurement uncertainty (U′) for the quantified pesticides was in the range of 17.4% to 41.8%, remaining below the 50% limit indicated by SANTE (2021). All pesticides included in this study fulfilled these performance criteria (*Table 1*).

3.2. Pesticide residues analyses in table grapes

The results of the pesticide residue analysis conducted on 54 grape samples are summarized in *Table 2*.

The analysis revealed that all 54 grape samples contained at least one pesticide residue. Specifically, five samples exhibited two pesticide residues, while 43 samples had three or more pesticide residues. Of the 301 quantified pesticide residues detected in the samples, 294 were below the EU-MRLs, while 7 residues exceeded the EU-MRL values. The results indicated the presence of a total of 32 pesticides in the grapes. Among these, nine were insecticides, including acetamiprid, buprofezin, chlorantraniliprole, lambda-cyhalothrin, deltamethrin, flubendiamide, indoxacarb, methoxyfenozide, and tebufenozide. The remaining 23 were fungicides, which included ametoctradin, azoxystrobin, boscalid, bupirimate, cymoxanil, cyprodinil, difenoconazole, dimethomorph, famoxadone, fluopicolide, fluopyram, kresoxim-methyl, mandipropamid, metalaxyl-M, metrafenone, myclobutanil, penconazole, pyrimethanil, spiroxamine, tetraconazole, tebuconazole, triadimenol, and trifloxystrobin. The pesticides detected in ten or more samples are presented in *Figure 2*, most frequently being pyrimethanil, boscalid, and cyprodinil.

Excluding bupirimate, all pesticides exceeding the Maximum Residue Limits (MRLs) were insecticides, namely acetamiprid, buprofezin, deltamethrin, and methoxyfenozide. Notably, buprofezin is not authorized for use against any pests in vineyards. The high levels of insecticide residues above MRLs are thought to be associated with frequent applications and insecticide resistance. Indeed, in applications against the grape berry moth in Manisa province, varying degrees of resistance have been detected for active ingredients such as emamectin benzoate, deltamethrin, spinosad, lambda-cyhalothrin, indoxacarb, and chlorpyrifos-ethyl (Mermer, 2012; Hatipoğlu et al., 2015). Consequently, due to the pests developing resistance, producers resort to repeated applications and overdoses, which subsequently reflect in residue outcomes.

Table 1. Method validation parameters

Pesticide	RT (min)	Precursor ion, m z^{-1}	Product ion, m z^{-1} (CE, eV)	Correlation coefficient (R^2)	LOD (µg kg^{-1})	LOQ (µg kg^{-1})	Fortification (µg kg^{-1})	Measured (µg kg^{-1})	Recovery (%)	RSD (%)	U (%)
Acetamiprid	4.182	222.90	126.00 (-20) 56.10 (-15)	0.9958	1.13	3.76	10 50	8.51 49.62	85.10 99.24	3.02 2.22	24.3
Ametoctradin	9.915	276.10	149.15 (-36) 176.10 (-28)	0.9955	1.25	4.17	10 50	10.07 50.83	100.70 101.66	9.93 4.75	30.1
Azoxystrobin	8.073	403.70	272.05 (-14) 329.00 (-29)	0.9906	0.78	2.61	10 50	9.33 39.35	93.31 78.70	3.77 3.55	25.9
Boscalid	8.230	342.90	307.00 (-21) 140.00 (-19)	0.9955	2.18	7.26	10 50	10.45 50.05	104.50 100.10	7.64 8.47	28.2
Bupirimate	9.386	316.90	108.00 (-25) 166.10 (-23)	0.9935	2.80	9.33	10 50	10.11 47.73	101.13 95.46	7.09 4.82	27.9
Buprofezin	10.350	305.90	57.05 (-23) 201.10 (-11)	0.9920	1.56	5.21	10 50	9.69 47.88	96.86 95.77	6.51 3.92	22.8
Chlorantraniliprole	7.743	483.90	452.90 (-19) 285.90 (-17) 112.00 (-55)	0.9908	2.22	7.42	10 50	8.87 44.36	88.66 88.72	8.58 4.81	25.6
Lambda-Cyhalothrin	10.927	467.20	450.10 (-10) 225.00 (-16)	0.9912	2.47	8.24	10 50	8.95 46.11	89.45 92.22	10.86 4.92	23.1
Cymoxanil	4.376	199.20	128.15 (-8) 111.00 (-16)	0.9968	1.08	3.60	10 50	8.05 41.93	80.52 83.87	5.45 2.31	38.8
Cyprodinil	9.847	226.00	93.10 (-37) 108.10 (-26) 77.10 (-48)	0.9970	2.19	7.31	10 50	10.71 55.63	107.13 111.26	11.92 4.61	29.7
Deltamethrin	11.176	523.00	280.70 (-16) 505.90 (-9)	0.9965	2.51	8.36	10 50	9.41 48.25	94.10 96.51	10.23 7.40	30.3
Difenoconazole	9.898	406.00	251.00 (-25) 111.00 (-55)	0.9944	1.98	6.60	10 50	10.09 49.45	100.87 108.89	7.87 4.06	26.5
Dimethomorph	8.424	388.00	301.05 (-21) 165.10 (-33)	0.9902	1.60	5.35	10 50	9.78 45.55	97.80 91.10	3.91 3.95	33.5
Famoxadone	9.537	392.10	331.10 (-9) 93.10 (-37)	0.9986	2.86	9.54	10 50	8.22 46.97	82.22 93.94	5.87 4.31	39.6
Flubendiamide	9.287	680.90	254.20 (29) 272.10 (18)	0.9924	2.30	7.67	10 50	9.69 47.35	96.86 94.70	11.82 3.73	22.9
Fluopicolide	8.308	383.00	172.85 (-13) 144.95 (-50) 108.95 (-55)	0.9968	2.79	9.29	10 50	8.00 42.17	80.01 84.34	7.36 4.19	21.8
Fluopyram	8.676	397.00	145.00 (-39) 173.00 (-19) 208.00 (-18)	0.9940	2.66	8.88	10 50	10.16 45.81	101.65 91.62	11.84 2.97	20.7
Indoxacarb	9.844	528.10	203.00 (-37) 150.10 (-24) 218.00 (-24)	0.9923	1.41	4.71	10 50	9.83 46.18	98.29 92.36	12.76 2.45	17.4
Kresoxim-methyl	9.282	314.10	206.00 (-40) 222.05 (-15)	0.9922	2.49	8.29	10 50	9.36 48.86	93.60 97.72	9.99 2.87	17.7
Mandipropamid	8.203	412.10	327.90 (-10) 125.10 (-36) 355.90 (-12)	0.9950	2.02	6.72	10 50	9.87 40.85	98.69 81.69	8.17 4.23	35.3
Metalaxyl-M	7.379	280.05	220.05 (-15) 192.05 (-20)	0.9949	2.60	8.67	10 50	8.97 42.82	89.66 85.64	9.80 7.08	41.8
Methoxyfenozide	8.499	369.20	149.15 (-8) 91.15 (-47) 312.95 (-9)	0.9913	2.23	7.42	10 50	9.21 43.50	92.10 86.99	10.05 5.48	33.5
Metrafenone	9.866	409.00	209.10 (-6) 227.00 (-23)	0.9906	1.80	5.99	10 50	9.19 49.20	91.91 98.39	8.50 6.17	22.3
Myclobutanil	8.431	289.10	116.10 (-36) 70.00 (-17) 125.00 (-30)	0.9904	2.10	7.01	10 50	8.87 46.93	88.74 93.86	12.42 4.52	29.4
Penconazole	9.335	284.05	70.05 (-16) 158.95 (-28)	0.9925	2.81	9.37	10 50	9.08 48.84	90.78 97.69	8.13 6.98	24.4
Pyrimethanil	8.592	200.10	107.00 (-23) 82.10 (-26)	0.9965	1.36	4.53	10 50	8.56 44.92	85.59 89.84	13.68 4.17	35.5
Spiroxamine	9.591	298.00	144.10 (-19) 100.10 (-29)	0.9941	1.15	3.84	10 50	8.11 48.45	81.08 96.90	11.07 6.62	28.8
Tebuconazole	9.341	308.10	70.05 (-22) 125.00 (-35)	0.9943	2.41	8.03	10 50	8.96 44.00	89.57 87.99	9.13 5.82	32.9
Tebufenozide	10.591	353.00	133.00 (-20) 105.10 (-43)	0.9927	2.99	9.96	10 50	10.50 56.48	105.03 112.96	11.28 4.59	34.7
Tetraconazole	8.760	371.80	159.00 (-30) 70.10 (-21) 159.00 (-29)	0.9979	2.04	6.80	10 50	9.72 49.89	97.20 99.77	12.15 5.62	34.4
Triadimenol	8.293	296.00	70.10 (-21) 227.00 (-9) 43.10 (-49)	0.9945	1.36	4.53	10 50	8.05 47.50	80.51 95.00	3.67 3.98	36.7
Trifloxystrobin	9.942	409.10	186.10 (-18) 145.05 (-44)	0.9926	1.98	6.61	10 50	9.07 49.89	90.74 99.78	9.97 5.28	36.3

Table 2. Frequency, Concentration Range, and Maximum Residue Limits of Detected Pesticides in Table Grapes

Number of Samples >LOQ and Percentage (%)	Number of Samples >MRL and Percentage (%)	Pesticide	Frequency of detection	Pesticide residue (mg kg^{-1})	Number of samples >MRL	MRL* (mg kg^{-1})
54 (100)	7 (13)	Acetamiprid	18	0.013-0.590	1	0.5
		Ametoctradin	16	0.019-0.564	-	6
		Azoxystrobin	7	0.006-0.757	-	3
		Boscalid	40	0.005-2.759	-	5
		Bupirimate	2	1.606-4.930	2	1.5
		Buprofezin	2	0.019-0.234	2	0.01
		Chlorantraniliprole	1	0.554	-	1
		Lambda-Cyhalothrin	10	0.005-0.132	-	0.08
		Cymoxanil	1	0.047-0.047	-	0.30
		Cyprodinil	37	0.008-1.506	-	3
		Deltamethrin	1	0.318	1	0.2
		Difenoconazole	2	0.075-0.348	-	3
		Dimethomorph	18	0.012-0.603	-	3
		Famoxadone	19	0.013-1.081	-	2
		Flubendiamide	3	0.082-0.132	-	2
		Fluopicolide	2	0.055-0.085	-	2
		Fluopyram	11	0.013-0.234	-	2
		Indoxacarb	1	0.016	-	2
		Kresoxim-methyl	5	0.034-0.172	-	1.5
		Mandipropamid	4	0.046-0.102	-	2
		Metalaxyl-M	16	0.015-0.713	-	2
		Methoxyfenozide	9	0.057-1.083	1	1
		Metrafenone	9	0.084-1.187	-	7
		Myclobutanil	2	0.048-0.079	-	1.5
		Penconazole	8	0.006-0.112	-	0.5
		Pyrimethanil	41	0.005-1.967	-	5
		Spiroxamine	1	0.264	-	0.6
		Tetraconazole	1	0.047	-	0.5
		Tebuconazole	6	0.060-0.217	-	0.5
		Tebufenozide	3	0.031-0.051	-	4
		Triadimenol	1	0.193	-	0.3
		Trifloxystrobin	5	0.027-0.118	-	3

*European Union pesticides database (EU-MRL, 2024)

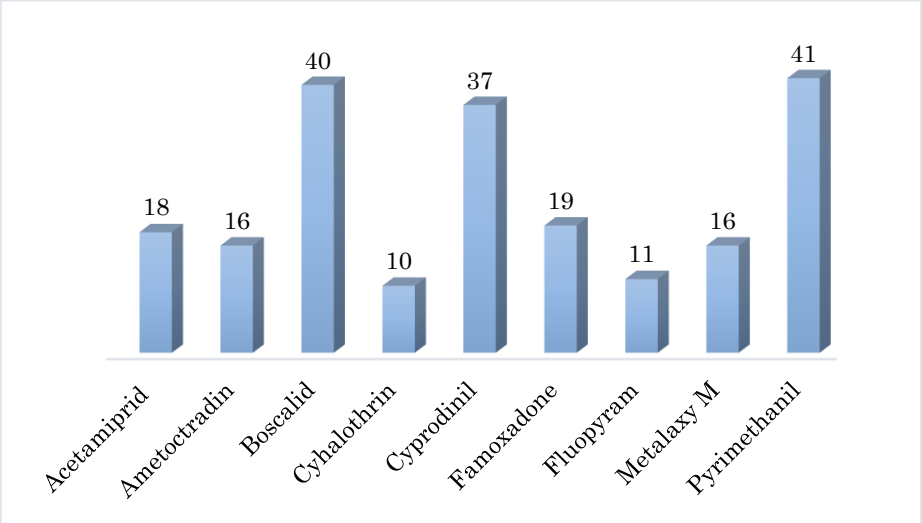

Figure 2. Pesticides in Ten or More Samples and Their Frequencies

Previous studies on pesticide residues in grapes have reported the presence of various active substances, several of which overlap with those detected in the present study. These include azoxystrobin and deltamethrin (Tatlı, 2006); myclobutanil and metalaxyl (Venkateswarlu et al., 2007); azoxystrobin, cyprodinil, deltamethrin, metalaxyl, myclobutanil, penconazole, tebuconazole, and trifloxystrobin (Poulsen et al., 2007); fluopyram, indoxacarb, metrafenone, pyrimethanil, tebuconazole, and tetraconazole (Dinçay et al., 2017); boscalid, azoxystrobin, metalaxyl-M, myclobutanil, dimethomorph, cyprodinil, famoxadone, pyrimethanil, trifloxystrobin, deltamethrin, tebuconazole, penconazole, and indoxacarb (Gazioğlu Şensoy et al., 2017); and acetamiprid, azoxystrobin, boscalid, cyprodinil, difenoconazole, dimethomorph, famoxadone, fluopyram, kresoxim-methyl, penconazole, spiroxamine, tetraconazole, and trifloxystrobin (Golge and Kabak, 2018). Similar findings were reported by Nalcı et al. (2018), Yakar (2018), Kaya and Tuna (2019), and Zengin and Karaca (2019), who identified a wide range of residues including azoxystrobin, boscalid, pyrimethanil, metalaxyl, and triadimenol. Likewise, İçli and Kahyaoğlu (2020), Schusterova et al. (2021), Mahdavi et al. (2022), and Kanbolat et al. (2023) detected multiple residues, most of which were below their respective EU-MRLs.

While the majority of previous studies reported residues below MRLs, several investigations have documented MRL exceedances in grape samples. Baša Česnik et al. (2008) identified azoxystrobin, cyprodinil, metalaxyl, myclobutanil, pyrimethanil, and trifloxystrobin, reporting that cyprodinil and pyrimethanil exhibited residue levels exceeding the MRLs. Turgut et al. (2011) identified chlorpyrifos-ethyl, chlorpyrifos-methyl, deltamethrin, and lambda-cyhalothrin in table grapes, reporting that all four pesticides exhibited residue levels exceeding the MRLs in multiple samples. Bouagga et al. (2019) detected acetamiprid, azoxystrobin, boscalid, cyprodinil, difenoconazole, dimethomorph, fluopyram, metalaxyl, metrafenone, pyrimethanil, spiroxamine, tebuconazole, deltamethrin, fluopicolide, myclobutanil, penconazole, triadimenol, and trifloxystrobin, with only deltamethrin showing MRL exceedances. Ashraf et al. (2022) reported acetamiprid exceeding MRL in one sample, while indoxacarb residues were found below MRL in all samples. Toptanci et al. (2021) found that 23% of grape samples contained residues above MRLs, including acetamiprid, chlorpyrifos, propamocarb, propargite, flufenoxuron, methomyl, and fenbutatin oxide. Similarly, Keklik et al. (2025) detected measurable pesticide residues in 97.7% of 462 grape samples, with 52 samples exceeding EU-MRLs.

Compared to previous studies on pesticide residues in grapes, our findings show both similarities and important distinctions. Like earlier investigations conducted in Türkiye (e.g., Dinçay et al., 2017; Golge and Kabak, 2018; Kanbolat et al., 2023), we frequently detected fungicides such as boscalid, pyrimethanil, and cyprodinil, confirming their widespread use in viticulture. However, unlike most previous reports in which residues were largely found within legal limits, our study identified 7 residue values exceeding the EU-MRLs, particularly for insecticides such as acetamiprid, buprofezin, deltamethrin, and methoxyfenozide. These exceedances align with recent findings by Toptanci et al. (2021) and Keklik et al. (2025), suggesting that compliance issues persist. These findings demonstrate the widespread use of various pesticides in grape cultivation and underscore the importance of monitoring compliance with safety standards.

3.2. Health risk assessment

Health risk assessment was conducted based on the average and highest pesticide residue concentrations detected in the samples, as presented in *Table 3*. Among the 32 detected pesticides, acetamiprid exhibited the highest acute risk, with an HQa value of 1.88%, which is still below the threshold level of concern (HQa < 100%). Similarly, deltamethrin (1.01%) and famoxadone (0.86%) were also ranked among the top pesticides in terms of acute risk, though their HQa values remained within acceptable limits.

For chronic exposure, bupirimate showed the highest HQc value of 5.20%, followed by famoxadone (3.85%), indicating that these pesticides may contribute more significantly to cumulative dietary exposure over time. However, all HQc values remained below 100%, indicating that none of the pesticides posed a health risk at the current levels of exposure. These results suggest that, despite the detection of multiple pesticide residues, both acute and chronic dietary risks associated with table grape consumption in this study are within acceptable safety margins for the general population.

Table 3. Health Risk Assessment of Pesticides Residues in Table Grapes

Pesticide	ADI** (mg kg bw^{-1} d^{-1})	ARfD** (mg kg bw^{-1} d^{-1})	ESTI (mg kg^{-1} d^{-1})	HQa (%)	EDI (mg kg^{-1} d^{-1})	HQc (%)
Acetamiprid	0.025	0.025	4.69E-04	1.88	1.31E-04	0.65
Ametoctradin	10	/	4.48E-04	/	2.33E-04	0.001
Azoxystrobin	2	/	6.02E-04	/	4.63E-04	0.01
Boscalid	0.04	/	2.19E-03	/	2.60E-03	1.16
Bupirimate	0.05	/	3.92E-03	/	1.00E-04	5.20
Buprofezin	0.01	0.5	1.86E-04	0.04	4.40E-04	1.00
Chlorantraniliprole	1.56	/	4.40E-04	/	3.87E-05	0.03
Lambda-Cyhalothrin	0.005	/	1.05E-04	/	3.75E-05	0.77
Cymoxanil	0.013	0.08	3.74E-05	0.05	1.96E-04	0.29
Cyprodinil	0.03	/	1.20E-03	/	2.53E-04	0.65
Deltamethrin	0.01	0.025	2.53E-04	1.01	1.68E-04	2.53
Difenoconazole	0.01	0.16	2.77E-04	0.17	1.84E-04	1.68
Dimethomorph	0.05	0.6	4.79E-04	0.08	2.31E-04	0.37
Famoxadone	0.006	0.1	8.59E-04	0.86	8.18E-05	3.85
Flubendiamide	0.017	0.1	1.05E-04	0.10	5.54E-05	0.48
Fluopicolide	0.08	0.18	6.76E-05	0.04	7.69E-05	0.07
Fluopyram	0.012	0.5	1.86E-04	0.04	1.26E-05	0.64
Indoxacarb	0.005	0.005	1.27E-05	0.25	8.52E-05	0.25
Kresoxim-methyl	0.4	/	1.37E-04	/	5.38E-05	0.02
Mandipropamid	0.15	/	8.11E-05	/	1.56E-04	0.04
Metalaxyl-M	0.08	0.5	5.67E-04	0.11	2.73E-04	0.19
Methoxyfenozide	0.1	0.1	8.61E-04	0.86	3.66E-04	0.27
Metrafenone	0.25	/	9.44E-04	/	5.06E-05	0.15
Myclobutanil	0.025	0.31	6.28E-05	0.02	2.78E-05	0.20
Penconazole	0.03	0.5	8.90E-05	0.02	3.72E-04	0.09
Pyrimethanil	0.17	/	1.56E-03	/	2.10E-04	0.22
Spiroxamine	0.025	0.1	2.10E-04	0.21	3.75E-05	0.84
Tetraconazole	0.004	0.05	3.74E-05	0.07	1.19E-04	0.94
Tebuconazole	0.03	0.03	1.73E-04	0.58	3.01E-05	0.40
Tebufenozide	0.02	/	4.05E-05	/	1.53E-04	0.15
Triadimenol	0.05	0.05	1.53E-04	0.31	6.02E-05	0.31
Trifloxystrobin	0.1	0.5	9.38E-05	0.02	1.31E-04	0.06

**ADI and ARfD values are from the Pesticides Properties DataBase (PPDB, 2024)
(ADI: Acceptable Daily Intake, ARfD: Acute Reference Dose, EDI: Estimated Daily Intake, ESTI: Estimated Short-term Intake, HQa: Acute Hazard Quotient, HQc: Chronic Hazard Quotient)

Previous studies have predominantly reported that acetamiprid does not pose a health risk (Golge et al., 2018; Mahadavi et al., 2022; Balkan and Yılmaz, 2022; Dong and Hu, 2023; Li et al., 2023). Nisha et al. (2021) and Keklik et al. (2025) stated that acetamiprid is not risky for adults but may be a concern for children. For bupirimate, researchers such as Alla et al. (2015), Lozowicka et al. (2016), Lemos et al. (2016), and Si et al. (2021) indicated an absence of health risks, whereas Ahmed et al. (2014) and Chen et al. (2023) suggested potential health concerns. Similarly, famoxadone was deemed safe by several researchers (Lemos et al., 2016; Aydin and Ulvi, 2019; Soydan et al., 2021; Mahdavi et al., 2022; Liu et al., 2023; Park et al., 2023), while Balkan and Kara (2022) indicated a health risk.

Overall, while acetamiprid, bupirimate, and famoxadone generally do not pose significant health risks to the adult population based on the current exposure estimates, some studies indicate possible risks under certain exposure scenarios. Therefore, continued monitoring and adherence to good agricultural practices are crucial to minimize potential health concerns.

4. Conclusions

Thirty-two pesticides were identified, including nine insecticides and 23 fungicides. Of the 301 residues found, 294 were within EU Maximum Residue Limits (EU-MRLs), while seven exceeded these limits, mainly involving acetamiprid, buprofezin, deltamethrin, methoxyfenozide, and bupirimate. The detection of unauthorized insecticides such as buprofezin raises concerns. Pesticide use is essential to protect agricultural products from pests and diseases and

enhance crop quality and yield in response to the nutritional needs of a rapidly growing global population. The responsible application of approved pesticides in vineyards, with consideration of pesticide concentrations and pre-harvest intervals, will significantly reduce health risks for consumers. Consequently, developing specific guidelines and regulations is essential for the safety and sustainability of agricultural practices. Therefore, strict monitoring and regulation of pesticide usage are necessary to mitigate these risks and protect human health and the environment. This study underscores the need for rigorous monitoring and regulation of pesticide use to protect consumer health and ensure environmental sustainability.

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Ethical Statement

There is no need to obtain permission from the ethics committee for this study

Conflicts of Interest

The authors have declared no conflict of interest.

Authorship Contribution Statement

Concept: Kara, K., Balkan, T., Eryılmaz, M.; Design: Kara, K., Balkan, T., Eryılmaz, M.; Data Collection or Processing: Balkan, T., Eryılmaz, M.; Statistical Analyses: Balkan, T.; Literature Search: Kara, K., Balkan, T.; Writing, Review and Editing: Kara, K., Balkan, T.

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