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Pesticide residues in peaches and nectarines: Three-year monitoring data and risk assessment

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ABSTRACT

Peaches and nectarines suffer from various diseases annually, prompting widespread pesticide use for pest management. As a result, pesticide residues remain a notable concern for food safety. This study assessed 474 pesticide residues in peaches and nectarines consumed in Turkey, analyzing 459 samples (164 peaches and 295 nectarines) over three harvest seasons (2021–2023). Using a validated method based on QuEChERS (quick, easy, cheap, effective, rugged, and safe) and liquid chromatography-tandem mass spectrometry, in compliance with the European SANTE/11312/2021 Guideline, residues were found in 71.3% of peaches and 71.9% of nectarines. Multiple residues were more prevalent in peaches (49.4%) than nectarines (37.6%), with 12.8% of peaches and 5.8% of nectarines exceeding maximum residue levels. Peaches contained 40 different residues, including 12 unapproved in the European Union, while nectarines had 35 residues, 13 unapproved. Common residues included thiophanate-methyl, acetamiprid, and boscalid. Hazard Index values (0.49% for adults, 0.60% for children) indicated no significant health risk from chronic pesticide exposure through peach and nectarine consumption among Turks.

1. Introduction

Peach and nectarine (*Prunus persica*), a member of the large plant family Rosaceae, are two of the most popular stone fruits worldwide. Peach and nectarine are nutritious and delicious summer fruits grown in temperate zones ranging from 30° to 45° latitude in both hemispheres (Bassi et al., 2016). In 2022, China was the biggest producer of peaches and nectarines, with a production volume of 16.8 million metric tons, accounting for 63.7% of global production. Four Mediterranean countries (Italy, Turkey, Greece, and Spain) contribute about 15% to world production. Other top-producing countries were the United States (2.5%), Iran (2.2%), Chile (1.2%), Egypt (1.0%), and Morocco (0.9%). Turkey was the third-largest producer, with more than 1 million metric tons in 2022. Turkey's peach and nectarine production has been trending upward for the last decade. In 2022, Turkey's peach and nectarine harvest was about 13% higher than in 2021 and nearly 57% higher than 10 years earlier (Food and Agriculture Organization (FAO), 2022). However, the challenging weather conditions, including unseasonably warm weather in late winter followed by late spring cold snaps can cause yield loss in some regions of Turkey.

Peach and nectarine are consumed fresh as well as in the form of juice, syrups, jams, marmalades, canned food, and peach spirits. The edible parts (exocarb and endocarb) of the fruits are a good source of health-promoting compounds, minerals (iron, fluoride, and potassium), and vitamins (vitamin C and vitamin A) (Aubert et al., 2019; Mrázová et al., 2021; Wu, Xu, et al., 2022). Peaches are a rich source of health-promoting bioactive compounds such as flavonoids (flavan-3-ols, anthocyanins), phenolic compounds (hydroxycinnamic acids), and carotenoids (lutein, zeaxanthin, and beta-cryptoxanthin). These chemicals operate as free radical scavengers, supporting healthy aging and reducing the risk of numerous diseases (Serra et al., 2020; Wu et al., 2022b, 2024).

Many of the fruit diseases recurrently afflict peaches and nectarines each year, albeit not simultaneously. Their manifestation follows a

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predictable pattern contingent upon weather conditions and the phenological stages of the stone fruit host, commencing in dormancy and persisting until fruit maturation. Consequently, implementing a comprehensive disease management regimen throughout the growing season becomes imperative to ensure a substantial yield of viable fruit (Douglas, 2024). Brown rot (*Monilinia fructicola*, *Monilinia laxa*), peach leaf curl (*Taphrina deformans*), powdery mildew (*Sphaerotheca pannosa*), peach scab (*Cladosporium carpophilum*), shot hole (*Wilsonomyces carpophilus*), and bacterial spot (*Xanthomonas* spp.) are among the most frequent diseases that affect peaches and nectarines (Gauthier, 2024; Integrated Pest Management (IPM]), 2024; Ministry of Food Agriculture and Livestock (MFAL), 2024). Weather parameters significantly influence disease occurrence and severity. Hence, controlling diseases proves most challenging during years characterized by elevated temperatures, heightened humidity, ample rainfall, and cloud cover (Douglas, 2024). There are also many insect pests (mites, aphids, thrips and leafhoppers) that affect peach and nectarine production in Turkey. Some of the most common of these are European red mites (*Panoncyhus ulmi*), brown mites (*Bryobia rubrioculus*), webspinning spider mites (*Tetranychus urticae*), black peach aphid (*Brachycaudus persicae*), Western flower thrips (*Frankliniella occidentalis*), peachtree borer (*Synanthedon exitiosa*), and Mediterranean fruit fly (*Ceratitis capitata*) (MFAL, 2024).

Pest infestations pose a significant threat to agricultural productivity, leading to substantial losses in crop yields. According to the FAO, plant pests and diseases are responsible for a reduction of approximately 20–40% in global crop yields annually. Increased population expansion and the impacts of climate change exacerbate food insecurity, compounding the losses. It has been reported that agricultural yields are becoming more and more affected by climate change and fluctuation, which could make pest-related problems worse (FAO, 2024).

These diseases and pests affecting stone fruits can be managed efficiently by employing a combination of sanitation, resistance, culture, and pesticide treatments. This comprehensive disease management method reduces dependence on a single type of control and typically leads to a high yield of top-quality fruit. Pesticides include insecticides for controlling a wide range of insects, fungicides for preventing mold and mildew growth, disinfectants for preventing bacterial transmission, herbicides for killing weeds and other undesired plants, and chemicals for controlling mice and rats. These plant protection products play a crucial role in plant health and food security. However, excessive use of pesticides as well as other inappropriate applications of these chemicals can have a detrimental effect on the biodiversity of agricultural soil, water, and plants. This can have adverse effects on the well-being of humans, animals, and plants (Leskovac & Petrović, 2023; Liang et al., 2022).

Currently, global pesticide use surpasses 3.5 million tons per year (FAO, 2023), with over 1000 commercial pesticide formulations available, including chemical, microbiological, and botanical agents. Low concentrations of pesticide residues enter the human diet because of the extensive application of agricultural chemicals in food production. Over the past years, a few studies have reported the presence of multiclass pesticide residues in peaches and nectarines cultivated in Turkey (Dülger & Tiryaki, 2021; Ersoy et al., 2011; Kanbolat et al., 2023; Kaya & Tuna, 2019; Serbes & Tiryaki, 2023). These monitoring studies have typically been carried out over a short period (one-year data). However, the use of pesticides can be influenced by some of the factors, such as climatic conditions, pest abundance, and farmer practices, that vary from year to year. Surveilling pesticide residues for over a year provides a more reliable and thorough dataset; it accounts for possible variations in pesticide application and fruit quality during various harvesting seasons.

Accordingly, it was hypothesized that pesticide residue levels would vary significantly across different harvest seasons due to annual variations in climatic conditions and agricultural practices. It was also predicted that a comprehensive analysis over three consecutive years would yield a more reliable assessment of pesticide contamination and associated health risks for consumers. This study distinguishes itself from previous research by providing a multi-year dataset, capturing seasonal variations in pesticide residues, and offering a comprehensive risk assessment, which enhances its relevance for consumer health and regulatory practices. Thus, the primary goal of the current study was to determine the presence and concentration of 474 multi-class residues in peaches and nectarines from Turkey throughout three consecutive years (2021–2023). To achieve this, the quick, easy, cheap, effective, robust, and safe (QuEChERS) method was used in combination with liquid chromatography-tandem mass spectrometry (LC-MS/MS). Additionally, the study aimed to assess the potential health risks posed to adult and child Turkish consumers due to the identified residues.

2. Materials and methods

2.1. Standards, reagents and chemicals

Standards of 474 pesticides from various chemical families were obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany) or Sigma-Aldrich (Steinheim, Germany). The purity of the standards was >95%. The pesticides chosen included both the most commonly used pesticides in peach and nectarine orchards as well as additional active compounds that were registered or non-registered. The stock solution containing multiple pesticides was generated by mixing them at a concentration of 10 μg mL^{-1} in acetonitrile. The standard mixtures were subsequently used for spiking in recovery experiments and for the preparation of matrix-matched calibration standards.

HPLC-grade acetonitrile (ACN) and water were acquired from PanReac AppliChem (ITW Reagents, Darmstadt, Germany), and methanol (MeOH) gradient grade for LC Lichrosolv® was from Supelco (Merck KGaA, Darmstadt, Germany). LC-MS grade formic acid (CH_2O_2) and analytical grade ammonium formate ($HCOONH_4$) were obtained from Sigma-Aldrich (Merck KGaA, Darmstadt, Germany), and glacial acetic acid (CH_3COOH) was from Isolab (Wertheim, Germany). The QuEChERS sample preparation kits were supplied by ChromaScience (Istanbul, Turkey). These kits included the salt extraction kit Association of Official Analytical Chemists (AOAC) 2007.01, which contained 6 g of magnesium sulfate ($MgSO_4$) and 1.5 g of sodium acetate (CH_3COONa), as well as the dispersive solid-phase extraction (d-SPE) kit, which included 1200 mg of $MgSO_4$ and 400 mg of primary secondary amine (PSA).

2.2. Samples

Between June 2021 and August 2023, 459 samples from the harvests of three consecutive years (2021, 2022, and 2023) were collected from two main production areas of Turkey, namely the Bursa and Izmir regions. The sampling consisted of 164 peach and 295 nectarine samples. The distribution of the collected samples per year is shown in Table 1.

The samples, weighing at least 1 kg each, were homogenized using a home food processor (Sefa Çelik, Beyoğlu, Istanbul, Turkey). During homogenization, only the entire edible portions of peaches and nectarines were used, excluding the stones and stems. The samples were homogenized at room temperature, forming a chemically active liquid/solid soup, and were immediately subjected to the QuEChERS sample preparation protocol to minimize pesticide degradation or volatilization. As specified in CAC (2011), residue levels were calculated based on the whole fruit, under the assumption that no residues were present in the stone.

Table 1
Summary of collected peach and nectarine samples.

Fruit	2021	2022	2023
Peach	42	54	68
Nectarine	28	154	113
Total	70	208	181

2.3. Sample preparation

Homogenized peach and nectarine samples were extracted using the AOAC 2007.01 QuEChERS procedure (Lehotay, 2007) as shown in Fig. 1. The technique consists of two stages: liquid micro-extraction and the d-SPE cleanup process. Analytes were extracted from the matrices (15 g of peach or nectarine) using ACN acidified with 1% CH_3COOH and salts/acetate buffer (6 g $MgSO_4$ and 1.5 g CH_3COONa) in the initial step. During the d-SPE clean-up phase, anhydrous $MgSO_4$ (1200 mg) and PSA (400 mg) were used to remove water and some interferences (carbohydrates, fatty acids, organic acids, lipids, and polar pigments), respectively, from the organic phase.

2.4. LC-MS/MS analysis

An LC-MS/MS system (Shimadzu LC-MS 8030 triple quadrupole-mass spectrometer) connected to a Shimadzu LC-20 series HPLC system was used to detect and measure 474 pesticide residues in peach and nectarine samples. The separation of multi-residue compounds was achieved by utilizing an SVEA Core C-18 reversed-phase column (2.6 μm, 3 mm × 100 mm) from Nanologica AB, Södertälje, Sweden. The chromatographic separation was achieved in the gradient elution mode specified in Table 2. A mobile phase consisting of eluents A (water-MeOH (98:2, v/v) containing 0.1% CH_2O_2 and 5 mM $HCOONH_4$) and B (MeOH-water (98:2, v/v) containing 0.1% CH_2O_2 and 5 mM $HCOONH_4$) was used at a flow rate of 0.6 mL min^{-1}. The column temperature was maintained at 40 °C. An injection volume of 10 μL was applied to the column.

Multiple reaction monitoring (MRM) mode was used to measure pesticide residues in positive or negative ionization modes (ESI). Two MRM transitions were measured for each residue (Table S1) as specified in the SANTE/11312/2021 Guidelines (European Commission, 2021). The spray voltage was set at 4.5 kV. Nitrogen was used as nebulizing and drying gas, with flow rates of 3.04 mL min^{-1} and 11 L min^{-1}. The heat block temperature was set at 400 °C, while the desolvation line temperature was maintained at 250 °C. The LabSolutions software, version 5.19, was used to control the instruments and collect data.

Table 2
Gradient conditions for LC.

Time (min)	Mobile phase A%[a]	Mobile phase B%[b]	Flow (mL min^{-1})
0.0	80	20	0.6
0.2	80	20	0.6
1.5	30	70	0.6
5.0	5	95	0.6
9.0	5	95	0.6
9.1	80	20	0.6
10.0	80	20	0.6

[a] Water–MeOH (98:2, v/v) with 0.1% CH_2O_2 and 5 mM $HCOONH_4$.
[b] MeOH–water (98:2, v/v) with 0.1% CH_2O_2 and 5 mM $HCOONH_4$.

The validation of the analytical method, conducted in accordance with the European SANTE/11312/2021 Guideline, was based on its linearity, accuracy, precision (repeatability and reproducibility), measurement uncertainty, and limit of quantification (LOQ), as previously described by Keklik et al. (2024). The assessment of linearity was conducted by analyzing matrix-matched calibration curves at five concentration levels (0.005–0.25 mg kg^{-1}). Both accuracy and precision were determined using fortified blank matrices at low and high concentration levels (0.01 and 0.05 mg kg^{-1}). The LOQs were determined as 10 times the standard deviation of replicate analyses of a blank sample matrix fortified with pesticide mix at a low fortification level.

2.5. Dietary exposure assessment

The assessment of dietary exposure to pesticides was conducted to evaluate the acute (short-term) and chronic (long-term) health risks for adults and children (aged 3–10 years) in Turkey. The estimation of chronic dietary exposure to pesticide residues in peaches and nectarines is achieved by multiplying the levels of residues by the data on fruit consumption, as shown in Equation (1).

$$Dietary\ exposure = \frac{Concentration\ of\ residue\ \left(\frac{mg}{kg}\right) x\ Fruit\ consumption (kg)}{Body\ weight\ (kg)} \quad (1)$$

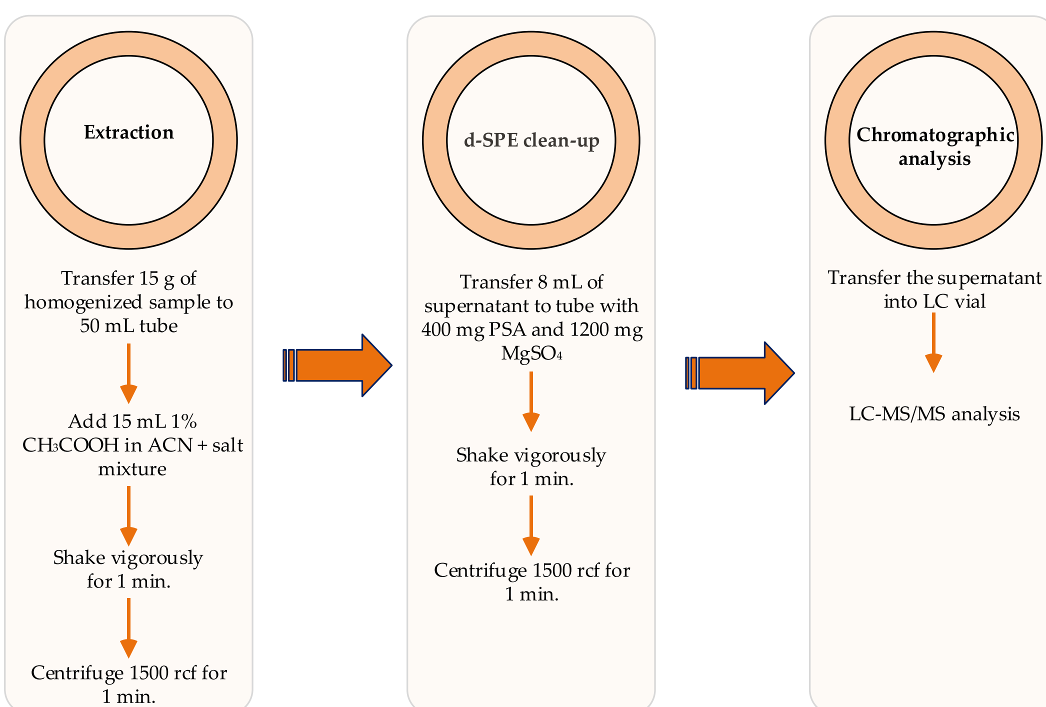

Fig. 1. The procedure of the QuEChERS sample preparation method.

Consumption patterns for the Turkish population were derived from the food balance sheets provided by the Turkish Statistical Institute, indicating an average consumption of 7.7 kg of peaches and nectarines per person per year, equivalent to 0.021 kg day^{-1} per capita (Turkish Statistical Institute (TUIK), 2023). Children's portion was calculated according to USEPA (2018) and was 40% of adult portion. The assessment considered both adult and child consumer groups, with recommended body weights of 70 kg for adults and 23 kg for children aged 3–10 years, as per European Food Safety Authority (EFSA) guidelines (EFSA, 2012).

At every stage of the exposure assessment process, there are inherent uncertainties in the estimate of dietary exposure to chemical hazards, including pesticide residues. Variations in food intake rates, individual body weights, sampling procedures, analytical biases and fluctuations, processing factors, and left-censored data are the primary sources of uncertainty (EFSA, 2007). The management and assessment of concentration data that falls below the limit of detection (LOD) or LOQ thresholds, also known as left-censored data, is a crucial factor that contributes to uncertainty. The substitution method, as described in the EFSA scientific report (EFSA, 2010), has been employed for left-censored data, where non-detectable results are substituted with zero, the LOQ value, and LOQ/2, corresponding to Lower, Upper, and Middle Bound (LB, UB, and MB) scenarios, respectively. Other sources of uncertainty were not considered in the study.

The Hazard Index (*HI*) method was used to assess the risks of cumulative pesticide residue exposure, which involves summing the Hazard Quotients (*HQ*c) for individual pesticide residues, following the approach outlined by Galani et al. (2020). The HQc for a specific pesticide is calculated by dividing the exposure to that residue by its acceptable daily intake (ADI) provided from EU pesticides database (European Commission, 2024). The calculations for HQc and HI are depicted in Equations (2) and (3), respectively.

$$\%\ Hazard\ Quotient\ (HQc) = \frac{Exposure\ of\ the\ concerned\ residue}{Reference\ value\ (ADI)}\ x\ 100 \quad (2)$$

$$Hazard\ Index\ (HI) = \Sigma\ HQ \quad (3)$$

The acute exposure risk *(%ARfD*) or acute hazard quotient (HQ_a) was calculated according to the following equations (WHO & FAO, 2014):

$$IESTI = \frac{Ue\ x\ HR\ x\ v + (LP - Ue)\ x\ HR}{bw} \quad (4)$$

$$HQa = \frac{IESTI}{ARfD} \quad (5)$$

$$\%ARfD = HQa \times 100 \quad (6)$$

where *IESTI* is the international estimation of short-term intake (mg kg^{-1} bw), *Ue* is the unit weight of edible portion (for a peach, *Ue* = 0.255 kg, and for a nectarine, *Ue* = 0.131 kg), *HR* is the highest residue in a composite sample of edible portion of the unit (mg kg^{-1}), *v* is the varibality factor ($v = 3$), *LP* is the large portion (For peaches, *LP* is 0.704 kg for the general population and 0.306 kg for children. For nectarines, the LP is 0.618 kg for the general population and 0.184 kg for children) (WHO, 2021), *bw* is the body weight (kg), and *ARfD* is the acute reference dose of pesticide (mg kg^{-1} bw), which were obtained from EU pesticides database (European Commission, 2024).

When *HQc*, *HQa,* or *HI* are higher than 1 (i.e. %HQc, %ARfD, or %HI are higher than 100%), there is a greater probability of adverse health effects due to the presence of pesticides.

3. Results and discussion

3.1. Pesticide residues in peaches and nectarines

Before the application of the LC-MS/MS method to real samples, the performance of the analytical method was assessed by several criteria as described in SANTE/11312/2021 Guideline. The total ion chromatogram of all the residues at 0.01 mg kg^{-1} in peach extract is shown in Fig. S1. The matrix-matched standard calibration showed good linearity for all residues over a concentration range of 0.005–0.25 mg kg^{-1}, with a coefficient of determination (R^2) >0.99 (Table S1 and Fig. S2). MRM chromatograms of matrix-matched calibration standards (0.005–0.25 mg kg^{-1}) for acetamiprid, boscalid, and thiophanate-methyl, along with the detected in real samples are illustrated in Fig. S3.

The LOQ, recovery, precision, and measurement uncertainty data for the target compounds are presented in Table S2. The method LOQs of all the analyzed residues were below 0.01 mg kg^{-1}. Acceptable recoveries (between 70% and 120%) and good precision values (lower than 20%) were obtained for all compounds. Quality control charts for individual recoveries is constructed based on the initial method validation data. Examples of control charts illustrating the results for acetamiprid, boscalid, and thiophanate-methyl over the initial 10 days of testing are presented in Fig. S4.

During the three harvesting seasons (2021–2023), a total of 459 samples, including 164 peaches and 295 nectarines, were analyzed for the presence of 474 different pesticides. The frequency of peach samples with or without residues per harvest year is shown in Fig. 2.

Pesticide residues were detected in 71.3% of the peach samples (117 samples), while residues of more than one pesticide were recorded in 49% of the samples analyzed throughout three consecutive years. 12.8% of the peach samples were determined to be non-compliant with European Union (EU) maximum residue limit (MRL) legislation. In peaches, 40 different residues were identified, 12 of which are currently not approved in the EU. The occurrence and concentration of residues in peaches are presented in Table S3. The most frequently identified residue in peaches was thiophanate-methyl (25.6%, 42 samples), followed by acetamiprid (23.2%, 38 samples), boscalid (17.1%, 28 samples), chlorantraniliprole (12.8%, 21 samples), and spirodiclofen (12.8%, 21 samples). The frequency of detection of residues in peaches is presented in Fig. 3.

In 2021, a relatively small number of peach samples (n = 42) were checked for the target compounds. In 57.1% of the peach samples, no pesticide residues could be quantified; 31% of the samples had residues below or at the MRL. Of the 42 peach samples, five (11.9%) contained residues above the MRL. All five individual determinations that exceeded the MRLs were reported for pesticides that are currently not approved in the EU. While 11 different residues were recorded in peach samples from 2021, the MRL was exceeded for only three pesticides: imidacloprid (two samples), phosmet (two samples), and triadimenol (one sample). Multiple residues were recorded in seven samples (16.7%); up to four different pesticides were detected in an individual peach sample. The most frequently found residues in peaches from 2021, which were quantified in more than 10% of the samples, were acetamipid (16.7%) and thiophanate-methyl (16.7%).

Compared to 2021 (42.9% incidence), the quantification rate of residues in peaches (87.0%) increased more than two-fold in 2022. In 13% of the peach samples, no quantifiable residues were detected. 70.4% of the 54 peach samples analyzed complied with the valid MRLs. The percentage of peach samples exceeding MRL (16.7%) in 2022 slightly increased as compared to 2021 (11.9%). These exceedances were related to residues of thiram (four samples), thiophanate-methyl (three samples), methoxyfenozide (two samples), phosmet (two samples), bitertanol (one sample), and imidacloprid (one sample). Among the 13 individual determinations that exceeded the MRL in 2022, 11 determinations were reported for pesticides that are currently non-approved.

In 2022, the number of residues identified in peaches was about three-fold higher than that of samples from the previous year. In 2022, 30 different residues were recorded in quantifiable concentrations in peaches; one third of them are currently not approved in the EU. The percentage of samples containing multiple residues in 2022 was 68.5%,

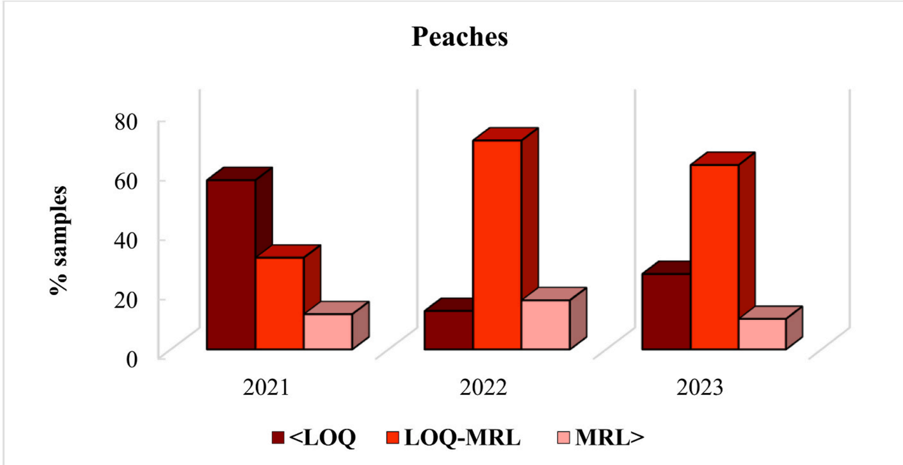

Fig. 2. The frequency of peach samples with or without residues per harvest year.

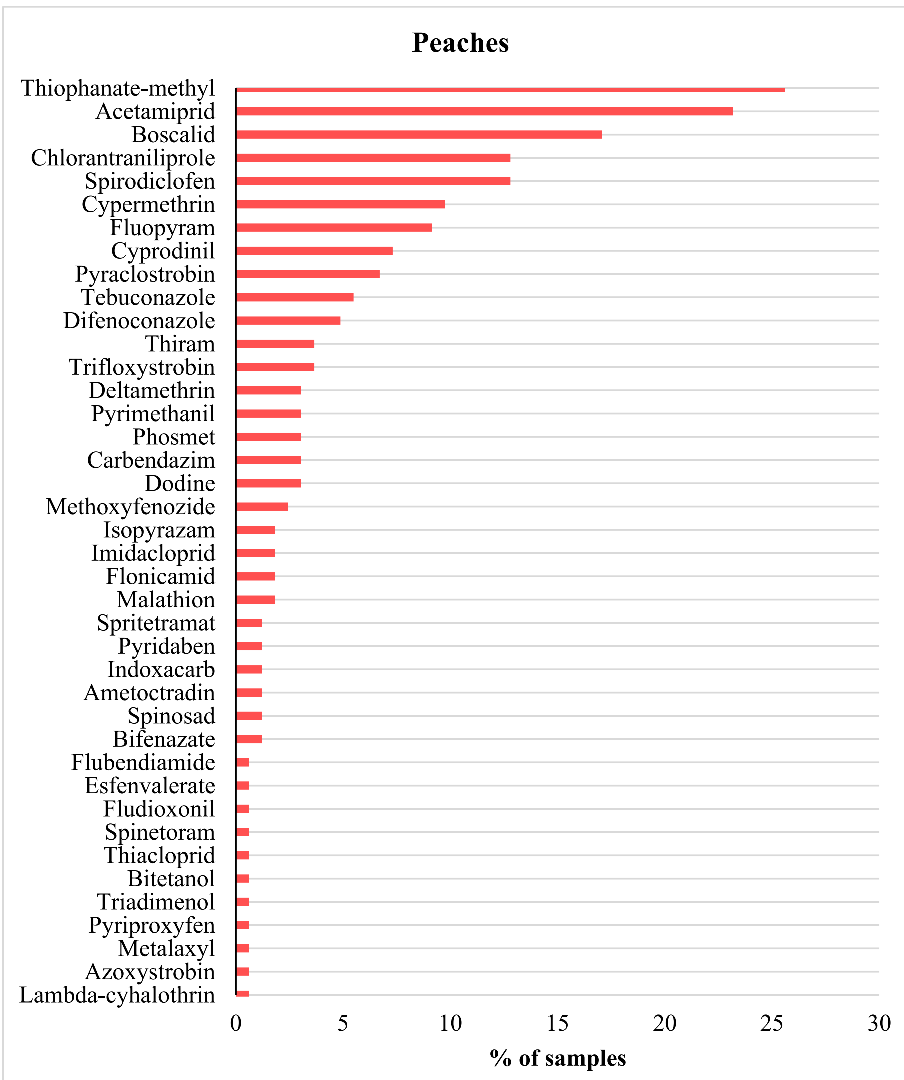

Fig. 3. Frequency of detected residues in peaches.

which is quite higher than in 2021 (16.7%). Only one compound (triadimenol), which was individually determined in one sample in 2021, was no longer identified in 2022. Like the previous year, the most frequent compounds recorded in peaches were acetamprid and thiophanate-methyl, with frequencies of 40.7% and 24.1%, respectively. Other of the most frequently identified pesticides measured in more than

10% of the samples analyzed were boscalid (22.2%), chlorantraniliprole (18.5%), cyprodinil (16.7%), and fluopyram (13.0%). Compared to 2021, the pesticide spectrum was comparable, with a higher quantification rate for acetamiprid (+24.0%), cyprodinil (+14.3%), thiophanate-methyl (+7.4%), difenoconazole (+5.0%), cypermethrin (+0.8%), and tebuconazole (+0.3%), and a lower quantification rate for imidacloprid (−2.9%), phosmet (−1.1%), and pyridaben (−0.5%).

In 2023, 68 peach samples were analyzed. 23.5% of the peach samples did not contain any pesticide residues. The percentage of peach samples containing at least one residue in 2023 was 76.5%, which is lower than in 2022 (87.0%), but a slightly higher than that of the year 2021 (42.9%). The rate of MRL exceedance in 2023 for peach fruit was 10.3% (seven samples). The MRL exceedances were related to seven residues: ametoctradin (two samples), methoxyfenozide (two samples), malathion (one sample), metalaxyl (one sample), phosmet (one sample), thiophanate-methyl (one sample), and thiram (one sample).

Among the 474 pesticides that were analyzed in peaches in 2023, 28 different pesticides were quantified; the most frequent pesticide residues quantified in more than 10% were thiophanate-methyl (32.4%), spirodiclofen (25%), boscalid (23.5%), chlorantraniliprole (16.2%), cypermethrin (16.2%), acetamiprid (13.2%), fluopyram (11.8%), and pyraclostrobin (10.3%). It should also be highlighted that six of the 28 residues detected in peaches are not approved in the EU. The percentage of peach samples with multiple residues was 54.4% in 2023, which is lower than in 2022 (68.5%), but higher than three-fold to the level in 2021 (16.7%). Of 37 samples of peach with multiple residues, 14 samples contained two residues, 5 samples contained three residues, 10 samples contained four residues, 4 samples contained five residues, 3 samples contained six residues, and one sample contained seven residues. While 11 residues identified sporadically in peaches in 2022 were no longer detected in 2023, nine pesticides, namely trifloxystrobin (six samples), pyrimethanil (five samples), ametoctradin (two samples), bifenozate (two samples), spinosad (two samples), azoxystrobin (one sample), lambda-cyhalothrin (one sample), metalaxyl (one sample), and pyriproxyfen (one sample) were recorded for the first time in 2023.

The frequency of nectarine samples with or without residues per harvest year is shown in Fig. 4. During the sampling period, the overall quantification rate of residues in nectarines (71.9%) was like that in peaches (71.3%). However, the MRL exceedance rates in nectarine samples (5.8%) were less than two-fold those recorded in peaches (12.8%). During the three years, 35 different residues were identified in nectarines, which is a bit less than those found in peaches. Out of the 35 residues, 13 are currently not approved in the EU. The occurrence and concentration of residues in nectarines are presented in Table S4. The percentage of nectarine samples that contained multiple residues was 37.6%, which is slightly lower than the level in peaches (49.4%). As in peaches, the three most frequently occurring residues in nectarines were thiophanate-methyl (28.1%), boscalid (25.4%), and acetamiprid (17.3%). The frequency of detection of residues in nectarines is presented in Fig. 5.

The lowest part of the nectarine samples analyzed belonged to the 2021 harvest season. In 11 samples of all 28 nectarines (39.3%), there is no detection of any residues. While 60.7% of the nectarine samples (17 peaches) had one or several pesticides, only one of them contained residue (imidacloprid) above MRL. Most of the positive samples had only one residue, while only three nectarine samples contained multiple residues. Of the eight different pesticides identified in nectarines in 2021, two residues, imidacloprid and thiophanate-methyl, are currently non-approved in the EU. Thiophanate-methyl (28.6%) and acetamiprid (25.0%) were among the two most frequently quantified compounds in nectarine samples in 2021.

In 2022, 154 nectarine samples were examined for target compounds. There were no detectable pesticide residues in 23.4% of the nectarine samples. Compared to 2021, an increase in the quantification rate was observed in the samples of nectarine in 2022. The number of samples with measurable residues above the LOQ but within the legally permitted level was 109 (70.8%). The MRL exceedance rate slightly increased from 3.6% in 2021 to 5.8% in 2022. Residues exceeding the MRL were related to five different residues: thiram (four samples), phosmet (two samples), ethirimol (one sample), malathion (one sample), and methoxyfenozide (one sample).

In 2022, peach samples had 29 distinct residues, with 12 of those being classified as non-approved. When compared to 2021, 22 new substances were included in the peach samples in 2022. Thiophanate-methyl (37%), boscalid (29.9%), acetamiprid (18.8%), pyrimethanil (11.7%), and chlorantraniliprole (10.4%) were among the most frequently recorded residues quantified in more than 10% of the peach samples. Compared to 2021, the pesticide spectrum was comparable, with a higher quantification rate for thiophanate-methyl (+8.4%) and boscalid (+26.3%) in 2022 and a lower quantification rate for acetamiprid (−6.2%).

The number of nectarine samples analyzed in 2023 was 113, of which 36 samples (31.9%) were free from target residues. Out of the 113 samples, 70 samples (61.9%) contained positive findings for any of the analyzed residues, but below the MRL. The findings for this reporting year represent a slight reversal compared to 2022 but are slightly higher than in 2021. The MRL was exceeded for seven samples (6.2%) in 2023 but was not observed for the same pesticides, except for thiram, in 2022. The MRL exceedances were noted for four residues: imidacloprid (two samples), phosmet (two samples), dodine (one sample), thiram (one sample), and tetraconazole (one sample).

In nectarines in 2023, 27 different substances were recorded in quantifiable concentrations. While eight residues (spirotetramat, clothinadin, dithianon, ethirimol, flutriafol, isopyrazam, methoxyfenozide, and thiacloprid) detected with less than 5% frequency in peaches in 2022 were no longer found in peaches, six new active substances

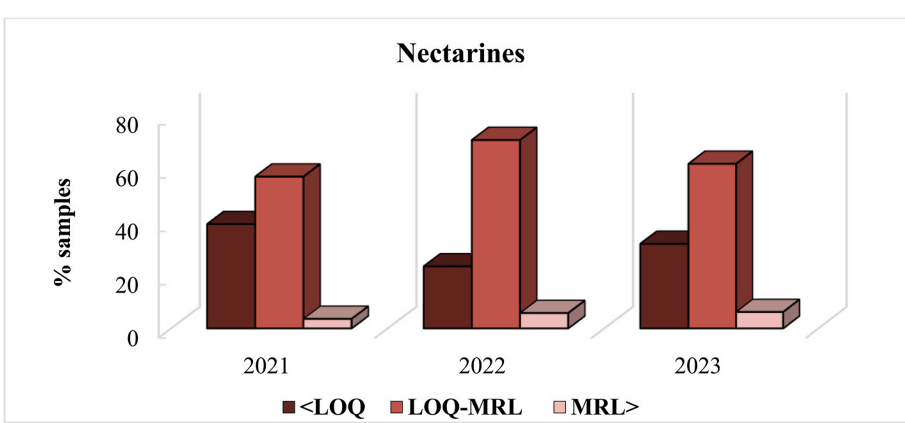

Fig. 4. The frequency of nectarine samples with or without residues per harvest year.

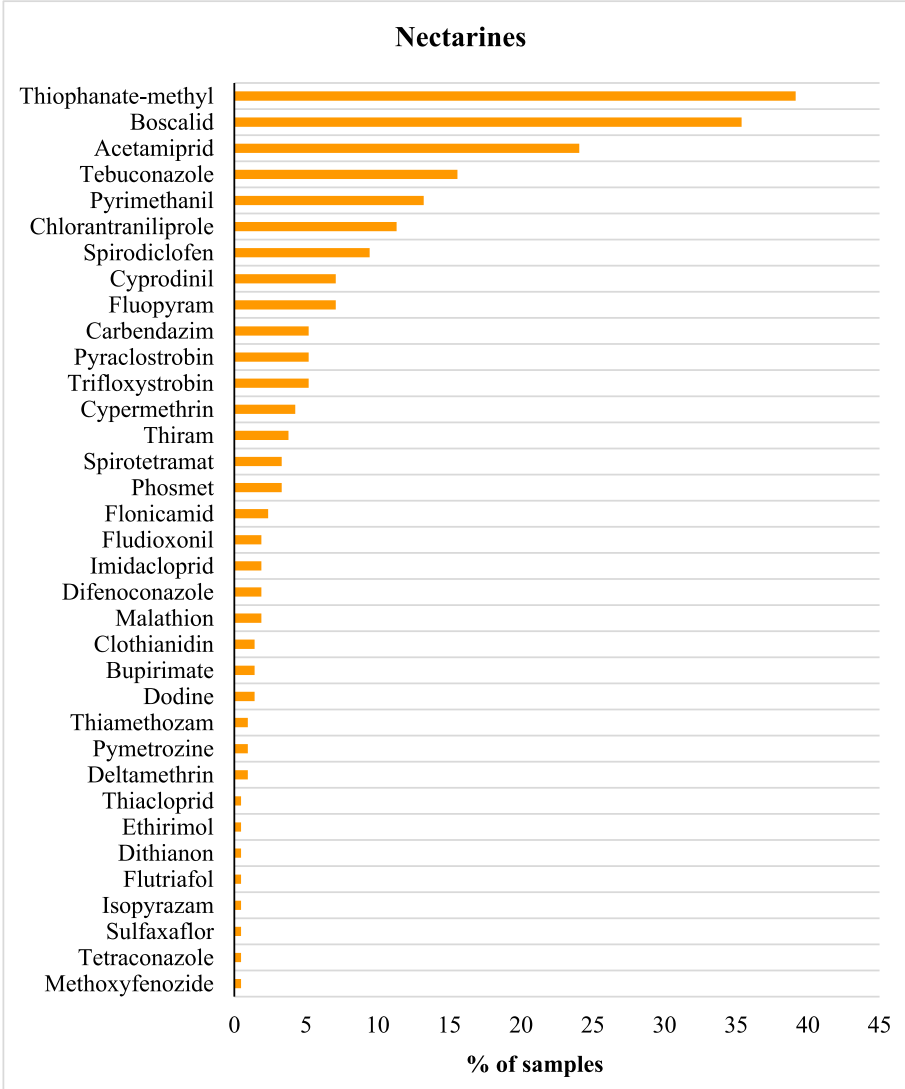

Fig. 5. Frequency of detected residues in nectarines.

(fludioxonil, dodine, pymetrozine, deltamethrin, sulfoxaflor, and tetraconazole) were identified in peaches in 2023. The rate of multiple residue incidence in peaches in 2023 (38.9%) was found to be a slightly lower than in 2022 (41.6%) but significantly higher than that of the year 2021 (10.7%). The most frequently found pesticides quantified in more than 10% of nectarines in 2023 were boscalid in 24.8%, tebuconazole in 17.7%, thiophanate-methyl in 15.9%, spirodiclofen in 14.2%, and acetamiprid in 13.3%. While the quantification rate has increased in 2023 for spirodiclofen (+11.6%) and tebuconazole (+10.6%), a decline in the quantification rate was observed for thiophanate-methyl (−21.1%), acetamiprid (−5.6%), and boscalid (−5.1%).

Each pesticide identified in the samples exhibits unique toxicological characteristics and poses possible health hazards. Thiophanate-methyl, the most frequently identified pesticide both in peaches and nectarines, belongs to the benzimidazole class of fungicides. The presence of thiophanate-methyl in peaches and nectarines is largely due to its use as a pre-harvest fungicide against various fungal pathogens, such as *Monilinia laxa* and *Monilinia fructicola*. It is a broad-spectrum systemic fungicide with both protective and curative properties that is absorbed by the roots and leaves. It effectively combats a wide range of fungal infections, including *Monilinia fructicola* and *Monilinia laxa* in a variety of crops (Gura et al., 2023). Systemic fungicides belonging to the methyl benzimidazole carbamate (MBC) family include benomyl, carbendazim, and thiophanate-methyl, which are closely related (Fungicide Resistance Action Committee (FRAC), 2024). Thiophanate methyl is classified as mutagenic category 2 and has been proposed for categorization as a carcinogen category 2 (EFSA, 2024), based on dose-dependent increases in liver tumors in male and female mice (EPA, 2004). The conversion of thiophanate-methyl to carbendazim, an intermediate metabolite, has been shown in apples, grapes, and beans. Specifically, after applying thiophanate-methyl to the leaves of stone fruits, the primary chemicals found were parent and carbendazim. These compounds accounted for up to 64.5% and 22.2%, and 44.5% and 33.4% of the total radioactive residue (TRR) on the 1st and 7th day samples, respectively (FAO, 1999). In the present study, the detection rates of thiophanate-methyl and carbendazim were 25.6% and 3.0% in peaches and 39.2% and 5.2% in nectarines, respectively. These results showed that thiophanate-methyl and carbendazim, banned in the EU, are still in use as plant protection products in Turkey.

Boscalid, the second most frequently found fungicide, is a carboxamide chemical that successfully inhibits a wide range of fungal infections in a variety of agricultural products such as stone fruits, fruiting vegetables, cucurbit fruits, and tree nuts (EFSA, 2014). The presence of boscalid in peaches and nectarines is largely due to its use as a pre-harvest fungicide against powdery mildew disease caused by *Sphaerotheca pannosa*. While no reproductive, developmental, immunotoxic, or neurotoxic concerns have been identified for boscalid, the liver and thyroid are the main targets in mammals. Boscalid has been observed to induce reduced body weights and elicit liver and thyroid effects in rats and dogs, as evidenced by subchronic and chronic feeding investigations (Environmental Protection Agency EPA, 2019).

One of the other most frequently approved pesticides, acetamiprid, belongs to the neonicotinoid class and is used for the protection of fruits and vegetables from insecticides, including Hemiptera, Lepidoptera, Thysanoptera, and Coleoptera (EFSA, 2018). The prevalence of acetamiprid in peaches and nectarines is primarily owing to its use as a pre-harvest insecticide against the green peach aphid, *Myzus persicae*. Acetamiprid exhibits relatively low acute and chronic toxicity in mammals, with no evidence of carcinogenicity, neurotoxicity, mutagenicity, or endocrine disruption (EFSA, 2022).

The present results are comparable to those of previous findings, in which a limited number of both samples and residues were monitored. In a previous study, ten peach and three nectarine samples collected from different bazaars and markets in Konya province, Turkey, were analyzed for 203 pesticides. A half of the peaches contained at least one residue; chlorpyrifos (0.006–0.929 mg kg^{-1}) was detected in three samples, while acetamiprid (0.020 mg kg^{-1}), amitraz (0.019 mg kg^{-1}), carbaryl (0.006 mg kg^{-1}), carbendazim (0.014 mg kg^{-1}), phosalone (0.121 mg kg^{-1}), imidacloprid (0.027 mg kg^{-1}), and omethoate (0.044 mg kg$^{-1)}$ were identified in one individual sample. All three nectarine samples were positive for residues; in two samples, chlorpyrifos (0.005–0.011 mg kg^{-1}) was found, while one individual sample contained acetamiprid (0.006 mg kg^{-1}) and carbendazim (0.006 mg kg^{-1}) (Ersoy et al., 2011).

In another study, peach and nectarine samples were collected from five different stands in a bazaar in Canakkale, Turkey, for 12 weeks during the period of June–September 2020 and analyzed only for three residues: chlorpyrifos, boscalid, and tebuconazole. While none of the peach and nectarine samples contained quantifiable concentrations of chlorpyrifos, which was banned in May 2020 in Turkey, boscalid and tebuconazole were recorded up to levels of 0.57 and 0.48 mg kg^{-1} in peaches and 0.32 and 0.57 mg kg^{-1} in nectarines (Dülger & Tiryaki, 2021). In another study carried out two years later, nectarines cultivar "Bayramiç beyazı" (210 analytical determinations) were collected from the bazaar in Canakkale, Turkey, during June–September 2022 and analyzed for the presence of 377 pesticides. Among the target analytes, ten residues, namely boscalid, bupirimate, carbendazim, cyflufenamid, difenoconazole, flusilazole, penconazole, tetraconazole, thiophanate-methyl, and triadimenol, were found at measurable concentrations but below the MRLs except for flusilazole and triadimenol (Serbes & Tiryaki, 2023). In a recent study conducted by Kanbolat et al. (2023), pesticide residues were detected in eight out of nine peach samples collected from orchards in the Tokat region of Turkey. Of these, pesticide levels exceeding the MRLs were identified in three samples. A total of nine distinct pesticides were found in the peach samples, with thiophanate-methyl (44.4%, 0.040–0.276 mg kg^{-1}) and tebuconazole (44.4%, 0.014–0.031 mg kg^{-1}) being the most frequently detected residues.

In China, 312 peach samples collected during the years 2013–2015 were analyzed for 105 pesticides. In peach samples, 39 different residues, including 21 insecticides, 13 fungicides, and 5 acaricides, were recorded in 92.3% of the samples. While 20% of samples contained only one residue, 76.3% of peaches had multiple residues. An individual peach sample contained up to 13 different pesticides. The residues carbendazim (60.6%), cyhalothrin (30.8%), chlorpyrifos (29.2%), acetamiprid (28.2%), cypermethrin (22.4%), imidacloprid (21.2%), difenoconazole (13.5%), tebuconazole (13.5%), and pyridaben (10.6%) were found more frequently among the 39 detected pesticides (Li et al., 2018). In a Hungarian survey from 2017 to 2021, 468 samples of peaches and nectarines were monitored for 445 active substances. While 26.3% of samples were free from target pesticides, 350 samples (78.7%) contained residues (up to nine different pesticides in individual samples) at measurable concentrations. Only one sample contained residue above the MRL (Ambrus et al., 2023).

In a Danish monitoring study conducted in the periods 2004–2011, 342 peach and nectarine samples imported mainly from Spain, Italy, and Chile were tested for pesticides. 69% of peach samples had one or more residues, and only 1% of samples exceeded the MRL. In the peach samples, 50 different residues were recorded (Petersen et al., 2013).

In the scientific report of the EFSA on pesticide residues in food in 2015, no quantifiable residues were found in 367 (24.7%) out of 1484 peach samples. Multiple residues were detected in 54.9% of peach samples, whereas the remaining 302 samples (20.4%) contained only one residue (EFSA, 2017).

In a field study in Greece, peaches were collected at different times during the vegetative period after the pre-harvest treatment of six residues (chlorothalonil, iprodione, bupirimate, pirimicarb, chlorpyrifos and fenoxycarb) and analyzed for the target analytes. The concentrations of bumipirimate, iprodione, fenoxycarb, chlorpyrifos, and pirimicarb in peach samples were below the EU MRLs at 33, 22, 22, 28, and 63 days, respectively, after their application, while chloranthalonil was not detected at measurable concentrations (Chatzicharisis et al., 2012).

The present study's findings are consistent with prior research, although the broader range of pesticides screened in this study, including banned compounds, highlights the ongoing presence of unauthorized pesticide use in Turkish peach and nectarine production. The frequent detection of thiophanate-methyl and boscalid, as well as the occurrence of acetamiprid and various other compounds, underscores the need for stricter regulatory measures to monitor and mitigate pesticide residues in fruit crops.

3.2. Multivariate analysis

The correlation matrix (0.05 levels of significance) of detected pesticide residues is shown in Table S5. A strong positive correlation is observed between spinosad and bifenezate (r = 1), trifloxystrobin and fluopyram (r = 0.798), pyraclostrobin and boscalid (r = 0.626), pyrimethanil and dodine (r = 0.625), metalaxyl and ametoctradin (r = 0.623), tebuconazole and spirodiclofen (r = 0.604), methoxyfenozide and isopyrazam (r = 0.597), and thiamethozam and carbendazim (r = 0.576), suggesting a common origin. A moderate positive correlation existed between thilacloprid and ethirimol (r = 0.478), malathion and lambda-cyhalothrin (r = 0.460), ethirimol and bupirimate (r = 0.438), esfenvelarate and difenoconazole (r = 0.435), lambda-cyhalothrin and fludioxonil (r = 0.384), thiamethozam and indoxacarb (r = 0.383), imidacloprid and fludioxonil (r = 0.368), pyrimethanil and cypermethrin (r = 0.347), thiamethozam and clothinadin (r = 0.342), ethirimol and acetamiprid (r = 0.339), tiophanate-methyl and clothinadin (r = 0.335), cypermethrin and bifenazate (r = 0.335), pyrimetrozine and carbendazim (r = 0.334), thiamethozam and thiacloprid (r = 0.325), spirotetramat and isopyrazam (r = 0.324), pyridaben and deltamethrin (r = 0.316), and flubendiamide and deltamethrin (r = 0.310). Correlations between other pesticide residues were insignificant, and in most cases, it was negative.

A principle component analysis (PCA) was used to visualize the dataset for the detected pesticides. The PCA of the measured concentrations of the 49 pesticides in peaches and nectarines gives three principle compounds with eigenvalues >1. The contribution rates of PC1, PC2 and PC3 were 94.8%, 1.6% and 1.1%, respectively. The cumulative contribution of the first three principal components was 97.5%. The 3D scatter plot graph (Fig. 6) demonstrates the segregation of certain pesticides into four different groups. The largest group included 42 pesticides. The second group included acetamiprid, pyrimethanil, azoxystrobin, and boscalid. The third group included thiamethoxam and carbendazim. The fourth group includes only thiram.

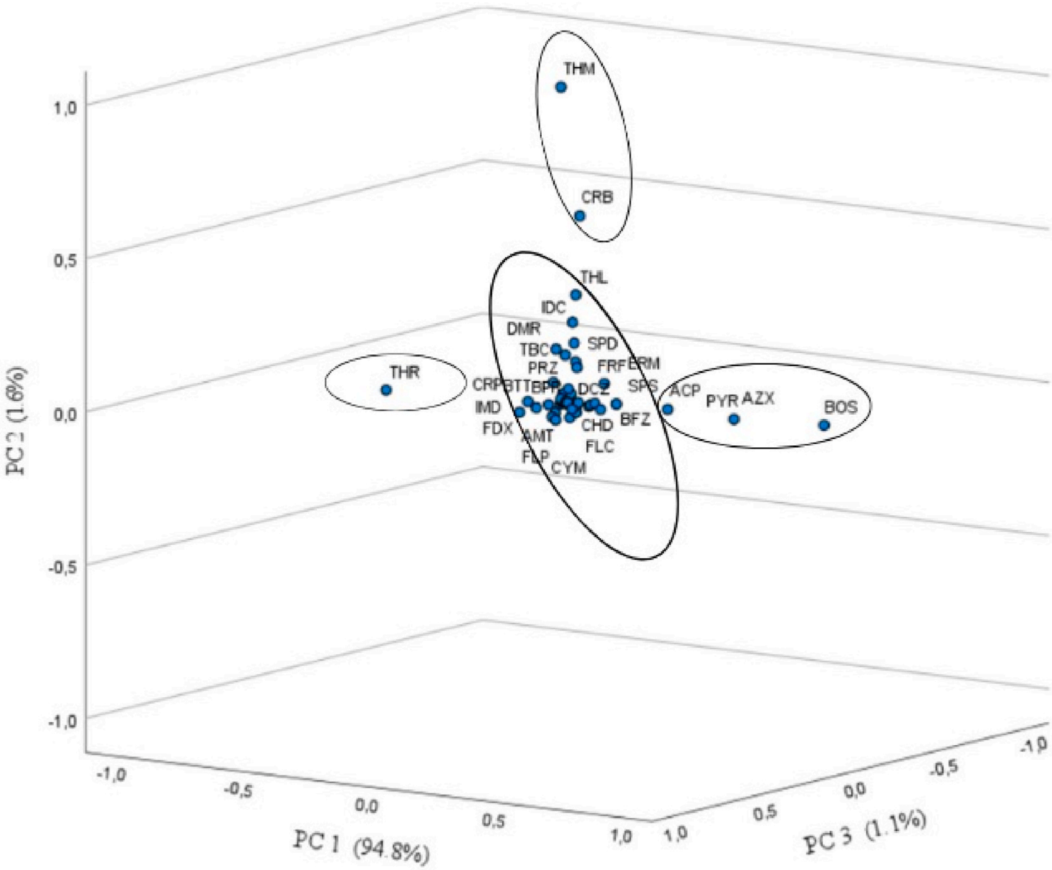

Fig. 6. Principal component analysis (PCA) of 49 pesticides detected in peach and nectarine samples.

3.3. Risk assessment

The analysis of peach and nectarine samples revealed that, out of the 474 pesticides tested, residues for 49 different pesticides were detected, while the remaining 425 pesticides were below detectable levels. As a result, it can be concluded that exposure to these residues is negligible. The estimated chronic dietary exposure and *HQ* of 49 pesticides found in peaches and nectarines for both adults and children aged 3–10 years old are provided in Table S6. The chronic exposure levels of individual pesticides for adults and children varied between 3.1×10^{-7} and 2.3×10^{-5} mg kg^{-1} b.w. day^{-1}, and between 3.8×10^{-7} and 2.8×10^{-5} mg kg^{-1} b.w. day^{-1}, respectively at the MB. Among the residues, thiophanate-methyl exhibited the highest contribution (33.6%) to cumulative exposure in both adult and child populations, followed by boscalid (6.2%), acetamiprid (4.0%), and chlorantraniliprole (3.1%). The cumulative exposures to detected pesticides for adults and children were calculated to be 6.9×10^{-5} μg kg^{-1} b.w. day^{-1} and 8.4×10^{-5} μg kg^{-1} b.w. day^{-1} at MB, respectively.

The *HQs* of individual residues for adults and children under the worst-case scenario (UB) exhibited a range of 0.00002%–0.18399% and 0.00002%–0.223984%, respectively. The children demonstrated slightly higher *HQs* for each residue in comparison to the adult consumer group, primarily because of their increased exposure per kilogram of body weight. It is important to note that there are significant variations in the ranking of residues based on their exposure or *HQ*, which can be attributed to differences in their ADI values. Thiophanate-methyl is identified not only as the main source of exposure from peaches and nectarines but also has the highest *HQ* for both adults and children at the MB.

The *HI* values of 0.49% for adults and 0.60% for children at MB suggest that there is no significant health risk for the Turkish population due to the cumulative exposure to detected pesticides through the consumption of peaches and nectarines. Fig. 7 depicts the proportional impact of pesticide residues on the *HI* at the MB. The most significant residues contributing to the HI at MB were identified as thiophanate-methyl (24%), phosmet (23%), lambda-cyhalothrin (7%), bitertanol (5%), and cypermethrin (4%).

One limitation of this study is the use of a deterministic approach for risk assessment. This method, while valuable for initial risk estimates, does not account for the variability and uncertainty in real-world exposure scenarios. Deterministic models use fixed parameters that may not fully capture differences in consumer behaviors, dietary intake, or the cumulative effects of multiple pesticides. To enhance accuracy, future studies could consider probabilistic methods, such as Monte Carlo simulations, which would better account for these factors and provide more comprehensive risk evaluations.

The values presented in this study are slightly lower than those reported by Petersen et al. (2013), who observed that peaches and nectarines contributed to the *HI* by 0.58% for adults. In another study, the health risk assessment for the quantified residues in peaches was determined for adults and children in China. Among the residues, the highest HQ was obtained for chlorpyrifos (0.13% for adults and 0.42% for children), followed by triazophos, carbendazim, and isazofos. The *HI* values of 0.40% and 1.3% at MB for the general population and children in China, respectively, are slightly lower than those obtained in our study (Li et al., 2018). In another study, only nectarines, which contain ten residues, contributed to the HI by 0.38% for the general population in Turkey, with flusilazole having the highest HQ (0.17%) (Serbes & Tiryaki, 2023).

The *HQa* values for each pesticide, calculated based on the highest measured concentrations, are presented in Fig. 8. Acute exposure risk assessments were not evaluated for 11 residues, ametoctradin,

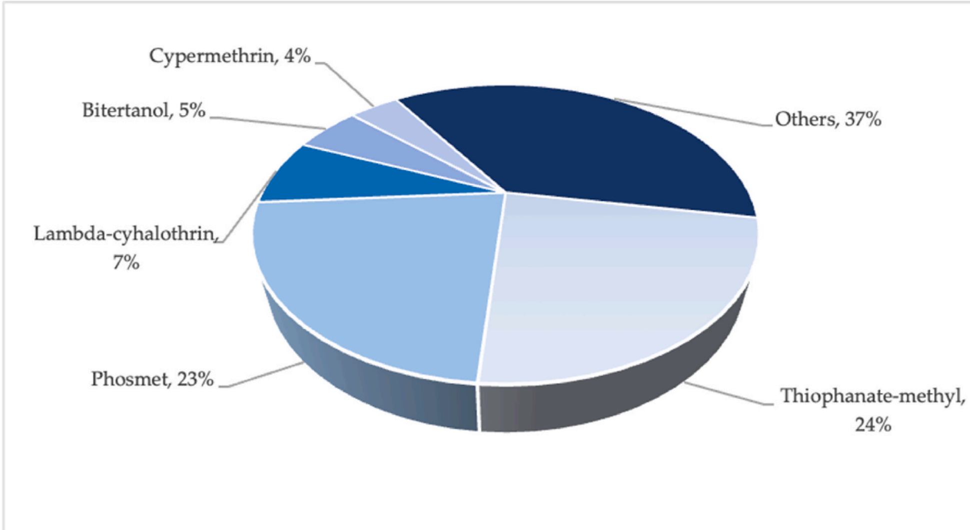

Fig. 7. Contribution of detected residues to HI for peaches and nectarines at MB.

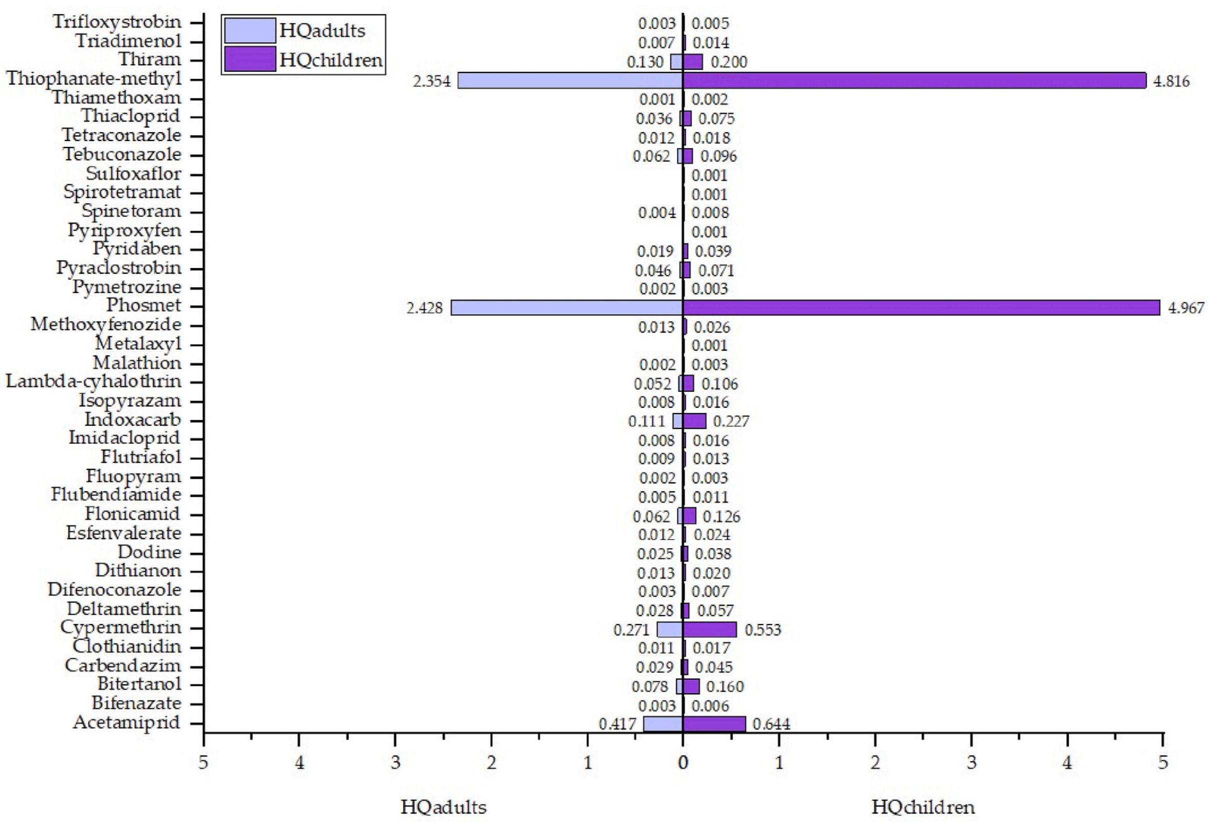

Fig. 8. *HQa* values of pesticides for the peach and nectarine consumers.

azoxystrobin, boscalid, bupirimate, chlorantraniliprole, cyprodinil, ethirimol, fludioxonil, pyrimethanil, spinosad, and spirodiclofen, as their *ARfDs* were not available (European Commission, 2024). The values for acute health risk ranged from 0.0003 to 2.428 for adults and 0.0005 to 4.967 for children. The highest exposure values were found for phosmet (HQ_{adults} = 2.428, $HQ_{children}$ = 4.967), followed by thiophanate-methyl (HQ_{adults} = 2.354, $HQ_{children}$ = 4.816), and acetamiprid (HQ_{adults} = 0.417, $HQ_{children}$ = 0.644). The exposure values above 1, obtained for 10 samples for adults and children, suggested that exposure risks for the phosmet and thiophanate-methyl are unacceptable in the short term. The acute risks of phosmet and thiophanate-methyl were unacceptable in 0.4% and 1.7% of samples for adults and 0.9% and 1.3% of samples for children, respectively. While considering the mean exposure values for acute health risks, it can be concluded that the detected residues do not pose a risk in the short term ($HQa < 1$).

4. Conclusions

This study aimed to assess pesticide residues in peaches and nectarines consumed in Turkey, as well as evaluate the potential health risks to adult and child Turkish consumers associated with the detected residues. The method of analysis, which involves the QuEChERS sample preparation method followed by LC-MS/MS analysis, has been successfully validated for the detection and precise quantification of 474 different residues in peach and nectarine samples. Over three years, 71.3% of 164 peach and 71.9% of 295 nectarine samples contained pesticide residues, with multiple residues present in 49.4% and 37.6% of peach and nectarine samples, respectively. Residue levels exceeded MRLs in 12.8% of peach and 5.8% of nectarine samples, involving ten pesticides in peaches and eight pesticides in nectarines. Peach samples contained 40 different residues; 12 of them are currently not approved in the EU, while for nectarines, non-approved pesticides were more frequent (13 out of 35 residues). Thiophanate-methyl (25.6% in peaches and 28.1% in nectarines), acetamiprid (23.2% in peaches and 17.3% in nectarines), and boscalid (17.1% in peaches and 25.4% in nectarines) were the most detected residues both in peaches and nectarines. Although the detectability rate was high and multiple residues were found in both peaches and nectarines, the *HI* of 0.49% for adults and 0.60% for children does not suggest any potential adverse effects from cumulative exposure to the detected residues. The findings offer valuable insights into the current levels of pesticide contamination in peaches and nectarines consumed in Turkey and the chronic dietary risks they pose to adults and children. To address the issue of non-approved pesticides and excessive use, it is essential for the Ministry of Agriculture and Forestry of Turkey to implement further measures. One potential solution could be the introduction of extension training programs for fruit growers. The initiatives should involve developing and distributing field guides and booklets that offer precise instructions on the correct application of pesticides, accurate pest identification, and effective methods to reduce pesticide residue buildup during fruit cultivation.

CRediT authorship contribution statement

Mehmet Keklik: Validation, Software, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ozgur Golge:** Validation, Resources, Methodology, Investigation. **Miguel Ángel González-Curbelo:** Writing – review & editing, Writing – original draft, Funding acquisition, Conceptualization. **Bulent Kabak:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Methodology, Investigation, Funding acquisition, Conceptualization.

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Declaration of competing interest

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

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodcont.2025.111141.

Data availability

Data will be made available on request.

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