

Ecochemical Assessment of Synthetic Pesticide Residues in a Tomato Sample Grown in Azerbaijan: Analysis of Plant Extract-Based Biopesticides as a Promising Alternative

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Abstract. Synthetic pesticides are widely used in modern agriculture to increase productivity and protect crops from pests, pathogens, and weeds, but their intensive and long-term application may result in pesticide residues in agricultural products and potential risks to human health and the environment. This study aimed to determine pesticide residues in a tomato sample grown in Azerbaijan using the QuEChERS–LC–MS/MS method, assess the analytical result from food-safety and ecochemical perspectives, and examine plant-extract-based biopesticides as potential alternatives to synthetic pesticides. The analytical procedure included sample preparation, citrate-buffered QuEChERS extraction, dispersive solid-phase clean-up, and LC–MS/MS determination using a Shimadzu LCMS-8045 Triple Quadrupole system with a Raptor Biphenyl column. Buprofezin was detected in the tomato sample at a concentration of 0.12 mg/kg. This value was compared with the applicable maximum residue level (MRL) for the intended European market, and the result was classified as non-compliant according to the source data. The findings demonstrate the importance of regular pesticide-residue monitoring and risk-based assessment of agricultural products. The literature review further indicates that plant-extract-based biopesticides may contribute to more sustainable crop-protection strategies, although their efficacy, stability, toxicological and ecotoxicological properties require case-specific evaluation.

Keywords: pesticide residues, tomato, ecochemistry, QuEChERS, LC–MS/MS, food safety, biopesticides

Introduction

Synthetic pesticides are widely used to increase agricultural productivity and protect crops from pests, diseases, and weeds (Rani et al., 2021; Tudi et al., 2021). Although these substances play an important role in reducing crop losses, their intensive and long-term application may result in residues in soil, water, and agricultural products, biodiversity loss, and potential risks to human health (Rani et al., 2021; Tudi et al., 2021; Carvalho, 2017). The entry of pesticide residues into the food chain may pose potential risks to human health and ecosystems (Nicolopoulou-Stamati et al., 2016; Kim et al., 2017; Carvalho, 2017). Therefore, monitoring pesticide residues in agricultural products using modern analytical methods is considered one of the key directions of food safety and environmental protection (European Food Safety Authority et al., 2023; Alder et al., 2006).

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Vegetables play an important role in human nutrition; therefore, their assessment with respect to pesticide residues is of particular importance (Carvalho, 2017). The establishment of maximum residue levels (Maximum Residue Levels – MRLs) and their regular monitoring are key elements of international food safety approaches to the assessment of pesticide residues in food products (EFSA et al., 2023; European Parliament and Council, 2005).

The QuEChERS extraction method and liquid chromatography–tandem mass spectrometry (LC–MS/MS) are widely used analytical approaches for the determination of pesticide residues across multiple active substances (Alder et al., 2006; Lehotay et al., 2010; Wilkowska & Biziuk, 2011). The high selectivity and sensitivity of these methods provide suitable opportunities for monitoring pesticide residues in various food matrices (Alder et al., 2006). In recent years, interest in biopesticides based on plant extracts has increased with the aim of reducing the potential environmental impacts of synthetic pesticides (Isman, 2020; Damalas & Koutroubas, 2018). Because these preparations are based on naturally derived active substances, they are considered promising alternatives to synthetic pesticides. Nevertheless, their efficacy, biodegradation, and effects on non-target organisms should be evaluated separately depending on the properties of the active components and the conditions of application (Isman, 2020; Pavela & Benelli, 2016; Isman & Grieneisen, 2014).

Research Objective

The objective of this study was to determine synthetic pesticide residues in a tomato sample grown in Azerbaijan using the QuEChERS–LC–MS/MS method, to assess the obtained result from food safety and ecochemical perspectives, and to substantiate, based on the scientific literature, the prospects for applying biopesticides based on plant extracts as potential alternatives to synthetic pesticides.

Scientific Novelty

The scientific novelty of this study lies in linking the analytical determination of synthetic pesticide residues in tomatoes grown in Azerbaijan using the QuEChERS–LC–MS/MS method with ecochemical assessment, and in analyzing, based on the scientific literature, biopesticides based on plant extracts as potential alternatives to synthetic pesticides. The general conceptual framework of the study is presented in Figure 1.

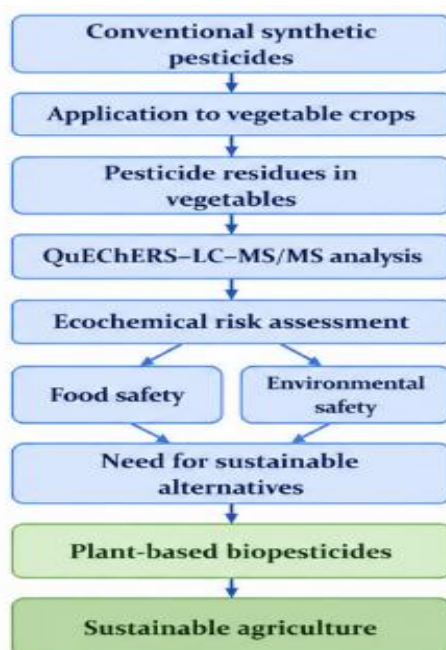


Figure 1
General conceptual framework of the study

Materials and Methods

General Study Design

The study was conducted to determine synthetic pesticide residues in a tomato sample grown in Azerbaijan and to assess them from an ecochemical perspective. The analytical process consisted of sample collection, sample preparation, extraction using the QuEChERS method, dispersive solid-phase extraction (d-SPE) clean-up, analysis using an LC–MS/MS system, and evaluation of the results against the relevant regulatory requirements.

The analytical methodology was applied in accordance with the European standard EN 15662:2015 and relevant quality control and method validation requirements for the analytical determination of pesticide residues (European Committee for Standardization, 2015; OECD, 2017). The general workflow of the analytical procedure is presented in Figure 2.

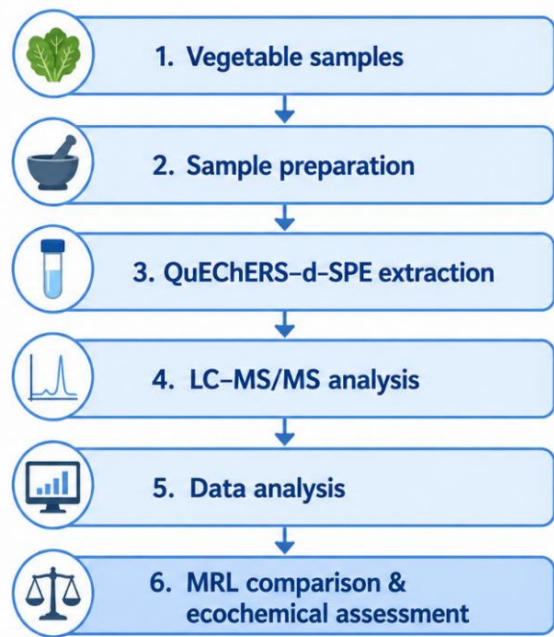


Figure 2
General workflow of the analytical procedure used in the study

Study Objects

Tomato (*Solanum lycopersicum* L.) samples grown in Azerbaijan were selected as the study objects. After being delivered to the laboratory, the samples were homogenized and stored under refrigerated conditions until analysis.

Equipment and Reagents Used

A Shimadzu LCMS-8045 Triple Quadrupole LC–MS/MS system, Raptor Biphenyl analytical column, refrigerated centrifuge, vortex mixer, analytical balance, and homogenizer were used for the analysis of pesticide residues (Table 1).

Table 1
Main laboratory equipment

Equipment	Specification
LC–MS/MS system	Shimadzu LCMS-8045 Triple Quadrupole
Analytical column	Raptor Biphenyl (100 × 3.0 mm, 2.7 μm)
Centrifuge	Refrigerated centrifuge

Vortex mixer	Laboratory vortex
Analytical balance	± 0.0001 g
Homogenizer	Laboratory blender

Only solvents and analytical reagents of a purity grade suitable for LC–MS analysis were used during the analysis. The main reagents used are presented in Table 2.

Table 2
Chemicals and reagents

Reagent	Purpose
Acetonitrile	Extraction solvent
Methanol	Mobile phase
Formic acid	Mobile phase modifier
Ammonium formate	Buffer
Magnesium sulfate (MgSO ₄)	Water removal
Sodium chloride (NaCl)	Phase separation
Trisodium citrate	Buffering salt
Disodium hydrogen citrate	Buffering salt
PSA sorbent	Matrix clean-up
C18 sorbent	Removal of lipids
Ultrapure water	Mobile phase preparation

Sample Preparation

A 10.0 ± 0.1 g portion of the homogenized sample was placed in a 50 mL polypropylene centrifuge tube, and 10 mL of acetonitrile of a purity grade suitable for LC–MS/MS analysis was added. The sample was homogenized until a uniform mixture was obtained; for low-moisture matrices, ultrapure water was added to improve extraction efficiency.

QuEChERS Extraction

Extraction was performed using the citrate-buffered QuEChERS method in accordance with EN 15662:2015 (Lehotay et al., 2010; Wilkowska & Biziuk, 2011; CEN, 2015; Anastassiades et al., 2003). Magnesium sulfate, sodium chloride, trisodium citrate dihydrate, and disodium hydrogen citrate sesquihydrate were added to the extraction mixture. After intensive vortex mixing, the mixture was centrifuged, and the upper acetonitrile phase was transferred to kits containing PSA, MgSO₄, and C18 sorbents for dispersive solid-phase extraction (d-SPE) clean-up. The purified extract was centrifuged again and prepared for LC–MS/MS analysis.

QuEChERS Extraction Procedure

10 g sample → 10 mL Acetonitrile → Extraction salts → Vortex → Centrifugation → Organic phase → PSA + MgSO₄ + C18 → Vortex → Centrifugation → LC vial for analysis → LC–MS/MS

LC–MS/MS Analysis

Identification and quantitative determination of pesticide residues were performed using a Shimadzu LCMS-8045 Triple Quadrupole LC–MS/MS system. Chromatographic separation was carried out on a Raptor Biphenyl column in gradient elution mode. Detection was performed using multiple reaction monitoring (MRM) in ESI (+) and ESI (–) ionization modes.

Analytes were identified based on retention time, precursor ion, product ion, and MRM transitions (Alder et al., 2006). The main operating parameters of the LC–MS/MS system are presented in Table 3.

Table 3
LC–MS/MS operating conditions

Parameter	Value
Instrument	Shimadzu LCMS-8045
Column	Raptor Biphenyl
Flow rate	0.45 mL/min
Column temperature	50 °C
Injection volume	5 µL
Ionization	ESI (+/–)
Acquisition mode	MRM

Regulatory Assessment of Results

The analytical results were processed using the instrument software. The detected buprofezin residue level was assessed based on the relevant regulatory requirements, taking into account the intended export destination of the product. For products intended for export to the European market, the maximum residue level (MRL) established in the European Union was used as the reference (Nicolopoulou-Stamati et al., 2016).

The detected buprofezin concentration was compared with the maximum residue level (MRL) specified in the relevant regulatory document, and the result was assessed in terms of food safety, regulatory compliance, and ecochemical risks (Nicolopoulou-Stamati et al., 2016; Kim et al., 2017). The result is presented in Table 3.

Results and Discussion

Analytical Assessment of Conventional Pesticide Residues

The analyzed tomato sample was assessed using the EN 15662:2015 method. Buprofezin was detected in the tomato sample. The detected buprofezin residue concentration was evaluated by comparison with the maximum residue level (MRL) established in accordance with the relevant regulatory requirement (European Parliament and Council, 2005). The representative LC–MS/MS chromatogram obtained for buprofezin in the tomato sample is presented in Figure 3.

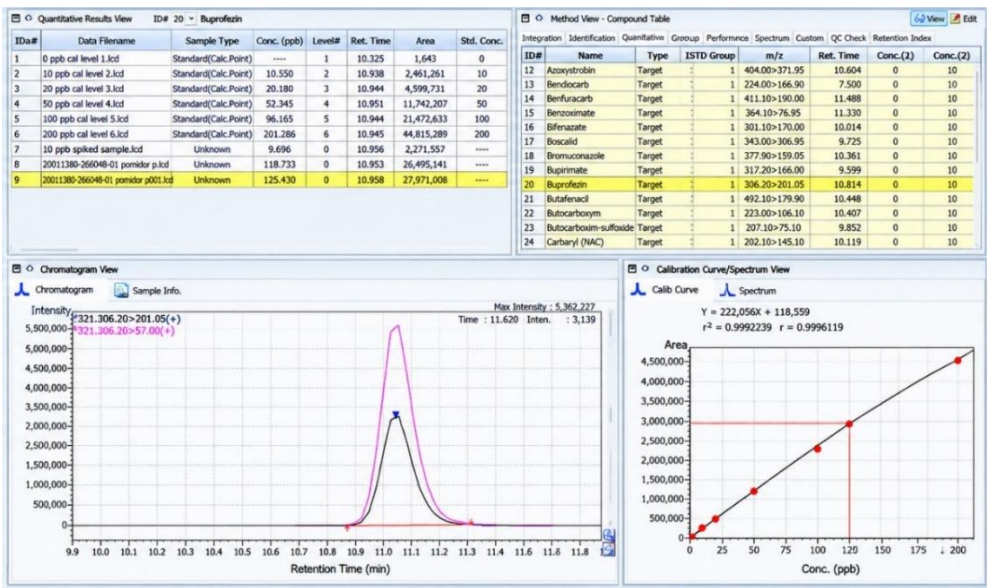


Figure 3
Representative LC–MS/MS chromatogram of buprofezin in the tomato sample

The determination of buprofezin residues in the tomato sample demonstrates the importance of regular monitoring of pesticide residues in agricultural products and the implementation of risk-based control measures (Carvalho, 2017; EFSA et al., 2023).

Table 4

Experimental pesticide residue results

Sample	Detected pesticide	Concentration (mg/kg)	Applicable MRL (mg/kg)	MRL source	Compliance
Tomato	Buprofezin	0.12	0.01	EU Pesticides Database	Non-compliant

Ecochemical Risks of Conventional Pesticides

The environmental impact of synthetic pesticides depends on their physicochemical properties and environmental fate (Tudi et al., 2021; Carvalho, 2017). After application, active substances may enter soil and water environments and undergo sorption, degradation, leaching, and other processes. These processes determine pesticide persistence, mobility, and the likelihood of exposure of non-target organisms (Tudi et al., 2021; Alengebawy et al., 2021).

Assessment from the Perspective of Food Safety and Human Health

Because vegetables are an important component of the daily diet, the determination of pesticide residues in them is of particular importance for food safety (Carvalho, 2017; EFSA et al., 2023). Risk assessment should be based not only on the detection of pesticide residues but also on their concentrations and compliance with existing maximum residue levels (MRLs) (EFSA et al., 2023; European Parliament and Council, 2005).

Modern analytical monitoring

systems enable early detection of pesticide residues, assessment of compliance with regulatory requirements, and protection of consumer health (EFSA et al., 2023).

Plant-Extract Biopesticides – a Safer Alternative

The potential environmental impacts of synthetic pesticide use have increased interest in biopesticides based on plant extracts (Isman, 2020; Damalas & Koutroubas, 2018). Scientific studies have demonstrated the biological activity of extracts and essential oils obtained from plants such as neem (*Azadirachta indica*), garlic (*Allium sativum*), thyme (*Thymus vulgaris*), rosemary (*Salvia rosmarinus*), clove (*Syzygium aromaticum*), and cinnamon (*Cinnamomum verum*) against various pests and plant pathogens (Isman, 2020; Pavela & Benelli, 2016).

The promising characteristics of plant-extract-based biopesticides are associated with their basis in naturally derived bioactive substances, their diverse modes of action, and, in some cases, their lower persistence in the environment (Isman, 2020; Pavela & Benelli, 2016; Regnault-Roger et al., 2012). However, the efficacy and ecotoxicological properties of these preparations may vary depending on the composition of the active components, formulation characteristics, and conditions of application (Pavela & Benelli, 2016; Isman & Grieneisen, 2014). Their relatively short duration of action, low stability, and the need for repeated application in some cases are among the factors that should be considered in practical use (Pavela & Benelli, 2016; Isman & Grieneisen, 2014; Regnault-Roger et al., 2012).

In this regard, rather than completely replacing synthetic pesticides, optimizing their use and gradually integrating plant-derived biopesticides into integrated plant protection systems is

considered a more realistic approach (Isman, 2020; Damalas & Koutroubas, 2018; European Commission, 2020). The application of plant-based preparations as alternatives to synthetic pesticides requires a comprehensive assessment of their efficacy, toxicological and ecotoxicological properties, residue behavior, and applicability under local agroecological conditions (Damalas & Koutroubas, 2018; Pavela & Benelli, 2016; Isman & Grieneisen, 2014).

Prospects for Application in Azerbaijan's Agriculture

Expanding the monitoring of pesticide residues using modern analytical methods, implementing risk-based control programs, and studying biopesticides based on plant extracts are considered promising directions for the development of sustainable agriculture in Azerbaijan (Damalas & Koutroubas, 2018; European Commission, 2020). Studying local plant resources as sources of biopesticides may contribute to reducing potential environmental impacts and improving the food safety system.

Conclusion

This study examined the applicability of the QuEChERS–LC–MS/MS method for determining synthetic pesticide residues in tomatoes grown in Azerbaijan and the ecochemical assessment of the obtained results. Modern analytical approaches enable the determination of pesticide residues and comparison of the obtained results with relevant regulatory requirements.

Scientific literature analysis indicates that biopesticides based on plant extracts can be considered among the promising alternatives for optimizing the use of synthetic pesticides and developing more sustainable plant protection approaches. However, their efficacy, safety, ecotoxicological properties, and applicability under local agroecological conditions should be evaluated separately.

Thus, regular monitoring of pesticide residues, application of risk-based assessment approaches, and scientifically based research on plant-derived biopesticides may support the development of sustainable and safe plant protection systems in Azerbaijan.

Conflict of Interest

The authors declare that they have no conflict of interest regarding this article.

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