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DETERMINATION OF PESTICIDE RESIDUES IN VEGETABLES USING GC-ECD WITH QuEChERS EXTRACTION

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Abstract: Pesticide residues present in primary and secondary agricultural products pose potential risks to human health. This study aimed to develop and validate an analytical method for assessing pesticide residues in commonly consumed vegetables from the Syrdarya region. Five vegetable samples were processed and analyzed for the separation and quantification of various pesticides using gas chromatography equipped with an electron capture detector (GC-ECD). An internal standard method was applied in accordance with international validation requirements for pesticide residue analysis in vegetable matrices. The method demonstrated excellent performance characteristics within globally accepted validation criteria, particularly at low concentration levels (0.02 µg/kg). High sensitivity, good linearity, and satisfactory precision were achieved. Among the five analyzed vegetable samples (hot pepper, cucumber, potato, bell pepper, and tomato), pesticide residue levels in four samples were below the maximum residue limits (MRLs) established by the Republic of Uzbekistan. However, in one tomato sample, the detected pesticide concentration exceeded the permissible limit. Although the overall contamination levels were moderate, continuous dietary exposure to pesticide residues may pose long-term health risks. Therefore, adherence to good agricultural practices (GAP) and pesticide safety regulations is strongly recommended. Continuous monitoring of pesticide residues in food products is also essential. Furthermore, the validated method can be effectively applied for the determination of pesticide residues in other vegetables, fruits, and cereal products.

Keywords: Pesticide residues; organochlorine pesticides; vegetable products; QuEChERS extraction; GC-ECD; gas chromatography; food safety; maximum residue limits (MRL); analytical validation; monitoring.

INTRODUCTION

Pesticides play a crucial role in agricultural production by increasing crop yield and protecting plants from pests and diseases. In modern intensive farming systems, they are considered essential tools for ensuring high productivity. Globally, pre- and post-harvest losses may reach up to 40–45%, which necessitates the implementation of effective pest control strategies [1,2].

The use of pesticides contributes to increased agricultural productivity, reduced production costs, and improved food security. In some cases, they also help suppress naturally occurring plant toxins, such as tropane and pyrrolizidine alkaloids [3]. Pesticides are classified according to their application purpose into insecticides, herbicides, fungicides, rodenticides, and other groups. Based on their origin, they are divided into natural and synthetic compounds. Synthetic pesticides are widely used worldwide due to their high efficiency and economic feasibility.

According to their chemical structure, pesticides can be categorized as organic or inorganic compounds. Inorganic pesticides include copper-based compounds, boric acid, silicates, sulfur, and arsenic-containing substances [4]. Among organic pesticides, organochlorine compounds are of particular concern due to their high stability and lipophilic nature.

Organochlorine pesticides (OCPs) are highly hydrophobic and degrade slowly in the environment, leading to persistence and bioaccumulation in biological systems [5]. These persistent chlorinated hydrocarbons were widely used in agriculture between the 1940s and

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1960s, and due to their environmental stability, they are still detected in various ecosystems today [6]. Pesticide residues refer to the parent compound, its metabolites, or degradation products detected in food or environmental matrices [7].

Plants can absorb OCPs through roots, stems, and leaves, leading to accumulation in plant tissues and potential transfer into the food chain [8]. In animals, these compounds tend to accumulate in adipose tissues, resulting in biomagnification [9]. Therefore, the determination and monitoring of pesticide residues in vegetables are of significant scientific and public health importance for ensuring food safety.

2. MATERIALS AND METHODS

2.1. Chemicals and Reagents

Analytical standards of organochlorine pesticides (2,4,5,6-tetrachloro-m-xylene, α-BHC, β-BHC, γ-BHC, heptachlor, aldrin, dieldrin, endrin, 4,4′-DDT, 4,4′-DDE, 4,4′-DDD, endosulfan isomers, and others) with 99.9% purity were purchased from Sigma-Aldrich (Germany). Decachlorobiphenyl was used as an internal standard.

Acetonitrile (99.6% purity), magnesium sulfate ($MgSO_4$), and sodium citrate of analytical grade were used for extraction. Dispersive solid-phase extraction (d-SPE) clean-up kits (50 mg PSA, 50 mg C18, 150 mg $MgSO_4$) based on AOAC 2007.01 protocol were obtained from Biocomma Limited (China).

2.2. Sample Collection and Preparation

Five commonly consumed vegetables in the Syrdarya region (cucumber, tomato, hot pepper, bell pepper, and eggplant) were selected for analysis. Samples were collected from a local market in Gulistan city using random sampling procedures. Sampling was performed in accordance with the European Commission Directive 2002/63/EC guidelines.

For each vegetable type, seven sub-samples were combined to form a representative laboratory sample (1–2 kg). Samples were transported to the laboratory under cooled conditions and stored in a refrigerator until analysis.

2.3. Preparation of Standard Solutions

A stock standard solution with a concentration of 200 ± 0.5 μg/cm³ was used to prepare a 1 μg/cm³ working calibration solution. Subsequent serial dilutions were performed to obtain intermediate calibration solutions at concentrations of 0.1, 0.05, and 0.01 μg/cm³.

All standard solutions were stored in tightly sealed glass containers at +4 °C. Calibration curves were constructed using the prepared standard solutions at different concentration levels.

Table 1. Concentration levels of calibration standard solutions for the determination of organochlorine pesticides using GC-ECD.

No.		Working solution (mL)	Solvent (mL)	Obtained concentration (μg/mL)
1	Solution 1	10	10	1
2	Solution 2	10	90	0,1
3	Solution 3	10 mL of Solution 2	40	0,05
4	Solution 4	10 mL of Solution 2	90	0,001

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2.4. Gas Chromatography Analysis (GC-ECD)

Pesticide analysis was performed using a Chromatec Kristall 9000 gas chromatograph equipped with an electron capture detector (ECD). Separation was achieved on a BP21 capillary column (50 m × 0.32 mm I.D., 0.5 µm film thickness; S/N 333927B11; P/N 054480).

The operating conditions were as follows:

- Air flow rate: 250 mL/min
- Hydrogen flow rate: 25 mL/min
- ECD temperature: 300 °C
- Inlet temperature: 190 °C
- Oven temperature: 250 °C
- Carrier gas pressure: 23.408 kPa
- Increased pressure mode: 170 kPa
- Total flow rate: 4.3 mL/min
- Split flow rate: 7.5 mL/min

2.5. QuEChERS Extraction and Dispersive SPE Clean-up Procedure

Sample extraction and purification were carried out using the QuEChERS method (Quick, Easy, Cheap, Effective, Rugged, and Safe) for pesticide residue analysis [12]. A 5 g portion of homogenized sample was transferred into a 50 mL polypropylene centrifuge tube. Subsequently, 15 mL of acetonitrile and 5 mL of distilled water were added. After adding two ceramic homogenizers, the mixture was shaken vigorously for 30 seconds. Then, 2.5 g of extraction salt mixture (magnesium sulfate : sodium citrate, 4:1, w/w) was added. The tubes were immediately shaken for 1 minute and centrifuged at 6000 rpm for 10 minutes. An aliquot of the supernatant was transferred into a dispersive SPE tube (AOAC 2007.01, 2 mL; containing 50 mg PSA, 50 mg C18, and 150 mg $MgSO_4$; Biocomma Limited, China). The mixture was vortexed for 10 minutes and centrifuged at 4000 rpm for 3 minutes. The purified extract was transferred into labeled GC vials for chromatographic analysis.

3. RESULTS

3.1. Data Processing

The concentration of pesticides in the analyzed raw material or product (X_2, mg/kg) was calculated based on the calibration curve using the following equation:

$$X_2 = \frac{m_1 \cdot V_1}{m_2 \cdot V_2},$$

where:

- m_1 – mass of the pesticide determined from the calibration curve (µg);
- V_1 – total volume of the extract solution from which the aliquot was taken (cm^3 or mL);
- m_2 – mass of the analyzed sample (g);
- V_2 – volume of the aliquot injected into the chromatograph (cm^3 or mL).

Calibration solutions were stored for no longer than two weeks. Fresh calibration standards were prepared to verify the calibration curves when necessary.

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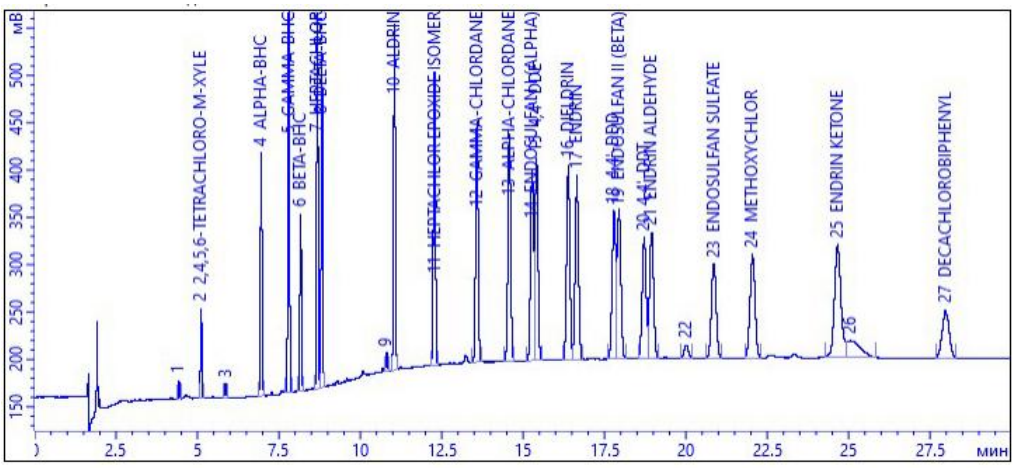

The chromatogram obtained from calibration analysis, showing the sequential elution of organochlorine pesticides, is presented in Figure 1.

3.2. Method Validation

In this study, organochlorine pesticide residues in commonly consumed vegetables from the Syrdarya region were determined using the QuEChERS extraction procedure combined with GC-ECD analysis, and the analytical performance of the method was evaluated.

The applied method demonstrated high sensitivity and selectivity. According to the validation results:

The calibration curves showed excellent linearity, with a coefficient of determination $R^2 \geq 0.99$**;**

Recovery values ranged between 70–120%**;**

Relative standard deviation (RSD) values were below 20%**;**

The limit of detection (LOD) allowed reliable determination even at low concentration levels.

The analytical results indicated that pesticide residue levels in four out of five vegetable samples were below the established maximum residue limits (MRLs) and therefore complied with sanitary and hygienic standards. However, in the tomato sample, the detected pesticide concentration exceeded the permissible level, which may indicate non-compliance with good agricultural practices or insufficient pre-harvest interval (PHI).

The findings highlight the necessity of regular monitoring of pesticide residues in vegetable products within the region. The validated high-sensitivity analytical method can be effectively applied for food safety assessment, strengthening sanitary control systems, and ensuring compliance with export regulations.

DISCUSSION

The results of the present study provide a reliable scientific basis for the determination of organochlorine pesticide residues in vegetable matrices. The QuEChERS extraction method proved to be rapid, cost-effective, and efficient, requiring minimal solvent consumption while maintaining high extraction efficiency. The dispersive SPE clean-up step effectively reduced matrix interferences and improved analytical accuracy. The GC-ECD detector exhibited high sensitivity toward organochlorine compounds, enabling reliable detection at low concentration levels. The excellent linearity of calibration curves ($R^2 \geq 0.99$), acceptable recovery values, and low RSD percentages confirm the validity and reproducibility of the analytical procedure.

Differences in pesticide residue levels among vegetable types were observed. While most samples complied with regulatory limits, the elevated concentration detected in the tomato sample may be associated with specific agricultural application practices. Morphological

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characteristics of vegetables, such as surface structure, wax layer composition, and moisture content, may influence pesticide retention and persistence.

Due to the lipophilic nature and chemical stability of organochlorine pesticides, these compounds may persist in plant tissues. Therefore, strict adherence to pre-harvest intervals is essential. Excessive or uncontrolled pesticide application may introduce residues into the food chain, potentially leading to long-term low-dose exposure risks for consumers. Overall, the study emphasizes the importance of regional monitoring systems. The combined QuEChERS and GC-ECD approach represents a reliable and economically feasible solution for the determination of organochlorine pesticide residues in vegetables and can be extended to other agricultural commodities.

CONCLUSION

In this study, organochlorine pesticide residues in commonly consumed vegetables from the Syrdarya region were successfully determined using QuEChERS extraction combined with GC-ECD analysis. The method demonstrated high sensitivity, excellent linearity, and satisfactory repeatability, ensuring reliable detection of pesticide residues in complex vegetable matrices.

The results showed that four out of five vegetable samples contained pesticide residues below the established MRLs and complied with sanitary standards. However, the elevated concentration detected in the tomato sample indicates the need for improved compliance with agricultural application practices and pre-harvest interval requirements.

The findings confirm the importance of continuous monitoring of pesticide residues at the regional level. The proposed analytical approach can be recommended as an effective and practical method for pesticide residue determination in vegetables and for strengthening food safety control systems.

REFERENCES

1. Tilman, D., Cassman, K.G., Matson, P.A., Naylor, R. and Polasky, S. (2002) Agricultural Sustainability and Intensive Production Practices. Nature, 418, 671-677.

2. Amirahmadi M, Yazdanpanah H, Kobarfard F, Shoeibi S, Pirali-Hamedani M, Rastegar H. Exposure Assessment for Some Pesticides through Rice Consumption in Iran Using a Multiresidue Analysis by GC-MS. Iran. J. Pharm. Res. 2018;17:124–139.

3. Abia, W.A.; Montgomery, H.; Nugent, A.P.; Elliott, C.T. Tropane alkaloid contamination of agricultural commodities and food products in relation to consumer health: Learnings from the 2019 Uganda food aid outbreak. Compr. Rev. Food Sci. Food Saf. 2021, 20, 501–525.

4. Sharma, A.; Shukla, A.; Attri, K.; Kumar, M.; Kumar, P.; Suttee, A.; Singh, G.; Barnwal, R.; Singla, N. Global trends in pesticides: A looming threat and viable alternatives. Ecotoxicol. Environ. Saf. 2020, 201, 110812.

5. Cindoruk SS, Sakin AE, Tasdemir Y. Levels of persistent organic pollutants in Pine tree components and ambient air. Environ Pollut. 2020;256: 113418. https://doi.org/10.1016/j.envpol.2019.113418.

6. Kiran RA, Mamata SA. 2019. Health and Environmental Effects of Organochlorine Pesticides in Developing Countries. Open Access J Environ Soil Sci. https://doi.org/10.32474/oajess.2019.02.000135.

7. Dasika R, Tangirala S, Naishadham P. Pesticide residue analysis of fruits and vegetables. J Environ Chem Ecotoxicol. 2012;4(2):19–28. https://doi.org/10.5897/jece11.072.

8. Rachina C, Sharpanabharathi N, Anjan KP, Rama MK, Azeez PA. Organochlorine pesticide residues in plants and their possible ecotoxicological and agri food impacts. Sci Rep. 2021. https://doi.org/10.1038/s41598-021-97286-4.

9. Manoranjan P, Bijoy KP, Dinabandhu J, Asit KP, Gayatri S. Effect of organochlorine pesticides on living organisms and environment. Chem Sci Rev Lett. 2020;9(35):682–6. https://doi.org/10.37273/chesci.CS2051063.

pISSN:2349-5707 Volume:13,Issue 2, February -2026 eISSN:2349-5715

10. European Commission (2002) Establishing Community Methods of Sampling for the Official Control of Pesticide Residues in and on Products of Plant and Animal Origin and Repealing Directive. The Official Journal of the European Union, 187, 30-43.

11. Anastassiades, M., Lehotay, S.J., Stajnbaher, D. and Schenck, F.J. (2003) Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce. Journal of AOAC INTERNATIONAL, 86, 412-431.