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Validation and Application of Combined QuEChERS Extraction With Cartridge Solid Phase Extraction Cleanup for Pesticide Multiresidue Analysis in Some Vegetables by GC-ECD

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Abstract:

In this work, a sample preparation and analysis method that combines QuEChERS extraction with c-SPE clean-up procedures and GC-ECD analysis method for detection and quantitation of fifteen multi-class pesticide residues in vegetable commodities were validated. The methodology was validated in terms of linearity, precision, and accuracy. The correlation coefficient results were ranged from 0.9949 to 0.9998. The repeatability was ranged from 0.11 to 8.46 % and the obtained limits of detection (LODs) for all investigated pesticides ranged from 0.0003 to 0.0906ng/ml. The validated procedures were then applied in the analysis of target pesticides in eighteen real vegetable samples (tomato, potato, cucumber, and carrot) purchased from the main markets located in Sana'a city (Yemen). The suitability of the proposed method was first verified by spiking vegetable blanks and calculating the accuracy, precision, and LOD for the selected pesticides in each vegetable commodity. For spiked vegetable samples, LODs were between 0.0044 and 2.4100 ng/g. Recoveries' values were between 82.75% and 109.60%, while the relative standard deviation (RSD) did not exceed 10.50 %. The analysis results of the real samples showed that out of eighteen different vegetable samples, only one sample was found clean from target pesticide residues. Six vegetable samples were found to have pesticides lower than the accepted maximum residue limits (MRLs) and eleven vegetable samples were found to have pesticides mostly higher than the accepted MRLs as adopted by the FAO/WHO Codex Alimentations or European Commission.

Keywords: QuEChERS Extraction, c-SPE Cleanup, Pesticide, GC-ECD, Vegetables, Yemen.

INTRODUCTION

Pesticides are considered as one of the major environmental pollutants that are originally designed to control or eradicate pests from agricultural fields and households (Akhtar *et al.*, 2018). They provide important benefits in raising agricultural products to grow the quantity and quality of food needed to tolerate the human population. Botanical pesticides have been used traditionally by human communities in many parts of the world against various pest species (Iqbal and Ashraf, 2019; Sattar *et al.*, 2016). The extensive use of pesticides or harvesting the crops before the complete degradation of the pesticides can end up with significant levels of residues in food samples which may cause critical harm to humans, animals and other harvests (Bonner and Alavanja, 2017; Kim *et al.*, 2017; Nicolopoulou-Stamati *et al.*, 2016; Osadebe *et al.*, 2018; Silva *et al.*, 2015).

The European Commission has set maximum residue levels (MRLs) to protect consumers from exposure to improper levels of pesticide residues in food and feed, The MRL is defined as the highest possible level of a pesticide residue that is legally authorized in food and feed (Regulation (EC), 2005). Because of the trace amount of target analytes and complex interference components in vegetable matrices, pesticide residues determination in vegetables stays a challenge (Rizzetti *et al.*, 2016). The efficient sample preparation and trace-level detection and identification are important aspects of analytical methodologies due to low detection levels needed to evaluate food safety and the complex nature of the matrices in which the target compounds are present (Lozano *et al.*, 2016; OECD, 2020).

Increasing efforts have been made to develop effective, simple, and quick sample preparation techniques, to eliminate interference and attain good performance of the analytical method. Quick, easy, cheap, effective, rugged and safe (QuEChERS) method developed by (Anastassiades *et al.*, 2003) has received great

interest and reached great successes (Camara *et al.*, 2017; Elgueta *et al.*, 2020; Lehotay *et al.*, 2010; Maciej, 2019; Oliva *et al.*, 2017; Turan *et al.*, 2020). The method involves an initial solvent extraction with acetonitrile and then purification of the extract using dispersive solid-phase extraction (d-SPE). Due to many advantages of QuEChERS method, it was established as a reference method for analysis of pesticide residues in food by AOAC (Gonzalez-Curbelo *et al.*, 2015; Lehotay *et al.*, 2007; Wilkowska and Biziuk, 2011) and Uruapan Union (Lemos *et al.*, 2016). Despite clear advantages of the method, it poses some limitations such as the lack of pre-concentration step as one gram of sample is extracted and purified into one milliliter of the final extract. The obtained concentration of the extract is usually lower than the concentration that could be obtained by the use of most traditional procedures. This necessitates the need for pre-concentration of the final extract to a greater extent to provide the necessary sensitivity and to achieve the low limits of quantification (LOQ). Moreover, the d-SPE cleanup procedure suggested in the method is found to be inadequate for purification of the intense colored extracts which could interfere with the identification and quantification of the target pesticides and jeopardize the performance of the analysis system performance (Alnedhary *et al.*, 2020). On the other hand, the use of higher amounts of adsorbents in d-SPE was found to affect the recovery of most of the target analytes (Huertas-Pérez *et al.*, 2019; Vaclavik *et al.*, 2018).

To overcome the downsides of the QuEChERS and d-SPE technique and achieve a better sample preparation, several researchers have attempted to combine the procedures of QuEChERS extraction with cartridge solid phase (c-SPE) cleanup (Alnedhary *et al.*, 2020; Michelle *et al.*, 2013). This combination facilitates the use of higher quantities of adsorbent materials without the fear of losing some of the analytes. This is a result of the method's flexibility where the user could choose an appropriate solvent mixture that selectively

elutes the analytes and retains the recovery, sensitivity, and accuracy of the method at high values (Alnedhary *et al.*, 2020). Besides, the high efficiency of the cleanup procedure is found to have two additional advantages. It reduces the eluent volume which improves the method sensitivity and also makes the filtration step unnecessary since c-SPE possesses built-in filtration features (Michelle *et al.*, 2013; Oshita and Jardim, 2015; Tayeb *et al.*, 2015).

The most frequently used technique in simultaneous pesticide analysis is gas chromatography (GC) due to its high-resolution capability and the availability of selective detectors includes mass spectrometry (MS), nitrogen phosphorous detector (NPD), and electron capture detector (ECD) (Colume *et al.*, 2001; Lu *et al.*, 2013; Zawiyah *et al.*, 2006). These detectors are relevant to classes of pesticides with comparable properties and thus, very low limits of detection are obtained. The Electron Capture Detector (ECD) is widely available in both research and commercial laboratories. ECD detector is very selective and sensitive to electronegative compounds, such as organochlorine, organophosphate, pyrethroid, and organonitrogen pesticides. The sensitivity of ECD for some of these compounds could be as low as parts per trillion (ppt) ranges (Chung and Chen, 2015). These unique properties encourage the use of the QuEChERS sample preparation method and gas chromatography coupled with ECD detector for pesticide residues analysis (Alder *et al.*, 2006; Elgueta *et al.*, 2020; Maciej, 2019). This work aimed to validate a sample preparation and analysis method that combines QuEChERS extraction with c-SPE sample preparation procedures and GC-ECD analysis method for sensitive detection and quantitation of pesticides residues in vegetable accommodates (tomato, potato, cucumber, and carrot). The validated procedures were then applied for the analysis of target pesticides in eighteen real vegetable samples purchased from

the main markets located in Sana'a city (Yemen).

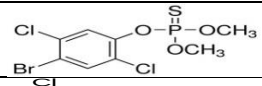
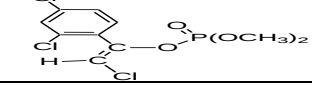
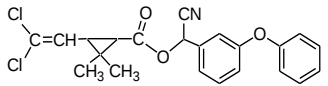
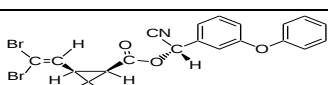
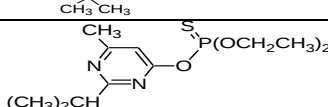
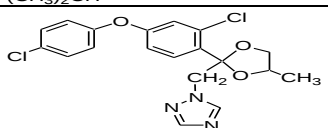
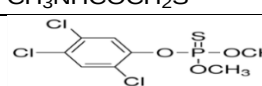
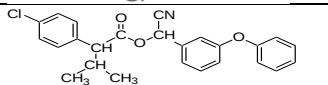
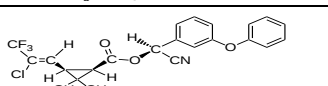
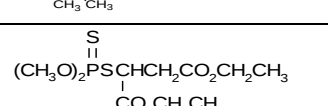
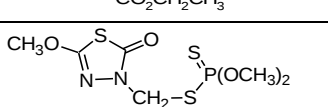
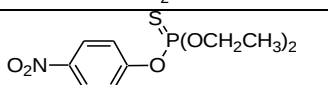
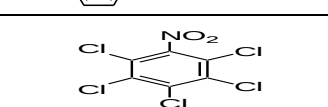
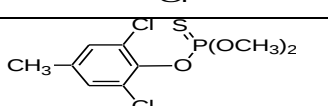
MATERIALS AND METHODS

Pesticides standards: Diazinon 98.3%, Fenchlorphos 99.1%, Malathion 97.2%, Parathion 98.8%, Methidathion 95.8%, Lambda-cyhalothrin 98%, Cypermethrin 95.8%, Tolclofos-methyl 99.3%, Bromophos-methyl 99.4%, Chlorfenvinphos 97.3%, Dimethoate 99.6%, Difenconazole 99.3%, Deltamethrin 98.0% and Fenvalerate 98.3%, Quintozene 99.7% were from Sigma-Aldrich/ Fluka/ Zwijndrecht, The Netherlands. Pesticide names, class, action, molecular formulas, and chemical structures are shown in (Table 1).

All the solvents used were pesticide residues or HPLC grades. Primary Secondary Amin (Agilent, US; PSA 40 mm particle size) and activated charcoal 15-30 mesh size (Merck, Germany) was also used.

The Shimadzu gas chromatography electron capture detector system (GC 2010) equipped with a DB-1 capillary column (0.32 mm x 30 m, 0.25 μ m film thickness, supplied by Agilent, Palo Alto, CA, USA) was used in all analysis. The temperatures of the detector and injector in the splitless mode were set at 280°C and 250°C, respectively. The Oven was set at 85°C (3 minutes.); raised to 170°C at 30°C minute⁻¹ and kept at 170°C for 4 minutes. The temperature was then raised to 270°C at 10°C minute⁻¹ and held at 270°C for 14 minutes. The total run time was 33.83 minutes. Helium (99.999%) was used as a carrier gas with a flow rate of 1.16 mL minute⁻¹. The identification of individual pesticide was based on a comparison of the retention time in the sample chromatogram and the standard solution chromatogram.

Table 1. Pesticide names, class, action, molecular formulas, and chemical structures of selected pesticides studied.

No.	Analyte	Pesticide class	Action	Formula	Exact mass	Structure*
1	Bromophos-methyl	OP	Insecticide	$C_8H_8BrCl_2O_3PS$	366.0	
2	Chlorfenvinphos-methyl	OP	Insecticide	$C_{10}H_{10}Cl_3O_4P$	331.5	
3	Cypermethrin	Pyrethroid	Insecticide	$C_{22}H_{19}Cl_2NO_3$	416.3	
4	Deltamethrin	Pyrethroid	Insecticide	$C_{22}H_{19}Br_2NO_3$	505.2	
5	Diazinon	OP	Insecticide, acaricide	$C_{12}H_{21}N_2O_3PS$	304.3	
6	Difenoconazole	Triazole	Fungicide	$C_{19}H_{17}Cl_2N_3O_3$	406.3	
7	Dimethoate	OP	Insecticide, acaricide	$C_5H_{12}NO_3PS_2$	229.3	
8	Fenchlorphos (Ronnel)	OP	Insecticide	$C_8H_8Cl_3O_3PS$	321.5	
9	Fenvalerate	Pyrethroid	Insecticide, acaricide, ixodicide	$C_{25}H_{22}ClNO_3$	419.9	
10	lambda-Cyhalothrin	Pyrethroid	Insecticide	$C_{23}H_{19}ClF_3NO_3$	449.9	
11	Malathion	OP	Insecticide, acaricide	$C_{10}H_{19}O_6PS_2$	330.4	
12	Methidathion	OP	Insecticide, acaricide	$C_6H_{11}N_2O_4PS_3$	302.3	
13	Parathion	OP	Insecticide, acaricide	$C_{10}H_{14}NO_5PS$	291.3	
14	Quintozene	aromatic hydrocarbon; chlorophenyl	Fungicide	$C_6Cl_5NO_2$	295.3	
15	Tolclofos-methyl	aromatic hydrocarbon; chlorophenyl	Fungicide	$C_9H_{11}Cl_2O_3PS$	301.1	

OP: Organophosphorus

*<http://pubchem.ncbi.nlm.nih.gov/compound/20/2/2020>

Preparation of Standard Solutions

Individual pesticide standard solutions (2000 µg/mL) for all target pesticides were prepared in hexane-acetone (9:1) and kept at (-4°C) until use. Mixture standards of 100 µg/mL of target pesticides were prepared in hexane-acetone (9:1) (Bozena *et al.*, 2015; Bozena *et al.*, 2016) and used for blank and real samples spiking. Calibration mixtures with concentration levels (0.01-2 µg/mL) were prepared in hexane-acetone (9:1) from the intermediate mixture standard solutions.

Blank Samples and Spiked Samples

Blank vegetable samples of cucumber, tomato, carrot, and potato were collected from organic cultivation sources and used for the validation study. They were first analyzed to ensure the absence of the target pesticides residues. They were chopped into small pieces before mixer blending then homogenized and spiked with suitable amounts of pesticide mixture. The spiked samples were properly homogenized and kept overnight before the extraction and cleanup procedures.

Sampling and Transportations

A total of 18 samples including 7 tomatoes, 4 potatoes, 4 cucumbers, and 3 carrots were obtained from wholesale fruit and vegetable markets in Sana'a city, Yemen. Real vegetable samples (1-2 kg) were randomly collected in polythene pages from the main wholesale markets in Sana'a city (i.e. Ali Mohsen, Alhaothili, and Raimat Homaid). The samples were transported to the laboratory, then they were cleaned by washing with tap and distilled water, sub-sampled, and made ready for further processing.

QuEChERS Extraction

The QuEChERS method was used for sample extraction (AOAC, 2011). A volume of 10 mL of acetonitrile containing 1% acetic acid was added to each weighted sample. 10 g of each cucumber and tomato and in the case of

vegetables with less water content, carrot, and potato (5 g of sample mixed with 5 mL deionized water) was also added and thoroughly mixed. After a one minute shake, buffering extraction salts (4 g anhydrous magnesium sulfate, 1 g anhydrous sodium acetate) were added. Following another 2 minute shakes, the sample was centrifuged for 5 minutes at 5000 rpm. Finally, the acetonitrile layer was separated and used for the cleanup procedures.

Cartridge Solid Phase Extraction Cleanup (c-SPE)

SPE Cartridges were prepared by 400 mg PSA/400 mg activated charcoal/1 g anhydrous sodium sulfate respectively from the bottom to the top. The SPE cartridge was conditioned with 5 mL of (3:1) acetonitrile: toluene followed by 8.0 mL of the acetonitrile layer that was passed through the cartridge which then eluted with 20 mL of (3:1) acetonitrile: toluene. The eluent was evaporated near to dryness using a rotary evaporator at 40 °C (Alnedhary *et al.*, 2020). The final volume was reconstituted to 2 mL using (9:1) hexane-acetone (Bozena *et al.*, 2015; Bozena *et al.*, 2016). The resulting final extracts for all matrices were analyzed by GC-ECD.

Validation Study

For method validation, Linearity, Calibration Curves, Accuracy, Precision, and Limit of Detection were studied as follow:

Linearity and Calibration Curves

Linearity was determined by the analysis of a series of seven standard solutions (0.01 to 2.00 µg/mL) in triplicates. The detector responses were used to develop a standard calibration curve for each pesticide. The linear regression equation and correlation coefficient were then calculated from the calibration curves.

Accuracy (Recovery) and Precision (Repeatability)

To carry out the accuracy and precision experiments, four independent analyses of

vegetable samples spiked with pesticides at a level of $1.0\mu\text{g/g}$ were performed. Repeatability of the method was evaluated using the relative standard deviation (RSD %) associated with recovery measurements of the pesticides. Recovery studies were performed to examine the efficiency of the developed cleanup method.

Limit of Detection (LOD)

limit of detection (LOD) for each pesticide was calculated from the chromatograms of the concentration of the standard of ($0.5\mu\text{g/mL}$).

RESULTS AND DISCUSSION

Validation results

For the validation of the analysis method, $1\mu\text{g/mL}$ mixture of standards of the selected fifteen pesticides was prepared. After adjusting the instrument parameters, all of the selected pesticides were determined simultaneously under the same conditions. The retention time for each standard was determined. Figure 1 showed the typical and representative GC-ECD chromatogram of the prepared pesticide mixture at a concentration of $1\mu\text{g/mL}$.

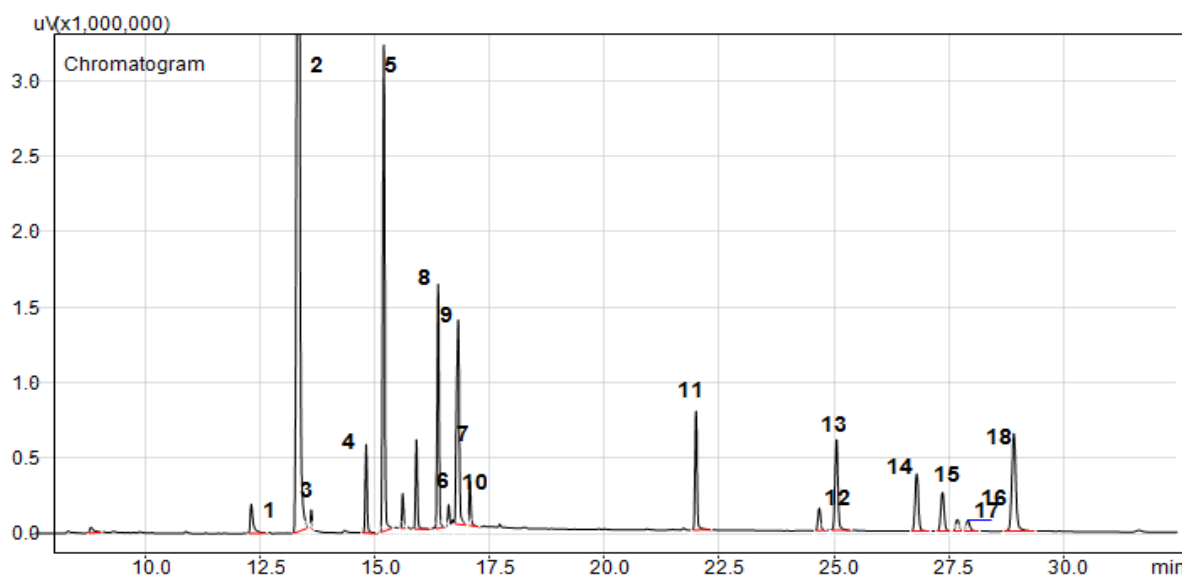


Fig. 1. Chromatograph of 16-pesticides Mixture Standard ($1.0\mu\text{g/mL}^{-1}$). Peaks are identified as follows: 1. Dimethoate, 2. Quintozene, 3. Diazinon, 4. Tolclofos-methyl, 5. Fenchlorphos, 6. Malathion, 7. Parathion, 8. Bromophose-methyl, 9. Chlorfenvinphos-methyl, 10. Methidathion, 11. Lambda-cyhalothrin, 12,13. Cypermethrin, 14,15. Fenvalerate, 16,17. Difenconazole, 18. Deltamethrin.

Table 2 summarized the retention time (R_t), calibration ranges, calibration equations, regression coefficients (R^2), LODs, and repeatability of the analysis methods (%RSD) for the fifteen pesticide standards at a concentration of $1\mu\text{g/mL}$. The correlation coefficient (R^2) results in Table 2 for all pesticide calibration curves were not lower than 0.9949 indicating

high linearity and accuracy of the analysis method. The repeatability results as % RSD ranged from 0.11 and 8.46 % which is within the acceptable range $< 20\%$ (European Commission, 2010) at this concentration level. The low values of % RSD are indicative of the high precision of the analysis method (Słowik-Borowiec and Walorczyk, 2015). The lowest

value of LOD was 0.0003 ng/mL for quintozene pesticide while the highest value was found to

be 0.0906ng/mL for diazinon.

Table 2. Validation Results of Pesticides Analysis Using GC-ECD.

Pesticide	Retention Time (min.)	Calibration range	Calibration equation	R ²	LOD	RSD*
		µg/mL			ng/mL	(n=3)
Dimethoate	12.29	0.01-2	y = 1.3E+06x - 18349	0.9996	0.0484	8.46
Quintozene	13.33	0.01-2	y = 3.2E+07x + 756165	0.9991	0.0003	0.11
Diazinon	13.64	0.01-2	y = 1.3E+06x + 4617.3	0.9965	0.0906	3.82
Tolclofos-methyl	14.80	0.01-2	y = 2.0E+06x + 7597.9	0.9982	0.0234	6.00
Fenchlorphos	15.19	0.01-2	y = 1.0E+07x + 209225	0.9991	0.0005	3.77
Malathion	15.60	0.01-2	y = 7.0E+05x + 2475.6	0.9975	0.0572	3.93
Parathion	15.90	0.01-2	y = 1.9E+06x + 18347	0.9987	0.0022	0.13
Bromophos	16.37	0.01-2	y = 5.4E+06x + 8943.7	0.9984	0.0009	4.03
Chlorfenvinphos-methyl	16.81	0.01-2	y = 6.4E+06x - 51478	0.9998	0.0011	2.28
Methidathion	17.06	0.01-2	y = 1.1E+06x - 17872	0.9991	0.0410	0.55
lambda-Cyhalothrin	21.99	0.01-2	y = 2.7E+06x + 26796	0.9949	0.0015	4.22
Cypermethrin	24.67	0.01-2	y = 4.1E+06x + 12307	0.9993	0.0016	6.24
Fenvalerate	26.79	0.01-2	y = 4.8E+06x + 3057.2	0.9976	0.0313	2.53
Difenoconazole	27.67	0.01-2	y = 2.1E+06x - 589.77	0.9966	0.0852	2.08
Deltamethrin	28.91	0.01-2	y = 5.8E+06x + 25835	0.9986	0.0014	7.47

The suitability of the proposed method for the pesticides residue analysis was first verified by spiking vegetable blanks and calculating the accuracy as recovery (%R), precision as repeatability (%RSD), and limit of detection (LOD) for the selected pesticides in cucumber, tomato, carrot and potato vegetables.

Method Verification

To verify the accuracy and precision of the proposed method and calculating LOD for multi-residue analysis in selected four vegetables, three blank samples from each vegetable were spiked with pesticides mix-standard at the concentration level of 1.0 mg/kg, and then each sample was carefully mixed and kept overnight before the extraction and purification procedures were carried out. The accuracy calculated as average recoveries (%R) values were between 82.75 and 109.60%, while %RSD did not exceed

10.50 % which indicative of the high accuracy and precision of the analysis method. LODs were ranged from 0.0044 to 2.4100 ng/g therefore, the method limit of quantification (LOQs) for all target pesticides in the examined vegetable commodities were below the MRLs values according to FAO/WHO (Codex Alimentarius, 2020) or EU (European Commission, 2020) which prove the sensitivity and suitability of the analysis method. The results were presented in (Table S1: Supplementary Files).

Real Samples Results

A total of eighteen real vegetable samples (seven tomatoes, four potatoes, four cucumbers, and three carrots) were analyzed for pesticide residues contents. The residue results of the selected fifteen pesticides in the four vegetable commodities showed that only one sample was

found clean from target pesticides residues, Six vegetables samples were found to have pesticides lower than the accepted MRLs and eleven vegetables samples were found to have pesticides mostly higher than the accepted maximum residue limits (MRLs) as adopted by the FAO/WHO (Codex Alimentarius, 2020) or EU (European Commission, 2020).

Table S2: (Supplementary Files) and Table 3 summarized the analysis results of the targeted pesticide residues in vegetables real samples and Figures (S1-S4: Supplementary Files) showed the analysis results of vegetables real samples.

Table 3. Pesticide Residue ($\mu\text{g/g}$) Detected in Cucumber and Carrot Real Samples.

Pesticide	RT	Cucumber				Carrot			*Codex MRL	**EU MRL
		(C1)	(C2)	(C3)	(C4)	(Ca1)	(Ca2)	(Ca3)		
Dimethoate	12.16	N.D	N.D	N.D	N.D	N.D	N.D	N.D	—	0.01
Quintozone	13.17	N.D	0.0081	N.D	0.0140	N.D	N.D	N.D	0.02	0.02
Diazinon	13.46	N.D	N.D	N.D	N.D	N.D	N.D	N.D	0.5	0.01
Tolclofos-methyl	14.66	N.D	N.D	N.D	N.D	N.D	N.D	N.D	—	0.01
Fenchlorphos	15.05	0.0305	0.0194	0.0227	0.0083	0.2331	0.0233	0.0067	—	0.01
Malathion	15.45	N.D	N.D	N.D	N.D	N.D	N.D	N.D	0.5	0.02
Parathion	15.76	N.D	N.D	N.D	N.D	N.D	N.D	N.D	—	0.05
Bromophos	16.23	0.0400	0.0200	0.032	0.036	N.D	N.D	N.D	—	0.02
Chlorfenvinphos-methyl	16.48	N.D	N.D	N.D	N.D	N.D	0.0670	N.D	—	0.01
Methidathion	16.69	N.D	N.D	N.D	N.D	N.D	N.D	N.D	0.1	0.02
Lambda-cyhalothrin	21.82	N.D	N.D	N.D	N.D	0.0052	0.0105	N.D	0.05	0.07
Cypermethrin	24.67	N.D	N.D	N.D	N.D	N.D	N.D	N.D	0.2	0.5
Fenvalerate	26.79	N.D	N.D	N.D	N.D	N.D	N.D	N.D	—	0.1
Difenoconazole	27.67	N.D	N.D	N.D	N.D	N.D	N.D	N.D	0.6	2
Deltamethrin	28.02	N.D	N.D	N.D	N.D	N.D	N.D	N.D	0.2	0.07

* Codex (FAO/WHO) (mg/kg) (FAO, 2020) **EU (mg/kg) (Europa, 2020)

The results of pesticide analysis in real samples were as follows: in tomato, out of the seven samples, only one showed no pesticide residues which could mean that they were absent or their level was below the method detection limit. Six samples showed the presence of fenchlorphos but its concentration

(0.0179 $\mu\text{g/g}$) exceeded the EU MRL in only one of them. The level of lambda-cyhalothrin was detectable by the developed method in three samples but below the MRL. Tolclofos-methyl was found in only one real sample but its concentration was higher than the MRL by more than 28 times.

In the case of potato, all samples were contaminated with fenchlorphos and its concentration exceeded the EU MRL. Similarly, bromophos was also quantified in the four samples and its concentration exceeded the MRL in three samples. Although quitozene was detectable in two samples, its quantity was not significant.

The four samples of cucumber were found to contain fenchlorphos with concentrations exceeded the EU MRL in three of them (0.0194 to 0.0305 µg/g), two samples contained quitozene with concentrations 0.0081 and 0.0140 µg/g. All of the samples were also found contaminated with bromophos with concentrations exceeded the EU MRL.

In the case of carrot, the results showed that Fenchlorphos was detected in the three samples, Out of these, two exceeded the EU MRL with concentrations of 0.02331 and 0.2331 µg/g. Lambda-cyhalothrin (0.0052 to 0.0105 µg/g) was detected in two samples but below the MRL, Chlorfenvinphos-methyl (0.0670 µg/g) was found in only one carrot sample but its concentration was higher than the MRL by more than 6 times.

All other investigated pesticides in the samples were either not detected or were detected below the limits of quantifications and thus were not quantified. The analyzed samples contained residues from the monitored pesticides that were higher than the accepted maximum residue limits (MRLs) as adopted by the FAO/WHO Codex Alimentarius and the EU Commission. A previous study demonstrated the combined sample preparation method being cost-effective with good simplification, recovery, and cleanup capacity and proved to be efficient and suitable for the proposed application (Alnedhary *et al.*, 2020).

CONCLUSION

Validation results of the proposed method (QuEChERS extraction with c- SPE cleanup) procedures in combination with the GC-ECD system for the analysis of multi-class pesticide residues in vegetable samples showed that the method has high linearity, repeatability, and sensitivity. The method was successfully verified and applied for the analysis of fifteen multi-class pesticides in four kinds of vegetable real samples. The verification results for all commodities examined showed that the method quantification limits for all target pesticides were below the codex and EU cited MRLs which reflects the sensitivity and suitability of the method for the intended application.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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