

Development and validation of modified quenchers - GC/MS method for simultaneous determination of current-use pesticide residues in soil

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Abstract. Pesticide residues in soil pose potential risks to ecosystems and human health, necessitating reliable and efficient analytical methods for their determination. This study developed and validated a modified QuEChERS-based extraction procedure combined with gas chromatography-mass spectrometry (GC/MS) for the simultaneous analysis of eight commonly used pesticides in soil, including herbicides, insecticides, and fungicides. Key extraction parameters were optimised for soil matrices, focusing on solvent composition and primary-secondary amine (PSA) clean-up levels. Optimal performance was achieved with 1% acetic acid in acetonitrile and 25 mg PSA per 1 mL of extract. The method was validated according to European Commission SANTE guidelines, demonstrating good linearity ($R^2 > 0.99$), low limits of detection (3–8 µg/kg), acceptable recoveries (69–114%), and satisfactory repeatability (RSD < 13%). The validated procedure was successfully applied to agricultural soil samples from central Vietnam, where residues of pretilachlor, fluazifop-p-butyl, and trifloxystrobin were detected. The results confirm that the proposed method is reliable, cost-effective, and suitable for routine monitoring of pesticide residues in soil.

Keywords: QuEChERS, pesticides, dispersive solid-phase extraction, soil

1 Introduction

In modern agriculture, pesticides have played a crucial role in increasing crop productivity and ensuring food security worldwide [1]. However, the intensive overuse of these chemicals has led to their widespread occurrence in various environmental compartments, particularly soils, which act as sinks for pesticide residues [2]. Once transported into soil, pesticides may undergo complicated pathways, including adsorption, degradation, leaching, volatilisation, and bioaccumulation, depending on their physicochemical properties and soil characteristics [3, 4]. Pesticide residues in soils would pose serious

risks to soil ecosystems, groundwater quality, and human health [5].

Determination of pesticide residues in soils is therefore essential for evaluating environmental risks and assessing compliance with regulatory limits. However, the analysis of pesticides in soil remains challenging because of the complex nature of the soil matrix, which contains varying proportions of organic matter, clay minerals, water, and biota [6]. These components can strongly interact with pesticide molecules, thereby interfering with their extraction and instrumental detection.

Over the past decades, a wide range of analytical methods has been developed for the determination of pesticide residues in soil. Some well-known extraction techniques include Soxhlet extraction, liquid-liquid extraction (LLE), ultrasonic-assisted extraction (UAE), pressurised liquid extraction (PLE), microwave-assisted extraction (MAE), supercritical fluid extraction (SFE) [6], followed by the chromatographic qualitative and quantitative techniques. Nevertheless, each of these methods has advantages and drawbacks, such as long working time, large volumes of required organic solvents, expensive instrumentation, and complicated optimisation procedures.

With the development of science and principles of green chemistry, priority criteria of a sustainable analytical method should be environmentally friendly, fast, cost-effective, simple, and capable of simultaneously determining a large number of pesticide residues in complex matrices. In this context, the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) extraction method has been introduced by Anastassiades et al. in 2003 for pesticide residue analysis in fruits and vegetables [7]. QuEChERS has since been widely adapted and validated [8, 9] or modified [10] for various food and soil matrices. The fundamental principle of the QuEChERS method involves a single-step extraction by using acetonitrile, followed by phase separation induced by the addition of inorganic salts, assisted by centrifuging and shaking, and a dispersive solid-phase extraction (d-SPE) clean-up step employing PSA (Primary-Secondary Amine), C18, or activated carbon to remove co-extracted matrix interferences. This simple, yet efficient approach significantly reduces sample preparation time and solvent consumption compared with traditional extraction techniques, aligning well with the principles of green analytical chemistry [7, 9].

Numerous studies have successfully applied QuEChERS-based methods for the simultaneous determination of pesticide residues in food [11, 12] or soils [13, 14]. Notably, Lesueur et al. [15] compared the QuEChERS method with three other extraction techniques: PLE, UAE and European Standards DIN EN 12393. Only the QuEChERS and UAE methods for analysing 24 pesticides allowed for the recovery of all analytes.

Despite these advances, this QuEChERS technique has not been widely applied in Vietnam. Current official methods on pesticide analysis primarily utilise traditional extraction processes, such as TCVN 8320:2010 and TCVN 8319:2010. A recently released TCVN 12848:2020 introduced QuEChERS extraction for the analysis of multi-residue pesticides in agricultural products of the plant origin. However, so far, there has been no official method (TCVN) employing QuEChERS, dealing with the soil matrix. Some laboratories in Vietnam started developing and modifying a QuEChERS-based method for the simultaneous analysis of pesticides in vegetables or soil [16, 17], with suitable accuracy and detection capacity. Most of these studies, yet, focus on one or a few target pesticide groups, mainly organophosphorous, carbamate, or neonicotinoid. In other words, the target pesticides in these studies are outside the broad range of pesticide types. Meanwhile, the pesticides currently used on the market are very diverse, belonging to numerous groups and types, with various physicochemical properties.

Based on the above considerations, we designed a procedure to apply the QuEChERS technique in combination with gas chromatography-mass spectrometry (GC/MS) for the simultaneous determination of some currently used pesticide residues in soil.

2 Methods

2.1 Reagents and materials

The chemicals used include toluene and acetonitrile solvents of chromatographic grade from J. T. Baker (USA), PSA, C18, and CH₃COONa·3H₂O adsorbents from Sigma Aldrich (USA), and anhydrous MgSO₄ of analytical grade from J. T. Baker (USA).

Certified standards (Sigma Aldrich, USA) are eight pesticides, namely, acetochlor,

chlorpyrifos, fipronil, pretilachlor, fluazifop-p-butyl, trifloxystrobin, tebuconazole, cypermethrin, along with the internal standard pyrene-D10 and surrogate δ-HCH. These eight pesticides are widely used in agriculture in Hue City (based on farmer interviews in Huong Tra and Quang Dien, not presented here), and can be analysed by using gas chromatography. Some physicochemical and toxicity characteristics of these eight pesticides are presented in Table 1.

Table 1. General physicochemical and toxicity characteristics of eight target pesticides [18]

Pesticides	Group	Melting point (°C)	Vapor pressure (mPa, 25 °C)	Solubility in water (mg/L, 20 °C)	logK _{ow}	Toxicity**
Tebuconazole	Triazole	105	1.30×10 ⁻³	36	3.7	II
Cypermetherin	Pyrethroid	42	6.78×10 ⁻³	0.009	5.6	II
Fipronil	Phenylpyrazole insecticide	203	0.002	3.78	3.75	II
Pretilachlor	Chloroacetanilide herbicide	-20	0.133	500	4.08	III
Acetochlor	Chloroacetamide herbicide	10.6	*2.2×10 ⁻²	282	4.14	III
Fluazifop-p-butyl	Aryloxyphenoxypr- opionate herbicide	-46	0.12	0.93	4.5	III
Chlorpyrifos	Organophosphate	42	*1.43	1.05	4.7	II
Trifloxystrobin	Strobilurin fungicide	72.9	3.40×10 ⁻³	0.61	4.5	III

*vapor pressure at 20 °C

** Toxicity: I – highly toxic; II – moderately toxic; III – slightly toxic (WHO classification) [19]

2.2 GC/MS analysis

The target pesticides were qualitatively and quantitatively analysed by using a triple quadrupole GC-MS system (GC/MS TQ8040, Shimadzu, Japan) equipped with a DB-5MS UI capillary column (60 m × 0.25 mm × 0.25 μm, Agilent, USA). Helium (99.9995%) was used as the carrier gas at a flow rate of 2.9 mL/min. The injection volume was 1 μL, and the analysis was performed in the splitless mode. Data acquisition

was carried out in the selected ion monitoring (SIM) mode. The injector and interface temperatures were set at 250 °C. Electron ionisation (EI) was operated at 70 eV with a detector voltage of 1.2 kV. The oven temperature program started at 70 °C (1 min), ramped to 180 °C at 10 °C/min, to 195 °C at 5 °C/min, then to 219 °C at 8 °C/min (held for 5 min), and finally to 280 °C at 10 °C/min (held for 10 min). The total run time was 42 min. Retention times and characteristic ions of the analysed pesticides are listed in Table 2, and a typical chromatograph is illustrated in Fig. 1.

Table 2. Retention times and target and reference ions of the studied pesticides* *Surrogate*; ** *internal standard*

No	Coumpounds	Retention times (min)	Target ions (m/z)	Reference ions (m/z)
1	δ -HCH*	17.85	181	219; 183
2	Acetochlor	20.73	188	88; 97
3	Chlorpyrifos	22.83	59	146; 162
4	Fipronil	24.57	367	213; 369
5	Pyrene-D10**	27.02	212	208; 106
6	Pretilachlor	27.84	162	238; 176
7	Fluazifop-p-butyl	29.65	282	254; 338
8	Trifloxystrobin	32.03	116	131; 59
9	Tebuconazole	33.13	125	70; 250
10	Cypermethrin	38.0–38.8	163	181;165

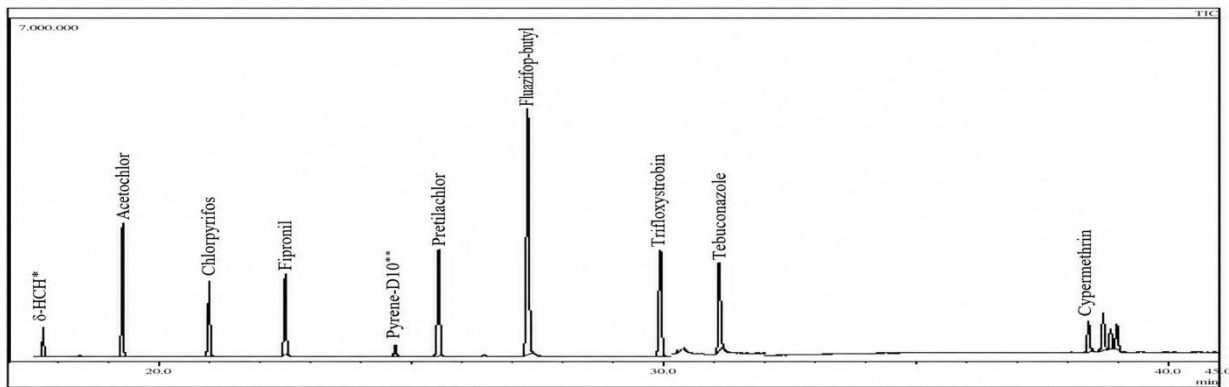


Fig. 1. Chromatogram of a mixture of eight target pesticides, surrogate, and internal standard at a concentration of 500 $\mu\text{g/L}$
 δ -HCH: *Surrogate*, Pyrene-D10: *internal standard*

2.3 QuEChERS-based modification for soil matrix

This study aimed to adapt and optimise the QuEChERS sample preparation method, originally proposed in AOAC 2007.1 for food matrices, for the simultaneous analysis of pesticide residues in soil. Briefly, 15 g of homogenised soil was extracted with 15 mL acetonitrile containing 1% acetic acid, followed by salt-induced partitioning with MgSO_4 and sodium acetate, and cleaning-up with MgSO_4 and primary-secondary amine (PSA). After vortexing and centrifugation, the cleaned extract was collected for instrumental analysis [20]. Based

on a preliminary literature review, we optimised key extraction parameters for soil matrices with three extraction solvents (15 mL of 1% acetic acid in acetonitrile, 15 mL of acetonitrile, and 15 mL of 1% formic acid in acetonitrile) and four PSA levels (0, 25, 50, and 75 mg). The blank soil samples collected at Hue University of Sciences and confirmed to be free of the target pesticides were spiked with 500 $\mu\text{g/kg}$ of analytes and surrogates. Extraction recoveries were used to evaluate the method performance and select the optimal conditions.

2.4 Method evaluation

The analytical method was validated in accordance with the European Commission guidelines (SANTE 11312/2021 v2026) [21], including evaluation of calibration linearity, limits of detection (LOD) and quantification (LOQ), accuracy, and repeatability. Calibration curves were established at seven concentration levels (10–1000 µg/L) by using external calibration with an internal standard in toluene. LOD and LOQ were determined from a signal-to-noise ratio (S/N), defined as $S/N \geq 4$ and $LOQ = 3 LOD$, respectively. Method accuracy and precision were assessed by analysing spiked residential soil samples at two concentration levels (25 and 200 µg/kg), with acceptable recoveries ranging from 70 to 120% and relative standard deviations (RSD) not exceeding 20%. A surrogate standard (δ -HCH) was added at the beginning of the procedure to monitor procedure performance, and samples with surrogate recoveries outside the acceptable range (70 to 120%) were excluded from further evaluation.

2.5 Soil sampling

Six soil samples in agricultural areas in Hue City and Phu Yen province were collected (this study aimed to test the applicability of the developed method; the sampling locations are not shown). At each sampling site, surface soil samples were collected from the 0–20 cm layer by using a clean stainless-steel auger. Three subsamples were taken within a defined plot and homogenised to form one composite sample. The collected samples were placed in cleaned aluminum foil, labeled, and

transported to the laboratory under cooled conditions. In the laboratory, the soil samples were air-dried at ambient temperature; surface debris and plant residues were removed and sieved through a 2-mm mesh prior to analysis.

2.6. Data statistical analysis

The chromatographic results were processed by using LabSolutions software (GCMS Solution-Shimadzu). Experimental data were processed by using statistical methods in Excel 2019. The non-parametric Kruskal-Wallis test was applied to compare differences between groups with statistical significance set at p less than 0.05.

The concentration unit ($\mu\text{g}\cdot\text{kg}^{-1}$) was converted from $\mu\text{g}\cdot\text{L}^{-1}$, as obtained from the calibration curve, by using the following equation:

$$C\left(\frac{\mu\text{g}}{\text{kg}}\right) = C_x \times \frac{V}{m}$$

where C_x is the measured concentration in the extract ($\mu\text{g}\cdot\text{L}^{-1}$); V is the final extract volume (L); m is the sample mass (kg).

3 Results and discussion

3.1 Calibration curves of studied compounds

The calibration curve equations (seven calibration points from 10 to 1000 µg/L in toluene) and the corresponding coefficient of determination R^2 of the studied substances are presented in Table 3. The results in Table 3 show that all the calibration curves of the studied pesticides yielded high determination coefficients ($R^2 > 0.99$) in the concentration range from 10 to 1000 µg/L.

Table 3. Calibration curve equations and coefficient of determination R^2 of eight studied pesticides and surrogate

STT	Compounds	Calibration curves	R^2
1	* δ -HCH	$y = 0.0057 \times x - 0.0129$	0.9991
2	Acetochlor	$y = 0.0059 \times x - 0.0413$	0.9985
3	Chlorpyrifos	$y = 0.0026 \times x - 0.0386$	0.9994

STT	Compounds	Calibration curves	R ²
4	Fipronil	$y = 0.0050 \times x - 0.1364$	0.9984
5	Pretilachlor	$y = 0.0150 \times x - 0.3039$	0.9987
6	Fluazifop-p-butyl	$y = 0.0122 \times x - 0.1041$	0.9978
7	Trifloxystrobin	$y = 0.0067 \times x - 0.1225$	0.9980
8	Tebuconazole	$y = 0.0060 \times x - 0.1403$	0.9980
9	Cypermethrin	$y = 0.0025 \times x - 0.3319$	0.9982

* surrogate

3.2 Selection of extraction solvent and PSA amount

Selection of extraction solvent

Based on the original methods for pesticide analysis in foods proposed by AOAC 2007.1, demonstrated in Section 2.3, we tested three extraction solvents, namely, DM1: 15 mL of 1% acetic acid in acetonitrile, DM2: 15 mL of

acetonitrile, and DM3: 15 mL of 1% formic acid in acetonitrile. The results are demonstrated in Table 4. The results reveal that the recovery rate of the pesticides extracted with DM1 (1% acetic acid in acetonitrile) was very good, ranging from 87.0% (acetochlor) to 113.9% (pretilachlor) compared with other solvent systems. Based on these results, we selected 1% acetic acid in acetonitrile as the optimal solvent for further experiments.

Table 4. Effect of extraction solvent type on sample extraction efficiency

No	Compounds	Spiked concentration (µg/kg)	Measured concentration (µg/kg)			Recovery (%)		
			DM1	DM2	DM3	DM1	DM2	DM3
1	δ-HCH (*)	500	462.6	126.1	325.9	92.5	25.2	65.2
2	Acetochlor	500	434.8	163.8	381.6	87.0	32.8	76.3
3	Chlorpyrifos	500	474.5	193.2	321.0	94.9	38.6	64.2
4	Fipronil	500	543.7	308.7	454.4	108.7	61.7	90.9
5	Pretilachlor	500	569.7	257.8	520.0	113.9	51.6	104.0
6	Fluazifop-butyl	500	517.7	227.4	454.5	103.5	45.5	90.9
7	Trifloxystrobin	500	562.9	254.7	504.1	112.6	50.9	100.8
8	Tebuconazole	500	549.5	295.1	516.6	109.9	59.0	103.3
9	Cypermethrin	500	467.5	298.7	386.5	93.5	59.7	77.3

* surrogate

Selection of PSA amount

Four levels of PSA amount were tested, namely, 0, 25, 50, and 75 mg. The extraction efficiency is presented in Table 5.

The data in Table 5 show that the amount of PSA added significantly affected the recovery rate. Without PSA, the recovery rate of the method was really low (mostly <50%). With increasing the PSA levels from 0 to 50 mg, the recovery rate gradually increased, with an insignificant difference between

25 mg and 50 mg ($p > 0.05$). However, when the PSA level increased to 75 mg, the recovery rate began to decrease substantially ($p < 0.05$). We believe that PSA in large amounts may adsorb the

pesticides, reducing the recovery rate. However, further research is needed to verify this hypothesis. Therefore, to save costs, we chose an appropriate PSA level of 25 mg per 1 mL of extract.

Table 5. Effect of PSA amount on extraction efficiency

No	Compounds	Spiked concentration (µg/kg)	Measured concentration (µg/kg)				Recovery (%)			
			0 mg PSA	25 mg PSA	50 mg PSA	75 mg PSA	0 mg PSA	25 mg PSA	50 mg PSA	75 mg PSA
1	δ-HCH (*)	500	203.7	397.2	429.6	232	40.7	79.4	85.9	46.4
2	Acetochlor	500	311.3	463.2	470.1	212.8	62.3	92.6	94.0	42.6
3	Chlorpyrifos	500	198.8	426.7	414.5	152.5	39.8	85.3	82.9	30.5
4	Fipronil	500	157.5	399.9	412.8	370.6	31.5	80.0	82.6	74.1
5	Pretilachlor	500	270.0	434.7	407.7	320.1	54.0	86.9	81.5	64.0
6	Fluazifop-p-butyl	500	156.6	527.5	485.9	370.4	31.3	105.5	97.2	74.1
7	Trifloxystrobin	500	232.1	451.4	421.1	340.6	46.4	90.3	84.2	68.1
8	Tebuconazole	500	309.8	393.7	379.8	252.2	62.0	78.7	76.0	50.4
9	Cypermethrin	500	290.7	410.5	423.8	380.6	58.1	82.1	84.8	76.1

*surrogate

3.3 Evaluation of analytical procedure

Limit of detection (LOD) and limit of quantification (LOQ) of the method

Based on signal-to-noise (S/N) ratios, we determined the limits of detection (LOD) and quantification (LOQ) of the target pesticides (using

blank soil matrices spiked with increased concentration until $S/N \geq 4$). In this study, an S/N threshold greater than or equal to 4, rather than the conventional greater than or equal to 3, was applied to ensure that the LOD and LOQ accurately reflect analytical performance in real soil samples. Results are shown in Table 6.

Table 6. Limit of detection (LOD) and limit of quantification (LOQ) of the method

No	Compounds	S/N	LOD (µg/kg)	LOQ (µg/kg)
1	Acetochlor	7.12	5.0	15.0
2	Chlorpyrifos	6.45	5.0	15.0
3	Fipronil	6.87	5.0	15.0
4	Pretilachlor	7.74	3.0	9.0
5	Fluazifop-p-butyl	7.68	3.0	9.0
6	Trifloxystrobin	6.96	5.0	15.0
7	Tebuconazole	5.64	8.0	24.0
8	Cypermethrin	5.00	8.0	24.0

The results show that the method yielded low detection and quantification limits for the studied pesticides, with LODs ranging from 3 to 8 µg/kg, corresponding to LOQs from 15 to 24 µg/kg. These results are almost identical to those published elsewhere. [14, 17, 22].

Method accuracy and repeatability

The accuracy and repeatability of the method were evaluated by analysing spiked samples at two fortified levels: a LOQ level (25 µg/kg) and a medium level (8–10 times LOQ) (200 µg/kg) with

three replicates for each level to calculate the recovery (Rev%) and relative standard deviation (RSD%). The results are presented in detail in Table 7. The results in Table 7 show that the recovery rates met the AOAC and SANTE guidelines [21, 23]. Recovery rates reached 69–114% for the standard addition concentration of 25 µg/kg and 83–99% for the standard addition concentration of 200 µg/kg. The method also achieved good repeatability, specifically, RSD < 13%, which is smaller than the EU’s permissible limit of RSD < 20% [21].

Table 7. Method recovery and repeatability

No	Compounds	Measured concentration (µg/kg)			Rev%			Average recovery (%)	RSD (%)
25 µg/kg fortified level									
1	*δ-HCH	18.9	22.1	20.9	75.6	88.4	83.6	82.5	6.5
2	Acetochlor	16.7	16.6	18.3	66.8	66.3	73.2	68.8	3.9
3	Chlorpyrifos	23.8	19.2	17.6	95.0	80.0	70.4	80.8	12.9
4	Fipronil	28.3	30.2	26.3	113.3	120.8	105.3	113.2	7.8
5	Pretilachlor	26.5	29.1	22.8	106.1	116.6	91.0	104.6	12.8
6	Fluazifop-p-butyl	18.5	15.9	21.7	73.9	63.4	86.8	74.7	11.7
7	Trifloxystrobin	23.7	24.0	20.7	94.7	95.9	82.8	91.1	7.3
8	Tebuconazole	28.1	28.1	29.6	112.4	112.2	118.2	114.4	3.46
9	Cypermethrin	27.6	28.1	23.1	110.5	112.5	92.3	105.1	11.0
200 µg/kg fortified level									
1	*δ-HCH	159.1	171.2	185.4	79.5	85.6	92.7	85.9	6.6
2	Acetochlor	170.7	180.8	151.2	85.3	90.4	75.6	83.8	7.5
3	Chlorpyrifos	185.4	181.2	173.2	92.7	90.6	86.6	90.0	3.1
4	Fipronil	192.8	184.6	216.8	96.4	92.3	108.4	99.0	8.4
5	Pretilachlor	186.6	177.4	223.6	93.3	88.7	111.8	97.9	12.2
6	Fluazifop-p-butyl	173.2	171.2	187.4	86.6	85.6	93.7	88.6	4.4
7	Trifloxystrobin	190.2	184.4	179.2	95.1	92.2	89.6	92.3	2.8
8	Tebuconazole	164.4	162.8	171.2	82.2	81.4	85.6	83.1	2.2
9	Cypermethrin	160.5	173.4	164.7	80.3	86.7	82.4	83.1	4.0

* surrogate

From these results, we propose a procedure for the simultaneous analysis of some currently used pesticides in the soil matrix, as shown in Fig. 2.

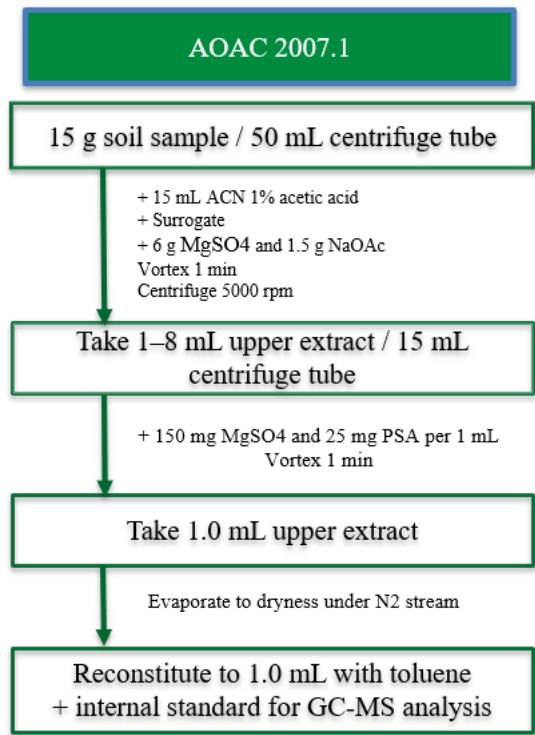


Fig. 2. Simultaneous analysis procedure for pesticide residues in soil

3.4 Application of analytical procedure to agricultural soil samples

The procedure was applied to analyse six soil samples in Hue City and Phu Yen Province. The

results (Table 8) revealed the residues of pretilachlor, fluazifop-p-butyl, and trifloxystrobin in the collected soil, demonstrating the potential risk of pollution and environmental hazards in agriculture in the area.

4 Conclusion

This study successfully adapted and optimised the QuEChERS extraction method, originally developed for food matrices, for the simultaneous determination of multiple pesticide residues in soil by using GC/MS for quantification. The optimisation of the extraction solvents and PSA cleaning-up significantly improved the recovery and analytical performance for diverse pesticide classes. The validated method met the requirements of European Commission SANTE guidelines, exhibiting good linearity, low detection limits, high accuracy, and acceptable repeatability. The method detected the presence of several pesticide residues, highlighting potential environmental contamination in the studied areas. Overall, the proposed QuEChERS-GC/MS procedure provides a simple, rapid, and reliable approach for routine pesticide residue monitoring in soil and represents a practical basis for further method standardisation and environmental assessment in Vietnam.

Table 8. Pesticide residues in soil samples collected in Hue City and Phu Yen province

No	Compounds	Pesticide residues (µg/kg)					
		ĐQ-H	ĐR-H	ĐL-H	ĐQ-PY	ĐR-PY	ĐL-PY
	Recovery of surrogate δ-HCH (*) (%)	79	88	81	80	92	76
1	Acetochlor	ND	ND	ND	ND	ND	ND
2	Chlorpyrifos	ND	ND	ND	ND	ND	ND
3	Fipronil	ND	ND	ND	ND	ND	ND
4	Pretilachlor	ND	ND	22.9	ND	ND	26.0
5	Fluazifop-p-butyl	26.8	37.0	ND	ND	ND	ND

No	Compounds	Pesticide residues (µg/kg)					
		DQ-H	DR-H	DL-H	DQ-PY	DR-PY	DL-PY
	Recovery of surrogate δ-HCH (*) (%)	79	88	81	80	92	76
6	Trifloxystrobin	64.1	37.8	ND	30.6	28.5	ND
7	Tebuconazole	ND	ND	ND	ND	ND	ND
8	Cypermethrin	ND	ND	ND	ND	ND	ND

(*) surrogate. Values are expressed as recovery (%); ND: Not detected

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