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Rapid Determination of 54 Kinds of Pesticide Residues in Vegetables by PTV-GC-MS/MS

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Introduction

In this study, we established a rapid detection method of 54 kinds of pesticide residues in vegetables, the analysis method of using QuEChERS pretreatment method, combining with the programmed temperature vaporization gas chromatography triple quadrupole tandem mass spectrometry. The programmed temperature vaporization (PTV) can volatilize acetonitrile, in order to improve the chromatographic column durability. In addition, compared with the single-quad mass spectrometry, triple quadrupole tandem mass spectrometry has characteristics of strong anti-interference ability, high sensitivity and high throughput of ion transport efficiency, so that it can complete the accurate identification of target compounds under complex matrix. According to the results, the proposed method is ideally suited for the routine monitoring and rapid screening of pesticide residues in vegetables.

Experimental

Sample pretreament

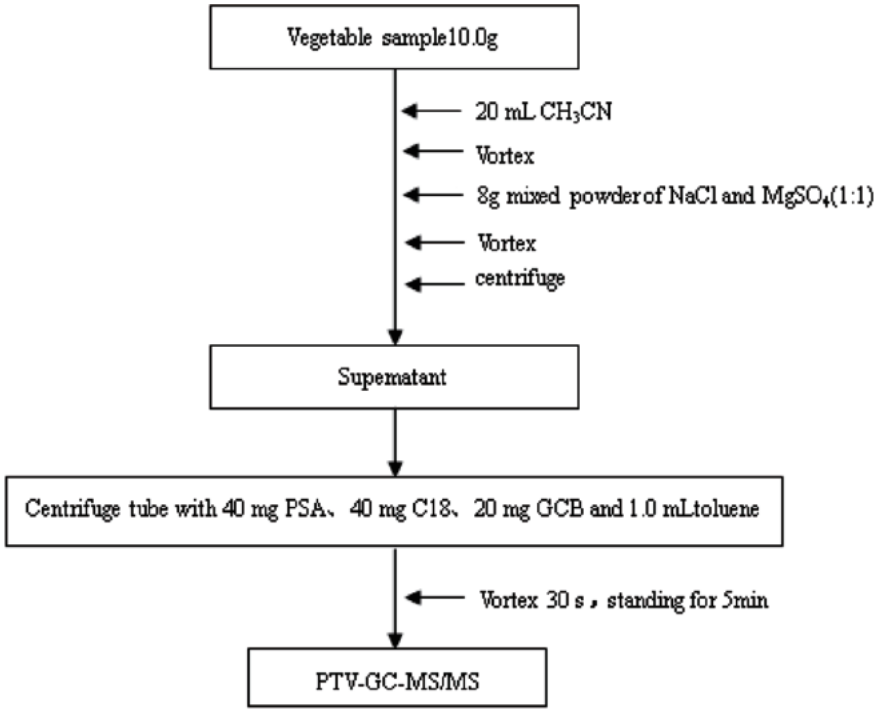

Figure 1 Schematic Flow diagram of the sample preparation

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Instrument and Method

Analytical Conditions	
Instrument	: Shimadzu GCMS-TQ8040 (coupled with PTV)
Method	
Column	: Rxi-5 Sil ms (30 m×0.25 mm×0.25 µm)
Injector temperature program	: 65 ℃ (1 min)_(200 ℃/min)_250 ℃ (15 min)
Split valve program	: 0-0.9 min Split:20:1
	0.9-3.5 min Splitless
	3.5 min Split:20:1
Oven temperature program	: 40 ℃ (4 min)_(25 ℃/min)_125 ℃_(10 ℃/min)_300 ℃ (5 min)
Linear velocity	: 36.2 cm/sec
Injection volume	: 2 µL
Interface temperature	: 280 ℃
Ion Source temperature	: 200 ℃

Results

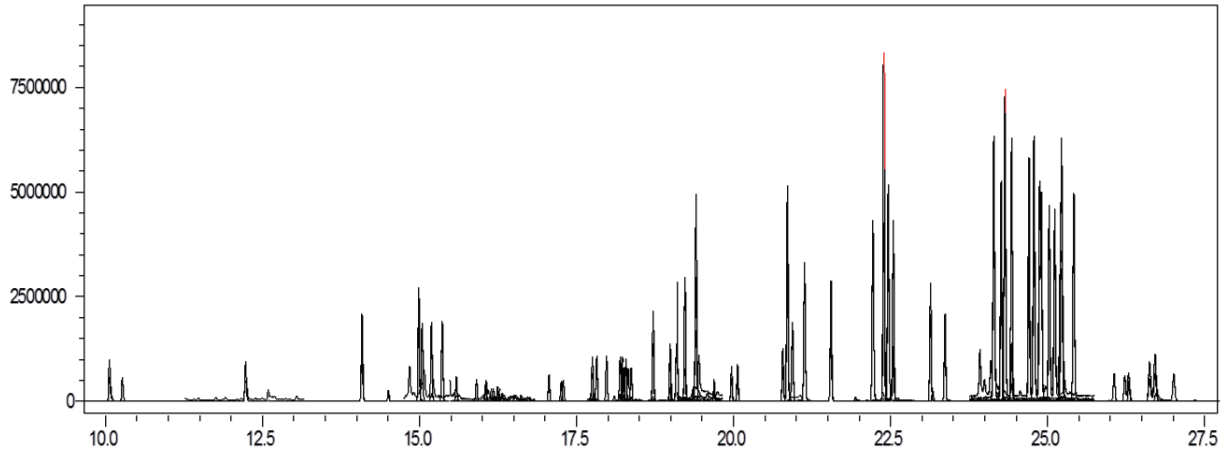

Figure 2 Chromatograms of MRM from mix standards

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Table 1 Average recoveries (RSD%) of 3 levels in Spinach, cowpea and chinese chives.

No.	Compound name	Correlation coefficient (R^2)	LOQ (μg/kg)	Spinach			Cowpea			Chinese chives		
				10 μg/kg	20 μg/kg	100 μg/kg	10 μg/kg	20 μg/kg	100 μg/kg	10 μg/kg	20 μg/kg	100 μg/kg
1	Methamidophos	0.999	4.0	90.5	101.5	86.5	83.1	111.0	86.3	88.6	101.6	91.5
2	Dichlorvos	0.999	4.0	97.9	98.5	89.0	99.6	120.8	95.7	83.2	93.8	98.5
3	Omethoate	0.998	2.0	90.6	103.9	84.8	100.1	111.0	96.7	98.6	96.1	93.1
4	Ethoprophos	0.986	2.0	107.4	105.9	91.7	88.3	112.4	96.7	99.5	92.3	90.9
5	Monocrotophos	0.998	2.0	98.8	103.5	86.8	92.4	105.8	97.9	100.9	91.6	91.2
6	Sulfotep	0.999	8.0	92.6	102.4	94.3	87.2	107.3	93.5	90.6	92.8	88.1
7	Phorate	0.999	8.0	110.1	99.8	93.5	89.8	109.7	98.6	88.8	104.7	92.4
8	alpha-HCH	0.999	8.0	100.	114.5	94.9	94.4	112.0	94.7	104.7	91.3	95.6
9	Dimethoate	0.999	8.0	116.2	112.7	93.9	94.4	112.2	97.1	98.6	90.6	92.5
10	beta-HCH	0.998	8.0	85.6	103.4	93.5	78.3	120.9	95.1	105.0	88.5	96.6
11	gamma-HCH	0.999	8.0	113.4	103.9	92.8	87.8	114.9	97.6	90.0	97.9	98.0
12	Terbufos	0.999	8.0	108.2	110.1	93.8	95.2	114.0	95.2	91.7	95.7	88.3
13	Quintozene	0.998	10.0	112.9	93.2	92.7	75.7	115.5	95.7	85.3	93.2	91.1
14	Fonofos	0.999	2.0	104.8	105.9	94.2	91.0	115.0	97.4	106.5	93.6	87.2
15	Pyrimethanil	0.991	4.0	100.0	109.2	95.5	83.3	104.3	97.3	106.6	85.5	94.5
16	Diazinon	0.998	10.0	87.0	121.4	94.3	87.9	98.6	92.0	82.3	84.5	88.8
17	delta-HCH	0.996	4.0	95.3	107.4	93.3	71.7	111.2	99.1	105.2	84.3	99.1
18	Vinclozolin	0.999	4.0	96.6	104.6	93.3	74.6	108.9	99.4	105.1	89.1	95.2
19	Parathion-methyl	0.997	2.0	115.4	101.3	87.9	82.4	112.6	94.9	102.8	90.9	86.2
20	Fenitrothion	0.998	2.0	83.4	104.3	87.4	77.0	116.2	98.1	90.8	92.5	91.9
21	Malathion	0.995	2.0	90.1	108.8	90.5	80.1	112.6	99.2	98.8	85.5	93.3
22	Fenthion	0.99	2.0	91.4	102.6	87.6	82.3	101.2	98.7	100.1	94.0	95.1
23	Chlorpyrifos	0.995	4.0	104.6	107.0	90.5	72.5	113.0	97.5	98.1	92.3	96.9
24	Parathion	0.999	2.0	106.9	104.3	87.9	85.4	106.0	96.1	99.9	100.9	88.7
25	Triadimefon	0.999	2.0	91.6	108.5	90.8	85.9	111.4	98.1	95.4	88.0	97.5
26	Dicofol deg.	0.998	2.0	96.7	106.4	88.8	91.9	109.1	99.2	104.9	96.1	97.3
27	Isocarbophos	0.999	4.0	93.7	110.7	91.2	83.3	99.1	97.7	101.2	88.3	89.6
28	Isofenphos-methyl	0.996	2.0	91.9	107.4	92.0	81.3	105.1	98.8	99.8	87.6	97.8
29	Phosfolan	0.998	4.0	95.9	87.9	88.3	108.2	94.4	100.2	83.7	105.4	88.0
30	Fipronil	0.998	2.0	97.5	104.2	85.3	101.9	102.4	99.0	106.5	91.6	90.0
31	Quinalphos	0.996	10.0	113.1	106.6	93.9	95.7	108.7	97.9	103.0	93.8	96.7
32	Phenthoate	0.998	2.0	93.0	105.7	93.8	80.8	104.9	98.2	101.2	90.6	98.1
33	Procymidone	0.999	2.0	99.5	101.6	89.1	90.8	104.6	96.9	94.7	90.3	95.5
34	Methidathion	0.999	2.0	123.4	110.6	92.2	99.7	104.0	97.3	106.6	92.4	96.4
35	Profenofos	0.999	2.0	98.4	103.7	88.8	99.5	102.8	99.2	102.0	93.0	95.0
36	Triazophos	0.999	2.0	100.4	102.4	91.2	100.0	97.9	99.4	100.4	92.1	97.3
37	Iprodione	0.999	2.0	96.1	107.6	85.6	98.2	102.3	102.3	94.3	90.0	91.9
38	Bifenthrin	0.994	2.0	96.4	104.2	93.0	106.5	104.4	98.8	98.8	90.2	98.1
39	Phosmet	0.998	2.0	97.8	102.4	91.1	104.5	106.2	99.5	88.3	81.6	97.7
40	Fenpropathrin	0.999	2.0	98.1	101.5	92.0	99.7	101.5	98.7	99.9	89.6	96.4
41	Phosalone	0.998	2.0	101.7	101.5	91.9	100.9	99.3	98.5	101.0	89.8	96.6
42	Pyridaben	0.998	2.0	101.7	105.4	92.9	104.9	101.1	98.1	102.3	91.5	97.1
43	Coumaphos	0.998	2.0	100.5	101.8	90.4	107.1	98.7	99.5	92.2	94.5	94.5
44	Phosphamidon	0.999	4.0	90.5	108.0	87.8	93.9	107.3	97.8	94.9	82.7	91.1
45	Endosulfan	0.999	10.0	99.6	93.6	98.1	96.6	111.6	100.9	100.4	78.7	96.7
46	Cyhalothrin	0.996	2.0	114.0	105.5	94.1	102.7	99.4	98.4	101.7	89.3	93.1
47	Permethrin	0.999	8.0	103.3	102.9	92.9	91.1	88.8	97.7	92.9	97.3	98.5
48	Cyfluthrin	0.997	8.0	102.1	93.8	90.4	100.1	97.3	98.9	98.5	101.6	93.2
49	Cypermethrin	0.997	2.0	103.9	97.9	93.3	103.0	99.3	97.2	94.7	89.4	91.0
50	Flucythrinate	0.996	2.0	104.6	100.0	93.2	101.3	107.3	97.9	100.6	91.0	92.4
51	Fenvalerate	0.996	2.0	97.6	102.6	90.9	97.7	99.3	99.6	94.5	90.0	93.7
52	Fluvalinate	0.993	2.0	99.2	96.4	91.5	98.9	103.0	95.9	97.9	92.9	87.8
53	Difenoconazole	0.997	2.0	115.5	102.7	92.3	95.8	103.7	95.9	96.8	94.6	87.6
54	Deltamethrin	0.993	2.0	116.0	92.9	91.9	92.5	110.2	90.8	102.7	95.1	82.7

*Average RSD (%) of recoveries are less than 10%

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Conclusion

The multi-residue method developed using PTV-GCMS-TQ8040 for the determination of pesticides in vegetable samples is simple, efficient and reliable. The performance of the method is very satisfactory with results meeting validation criteria. The method described here is found to have good practicability for routine residue analysis of pesticides in various vegetables.

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