

Research of Polychlorinated Biphenyls (PCBs) in Vegetables by GC-MS/MS

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Introduction

Polychlorinated biphenyls (PCBs), one of the most famous “dirtydozen” persistent organic pollutants (POPs) with carcinogenicity, teratogenicity and mutagenicity, are used to be produced and commercially used as mixtures. Even PCBs were banned by most countries as early as 1970s, they can still be detected in air, soil, water, sediment and biota at a global scale, even in remote areas such as the polar regions, deep seas and high mountains. In this study, we established a rapid detection method of 7 kinds of Polychlorinated biphenyls (PCBs) in vegetables, the analysis method of using QuEChERS pretreatment

method, combining with MRM mode of gas chromatography triple quadrupole tandem mass spectrometry which 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 the 7 kinds of Polychlorinated biphenyls (PCBs) in vegetables.

Experimental

Sample pretreatment

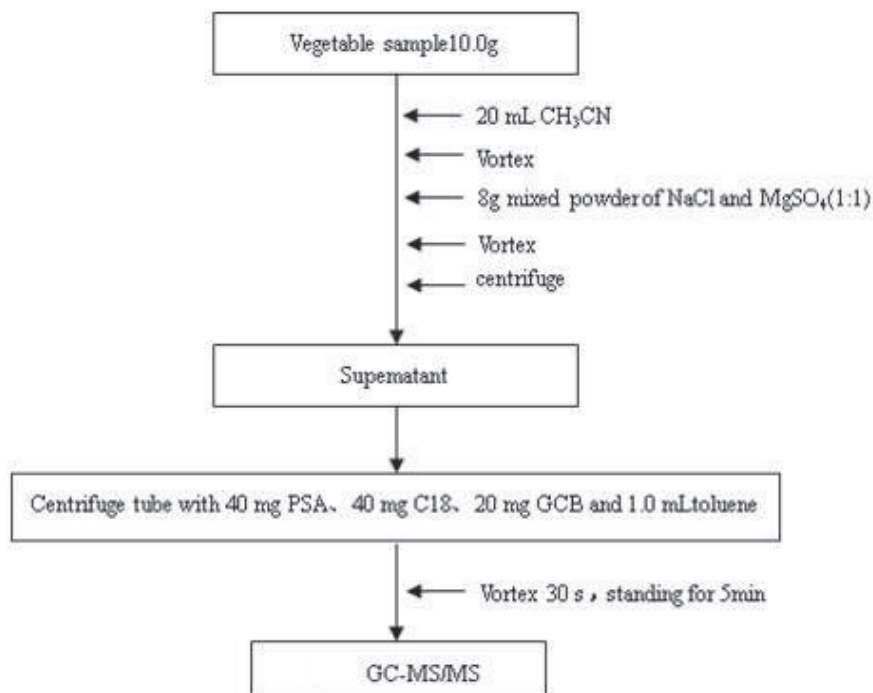


Figure 1 schematic Flow diagram of the sample preparation

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Instrument and Method

Instrument	
Shimadzu GCMS-TQ8040	
Method	
Column	: Rxi-5 MS, 30m×0.25mm×0.25µm
Column oven temp.	: 50 °C (1min)-25 °C/min-125 °C-10 °C/min-300 °C (3.5min)
Injection temp.	: 250 °C
Injection mode	: Splitless
Flow control mode	: Linear velocity (47.2cm/sec)
CID gas	: Argon
Detector voltage	: Tuning result+0.6kv
Interface temp.	: 250 °C
Ion source temp.	: 200 °C

Table 1 GCMSMS parameters for PCBs

NO	Compound	Retention time	Quantitation ions	CE	Qualification ions	CE
1	PCB28 (2,4,4'-Trichlorobiphenyl)	11.77	256.00>186.10	27	256.00>151.10	36
2	PCB52 (2,2',5,5'-Tetrachlorobiphenyl)	12.44	292.00>221.90	27	292.00>219.90	27
3	PCB101 (2,2',4,5,5'-Pentachlorobiphenyl)	14.11	326.00>255.90	27	326.00>253.90	27
4	PCB118 (2,3',4,4',5-Pentachlorobiphenyl)	15.27	326.00>255.90	30	326.00>253.90	30
5	PCB138 (2,2',3,4,4',5'-Hexachlorobiphenyl)	15.69	360.00>289.80	30	360.00>287.80	30
6	PCB153 (2,2',4,4',5,5'-Heptachlorobiphenyl)	16.19	360.00>289.80	30	360.00>287.80	30
7	PCB180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)	17.40	394.00>323.80	27	394.00>321.80	30

Results

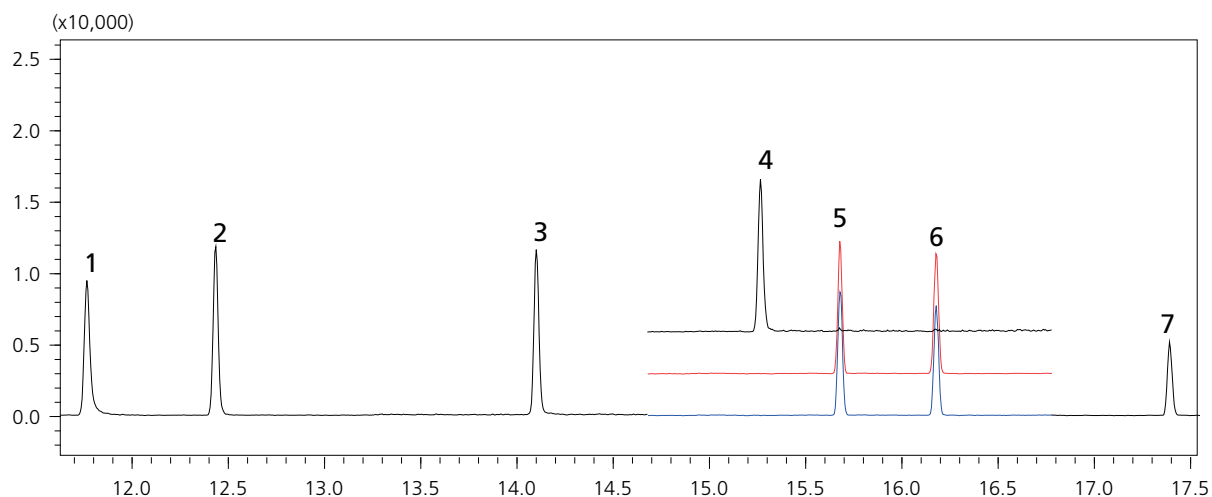


Figure 2 Chromatograms of MRM from mix standards

The results of this research indicated that the relative coefficients of the 7 kinds of PCBs ranged from 1 to 500 $\mu\text{g/L}$ were above 0.999. Precision ($n=6$) of this method was measured by analyzing the sample at 1 $\mu\text{g/L}$. The overall RSDs of analysis were below 5%. The limit of detection (LOD; $S/N=3$) of most compounds were below 0.09 $\mu\text{g/L}$. Commercially available carrot, potato and

ginger were used for recovery test, spiked concentration was 10 $\mu\text{g/kg}$ and the recoveries were 72.0~108.2%, 76.2~110.3% and 93.9~120.6%. The developed method in this study was proved to be reliable and accurate, and permits rapid determination of PCBs can be easily applied for quality control of vegetables.

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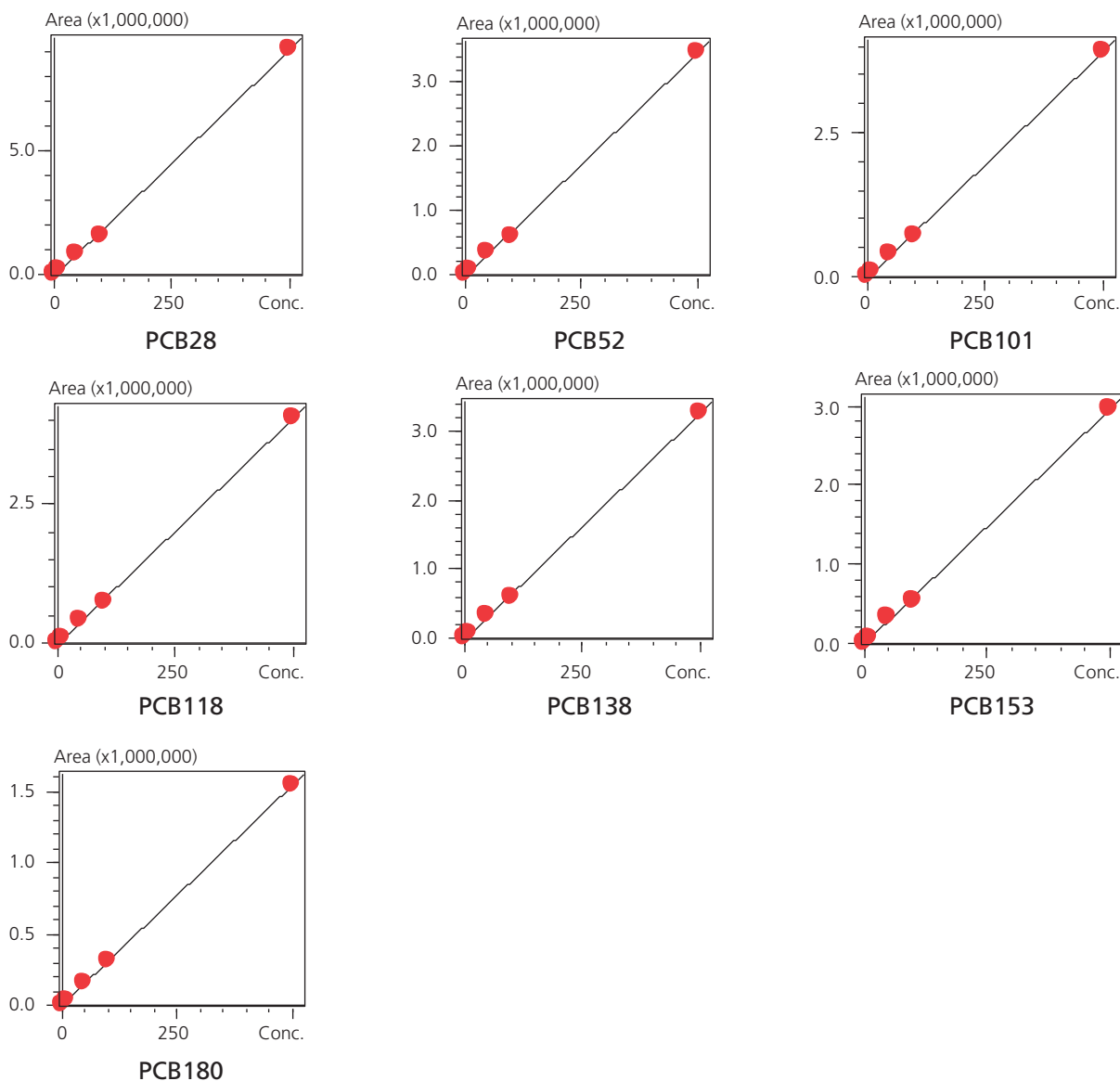


Figure 3 Calibration curve

Table 2 Relative coefficients, limit of detection (LOD, S/N=3) and recovery of the 7 kinds of PCBs

No.	Compound	Relative coefficients	LOD (µg/L)	Recovery		
				Carrot	Potato	Ginger
1	PCB28	0.9990	0.01	79.0	85.9	103.7
2	PCB52	0.9991	0.03	72.0	80.7	99.1
3	PCB101	0.9991	0.02	84.5	94.7	112.0
4	PCB118	0.9991	0.01	92.3	95.7	120.6
5	PCB138	0.9994	0.07	108.2	110.3	119.8
6	PCB153	0.9995	0.09	95.6	98.8	117.0
7	PCB180	0.9997	0.06	75.5	76.2	93.9

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Table 3 Precision of PCBs (1 ng/mL each, n=6)

No.	Compound	Area						RSD (%)
		1	2	3	4	5	6	
1	PCB28	20882	21137	21076	21016	20311	21474	1.83
2	PCB52	13494	13944	13702	13926	13942	13706	1.33
3	PCB101	11428	11678	11653	11397	11349	11558	1.21
4	PCB118	13891	14251	13501	13602	14160	14300	2.45
5	PCB138	5965	5993	5557	5777	5800	5861	2.70
6	PCB153	5469	5470	5063	5148	5364	5091	3.58
7	PCB180	1141	1082	1044	1061	1109	1100	3.20

Conclusion

The method developed using GCMS-TQ8040 for the determination of polychlorinated biphenyls (PCBs) 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 polychlorinated biphenyls (PCBs) in various vegetables.

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