



# An application of new microwave absorption tube in non-polar solvent microwave-assisted extraction of organophosphorus pesticides from fresh vegetable samples

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## ABSTRACT

A new self-designed microwave absorption tube was used in microwave-assisted extraction of seven organophosphorus pesticides from four kinds of vegetable samples. The non-polar solvent was used as extraction solvent, and a new portable microwave extraction apparatus was used. By sealing graphite powder in glass tube, microwave absorption tube was made and used to heat samples directly. The extracts were directly analyzed by GC–MS without any clean-up process. The effects of some experimental parameters on extraction efficiency were investigated and optimized. 3.0 g of sample, 25 mL of hexane and three microwave absorption tubes were added in the microwave extraction vessel, the extraction was carried out under 425 W irradiation power at 70 °C for 8 min. The recoveries were in the range of 76.5–109.4% and the relative standard deviations were lower than 13.1%.

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## 1. Introduction

Organophosphorus pesticides (OPPs) are generally used as insecticides in a wide variety of crop. OPPs are used extensively in agriculture due to their relatively low cost, broad spectrum of activity and high efficiency on insects. Although the use of OPPs provides unquestionable benefits to farmers and enhances the antioxidant potential in plants [1], these pesticides are harmful and frequently found in soil and other environmental matrices. The effects of carcinogenic, genotoxic, and teratogenic of OPPs have been individually investigated [2–5]. The OPPs can cause irreversible phosphorylation of esterase in the central nervous system of human and act as cholinesterase inhibitor [6], which can cause risks to human health and life. So, it is necessary to develop effective methods for determination of OPPs in foods and environmental matrices.

A key aspect in the determination of OPPs is sample preparation. The sample preparation step before chromatographic analysis is considerable complex, waste and expensive [7]. Many routine pretreatment methods used in the determination of OPPs are often time consuming, labor intensive and large amount of organic solvent wasting. Additionally, more than one clean-up step are usually required. A wide variety of techniques have been employed to extract and purify pesticides from fruits and

vegetables, such as soxhlet extraction [8], liquid–liquid extraction [9], dispersive liquid–liquid micro-extraction [10], solid-phase extraction [11], solid-phase micro-extraction [12,13], matrix solid-phase dispersion [14,15], supercritical fluid extraction [16], sonication extraction [17], accelerated solvent extraction [18,19], microwave-assisted extraction (MAE) [20–23]. In MAE, a wide range of polar or mixed organic solvents are needed and frequently used as extraction solvent. However, non-polar solvents, such as ether and hexane, cannot used directly in MAE. When MAE was employed to extract non-polar compounds, some polar solvents had to be added to absorb microwave energy and heat sample system, which resulted in the complexity of extraction. In recent years, single non-polar solvent has been successfully used as extraction solvent in improved microwave-assisted extraction without adding any polar solvent [24]. Some kinds of carbon materials have recently been attracting more and more interest due to their excellent adsorption properties [25] and microwave absorbing capability [26–30]. These carbon materials were employed to heat samples and accelerate the extraction process. But in most of reported literature the materials were directly added into samples and could not be reused. Beside, a stirrer bar (Weflon) made by a synthetic of polytetrafluoroethylene and graphite was used in microwave-integrated Soxhlet extraction [31].

In this study, a new microwave absorption tube was designed and could be reused in MAE. By using microwave absorption tube, MAE was developed, and would be applied to the extraction of OPPs from vegetable samples with non-polar solvent only. Because polar solvent was not used, the polar interferents could hardly be

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**Table 1**  
Qualitative and quantitative data of seven OPPs.

Analytes	Retention time (min)	Main fragment ion (m/z)	Calibration curve <sup>a</sup>	Linear range (μg/kg)	r	LOD (μg/kg)	LOQ (μg/kg)
Phorate	9.842	260 <sup>b</sup> , 75, 121, 65	A = 604.21 c-358.11	1.7–170	0.9998	0.42	1.4
Diazinon	10.950	304 <sup>b</sup> , 137, 179, 93	A = 645.91 c-576.47	1.7–170	0.9997	0.40	1.3
Tolclofos-methyl	12.700	265 <sup>b</sup> , 125, 267	A = 2629.08 c-1722.97	0.85–85	0.9998	0.15	0.5
Malathion	13.825	173 <sup>b</sup> , 93, 125, 127	A = 1314.63 c-1603.19	1.7–170	0.9997	0.29	0.9
Chlorpyrifos	14.142	314 <sup>b</sup> , 258, 286	A = 1239.17 c-1268.16	3.3–330	0.9996	0.38	1.3
Fenthion	14.350	278 <sup>b</sup> , 125, 109, 169	A = 2652.51 c-3235.67	1.7–170	0.9995	0.42	1.4
Methidathion	17.467	145 <sup>b</sup> , 85, 93	A = 1345.26 c-1082.41	1.7–170	0.9995	0.54	1.8

<sup>a</sup> Where A and c are the peak area and concentration of the analytes, respectively.

<sup>b</sup> The ion for quantitative analysis.

involved, and the extract can be analyzed directly by GC–MS. Some experiment parameters, such as amount of microwave absorbing medium, kind of extraction solvent, solid-to-liquid ratio, extraction temperature and extraction time, were optimized, respectively. In addition, a portable microwave extraction apparatus was first used in this study.

## 2. Experimental

### 2.1. Chemicals and reagents

Seven OPPs including phorate, diazinon, tolclofos-methyl, malathion, chlorpyrifos, fenthion and methidathion, were purchased from National Institute of Metrology (Beijing, China), and the purity of OPPs is  $\geq 98\%$ . Stock solutions for the OPPs were prepared in hexane at 1000 μg/mL and stored at 4 °C. Working standard solutions were prepared daily by diluting the stock solution with hexane. Anhydrous sodium sulfate (99%), hexane, cyclohexane, isooctane, petroleum ether and carbon tetrachloride were of analytical grade and purchased from Beijing Chemical Factory (Beijing, China).

### 2.2. GC–MS analysis

A GC–MS system (GCMS-QP 2010 plus, Shimadzu, Kyoto, Japan) was used. Chromatographic separation was conducted with a DB-5MS capillary column (30 m × 0.25 mm I.D., film thickness of 0.25 μm, J&W Scientific, Folsom, CA, USA). Helium (purity  $\geq 99.999\%$ ) was used as carrier gas at a constant flow of 1.0 mL/min. The temperature program was set initially at 70 °C for 1 min to 180 °C at a rate of 25 °C/min, and then to 210 °C at a rate of 5 °C/min (held for 6 min), finally raised to 270 °C (held for 1 min). Injector temperature was maintained at 280 °C, and the injection volume was 1.0 μL in the splitless mode. The ion source, interface temperatures and electron impact ionization energy were 200 °C, 250 °C and 70 eV, respectively. The mass spectrometer was operated in the selective ion monitoring (SIM) mode and the characteristic ions are given in Table 1. Full-scan MS data were acquired

in the range of m/z 50–900 to obtain the fragmentation spectra of the analytes.

### 2.3. Vegetable samples

Fresh vegetable samples (cucumber, eggplant, cole, pepper) were purchased from local supermarket (Changchun, China). The vegetable samples were chopped and homogenized with food processor. 50 g of vegetable sample was weighed and spiked with working standard solutions. The spiked samples were stored for 24 h in the dark place at room temperature. The cucumber sample was used in the optimization of experimental conditions. All experiments were performed in triplicate.

### 2.4. Preparation of microwave absorption tube

In this paper, graphite powder was used as microwave absorbing medium. 0.4 g of graphite powder was filled in a glass tube ( $\varphi$  0.9 cm, L 3.0 cm, thickness 1.0 mm), and the tube was then sealed.

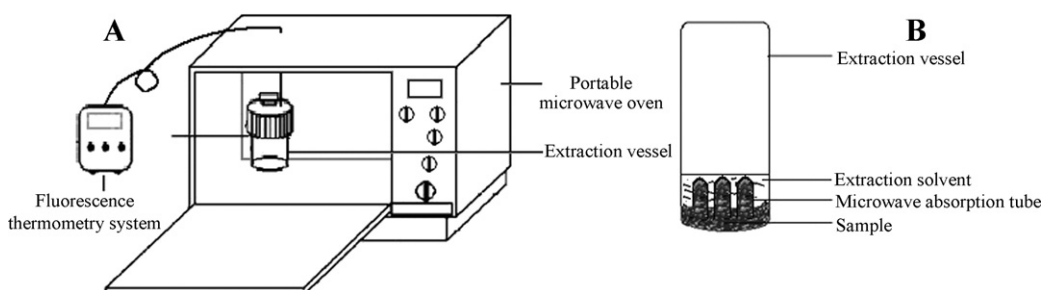
### 2.5. Portable microwave extraction apparatus

The portable microwave extraction apparatus (Fig. 1A) consists of a portable microwave oven (WBP-TP-660, WaveBox, USA), thermometry system, PTFE microwave closed-vessels (Fig. 1B) and microwave absorption tube. The microwave energy of the portable microwave oven can be continuously transmitted to the extraction vessel. Its maximum microwave output power is 425 W with 2460 MHz of microwave irradiation frequency.

A fluorescence thermometry system (FOTS-100A, Probing Technologies Inc, Canada) with the detecting temperature range from –40 to 200 °C was used to measure the temperature of sample system.

### 2.6. MAE

3.0 g of vegetable samples, 25 mL of hexane and microwave absorption tube were transferred into PTFE microwave closed-vessel. Under 425 W of microwave irradiation, MAE was hold at



**Fig. 1.** Portable microwave extraction system (A) and the extraction vessel (B).

70 °C for 8 min. After extraction, the extract obtained was concentrated to dryness in rotary evaporator at 30 °C. The residue was dissolved in 1 mL of hexane, filtrated through 0.22 μm membrane, and then directly analyzed by GC–MS.

### 3. Results and discussion

#### 3.1. Microwave absorption tube

Generally, non-polar solvents cannot be used as extraction solvents in MAE, because the solvents are not microwave absorbing compounds. Some kinds of polar solvents, such as acetone, methanol, ethanol and so on, were frequently used as microwave absorbing medium, even though so many undesired interferents would be simultaneously extracted [31–34]. In this study, the solid microwave absorbing medium was used to replace polar solvent when non-polar solvent was solely used in MAE.

##### 3.1.1. Kind of microwave absorbing medium

In this study, three kinds of microwave absorbing mediums, including graphite powder, active carbon powder and carbonyl iron powder [27], were used as filling materials in microwave absorption tube. The heating capabilities of the microwave absorption tubes were compared under the same microwave irradiation. It was observed that the heating capabilities of three microwave absorption tubes were similar, but the microwave absorption tube stuffed with active carbon powder and carbonyl iron powder were both insecure to burst frequently. Finally, graphite powder was chosen to be filling materials in microwave absorption.

##### 3.1.2. Microwave absorbing capability of microwave absorption tube

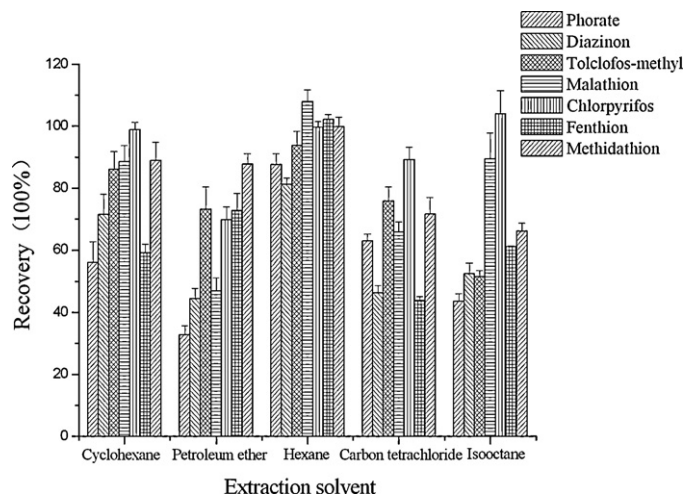
A comparison of microwave absorbing capability between microwave absorption tube with acetone was carried out. Under 425 W of microwave irradiation, it took about 50 s to heat 25 mL of hexane from 18 °C to 70 °C when three microwave absorption tubes (1.2 g of graphite powder) were used. Correspondingly, it took about 210 s when 25 mL of acetone was used. It is obvious that microwave absorbing capability of microwave absorption tube is much higher than that of acetone. It can be supposed that microwave absorption tube made by filling graphite powder would accelerate the MAE process.

#### 3.2. Optimization of MAE conditions

In MAE, many conditions can affect the extraction, such as amount of microwave absorbing medium, kind of extraction solvent, solid-to-liquid ratio, extraction temperature and extraction time. In this paper, the effects of MAE conditions on the extraction process and recoveries of OPPs were studied.

##### 3.2.1. Amount of microwave absorbing medium

Three microwave absorption tubes were filled with different quantities of graphite powder (0.2, 0.4 and 0.6 g), respectively, and the heating speeds of the tubes were compared under the same microwave irradiation. The experiment was performed on a household microwave oven (SANYO, China) equipped with a shielded non-invasive thermometry system (EXERGEN, Irt/c.01, USA) with a maximum microwave output power of 360 W. The temperatures of these microwave absorption tubes were measured with the infrared thermometry system, and the heating speeds of microwave absorption tubes filled with 0.4 and 0.6 g of graphite powder were both favorable. However, the size of microwave absorption tube filled with 0.6 g of graphite powder was too big to be completely immersed in the extraction solvent. So, 0.4 g of



**Fig. 2.** Effect of extraction solvent \*Extraction temperature, 70 °C; extraction time, 8 min; solid-to-liquid ratio, 3:25 (g/mL); number of microwave absorption tubes, three.

graphite powder was finally chosen to be the optimal amount in microwave absorption tube.

The number of microwave absorption tube is closely related to the heating speed of sample system. The effect of number of microwave absorption tubes on the heating speed of sample system (25 mL of hexane and 3.0 g of vegetable samples) were tested, and the results showed that the heating speed increased obviously with the increase of microwave absorption tube number. Considering that the size of PTFE vessel and the volume of the extraction solvent, three microwave absorption tubes were finally selected.

##### 3.2.2. Kind of extraction solvent

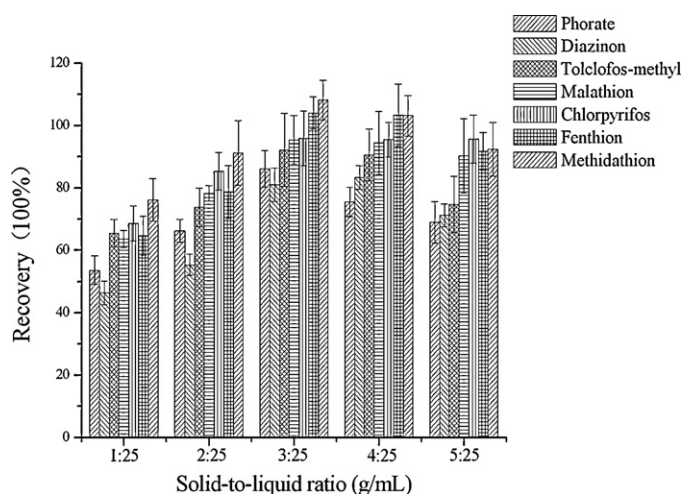
In this study, non-polar solvents, including hexane, cyclohexane, isooctane, petroleum ether and carbon tetrachloride, were tested, respectively. The experimental results were obtained in triplicate and are shown in Fig. 2. It can be seen that the recoveries of seven OPPs obtained using hexane as extraction solvent are the highest. After GC–MS analysis, the extract obtained with hexane was proved to be clean enough to be directly analyzed because few of polar interferent was detected. Therefore, hexane was chosen to be the optimal solvent for extraction of OPPs in MAE experiment.

##### 3.2.3. Solid-to-liquid ratio

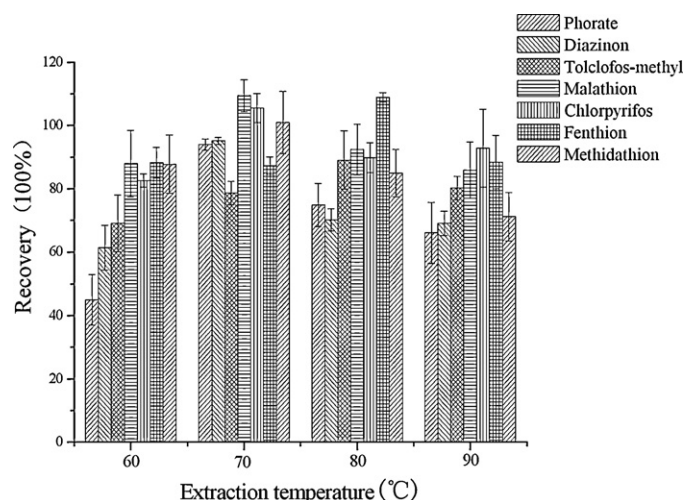
Several solid-to-liquid ratios were tested to investigate the influences on the recoveries of OPPs. In these tests, the volume of solvent was fixed at 25 mL in order to ensure the pressure of closed-vessel to be same. All the experiments were performed in triplicate. The results are shown in Fig. 3. Most of the recoveries of OPPs were the highest when the ratio was 3:25 (g/mL). Therefore, 3:25 (g/mL) was considered as the optimal solid-to-liquid ratio for the MAE process.

##### 3.2.4. Extraction temperature

The influences of extraction temperature on recoveries of OPPs were studied. Four different extraction temperatures (60, 70, 80 and 90 °C) were tested, respectively. The experimental results were obtained in triplicate and are shown in Fig. 4. It can be seen that the recoveries of OPPs are the highest when the extraction temperature is held to be 70 °C, except for those of tolclofos-methyl and fenthion. Generally, the recoveries of OPPs would increase along with the increase of extraction temperature. The recoveries of some OPPs decreased when extraction temperature exceeded



**Fig. 3.** Effect of solid-to-liquid ratio \*Extraction solvent, hexane; extraction temperature, 70 °C; extraction time, 8 min; number of microwave absorption tubes, three.



**Fig. 4.** Effect of extraction temperature \*Extraction solvent, hexane; extraction time, 8 min; solid-to-liquid ratio, 3:25 (g/mL); number of microwave absorption tubes, three.

70 °C, which may be due to the loss of volatilization of these analytes at higher temperature [35]. Finally, 70 °C was chosen to be the feasible extraction temperature.

### 3.2.5. Extraction time

The effect of extraction time on recoveries of OPPs was investigated. The experimental results indicate that when the extraction time is shorter than 8 min, the recoveries for OPPs increase with the increase of the extraction time, and most of the recoveries of OPPs are the highest when the extraction time is 8 min. Thus, 8 min was considered as the appropriate extraction time.

### 3.2.6. Statistical analysis

To evaluate the optimization of these extraction parameters, the student's *t*-test was applied, and the *t* values are listed in Table 2. The statistical analysis indicates that the differences between the optimal values and the nearest values for the experimental

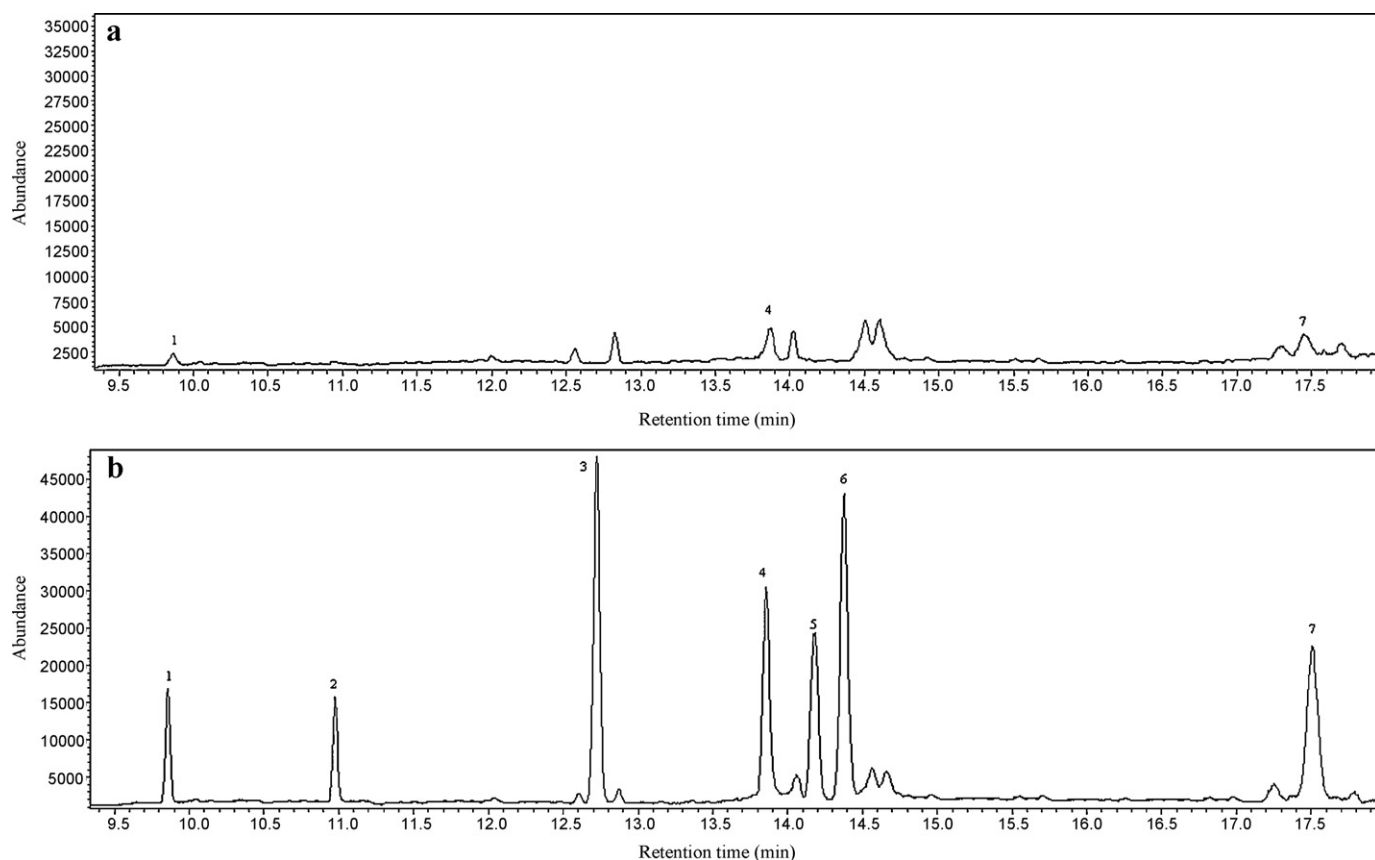
parameters are significant ( $P < 0.05$ ). So, the selected optimal extraction parameters should be reasonable.

### 3.3. Validation of the method

Seven OPPs were identified by their retention time and fragmentation spectra in the chromatograms. Standard curves were made with freshly prepared standard solutions. All the experiments were performed in triplicate. The linear regression equations, linear ranges and correlation coefficients are listed in Table 1. The correlation coefficients (*r*) ranging from 0.9995 to 0.9998 are obtained for all the analytes. The instrumental limits of detection (LODs) ( $S/N=3$ ) and quantification (LOQs) ( $S/N=10$ ) are listed in Table 1. The LODs and LOQs are in the range of 0.15–0.54  $\mu\text{g}/\text{kg}$  and 0.5–1.8  $\mu\text{g}/\text{kg}$  for all analytes, respectively.

**Table 2**  
*t* values between experimental parameter.

Experimental parameter	Parameter values	Analytes						
		Phorate	Diazinon	Tolclofos-methyl	Malathion	Chlorpyrifos	Fenthion	Methidathion
Solid-to-liquid ratio (g/mL)	1:25	3.7	3.1	1.9	6.8	3.5	2.3	2.1
	2:25	5.0	6.9	2.4	3.6	1.7	4.4	2.4
	3:25	2.4	0.6	0.2	0.1	0.1	0.1	0.9
	4:25	1.4	3.9	2.2	0.5	0.1	1.7	1.7
	5:25	10.4	8.2	1.7	3.2	7.9	0.3	1.7
	60	4.7	11.6	1.8	3.1	4.1	12.1	2.2
	70	1.3	0.3	1.5	0.9	0.4	4.1	2.2
Extraction temperature (°C)	80	9.2	7.5	6.9	3.1	1.9	4.4	0.6
	8	5.5	3.6	2.5	0.6	1.2	0.1	0.5
	10	0.2	2.0	0.1	0.1	0.7	0.7	0.1
	12	1.4	1.3	0.1	0.5	1.9	6.1	1.7
	14							



**Fig. 5.** The GC–MS chromatograms of cucumber sample (a) and spiked cucumber sample (b) \* Concentration of each pesticide, 30  $\mu\text{g}/\text{kg}$  for phorate, diazinon, methidathion, fenthion and malathion; 15  $\mu\text{g}/\text{kg}$  for tolclofos-methyl and 60  $\mu\text{g}/\text{kg}$  for chlorpyrifos. 1. phorate, 2. diazinon, 3. tolclofos-methyl, 4. malathion, 5. chlorpyrifos, 6. fenthion, and 7. methidathion.

### 3.4. Real sample analysis

The proposed method was applied to the determination of OPPs residues in four vegetable samples (cole, eggplant, pepper and cucumber). The analytical results are shown in Table 3. Chromatograms of both unspiked and spiked samples are shown in Fig. 5. Some kinds of OPPs including phorate, tolclofos-methyl, malathion, chlorpyrifos, fenthion and methidathion are detectable in the real samples. The concentrations of the OPPs in the real samples are in the range of 1.67–8.57  $\mu\text{g}/\text{kg}$ . Diazinon was not detectable in all the real samples. These pesticide concentrations found are all lower than Maximum Residue Limits (0.01 mg/kg) [36,37]

**Table 3**

Concentrations of seven OPPs in four vegetable samples ( $\mu\text{g}/\text{kg}$ ) ( $n=3$ ).

Analytes	Cucumber	Eggplant	Cole	Pepper
	Mean $\pm$ SD	Mean $\pm$ SD	Mean $\pm$ SD	Mean $\pm$ SD
Phorate	1.83 $\pm$ 0.12	1.95 $\pm$ 0.11	1.77 $\pm$ 0.09	ND
Diazinon	ND	ND	ND	ND
Tolclofos-methyl	ND	ND	2.29 $\pm$ 0.11	ND
Malathion	2.41 $\pm$ 0.22	3.36 $\pm$ 0.14	4.48 $\pm$ 0.19	2.11 $\pm$ 0.09
Chlorpyrifos	ND	3.11 $\pm$ 0.03	ND	ND
Fenthion	ND	2.26 $\pm$ 0.09	1.73 $\pm$ 0.04	1.67 $\pm$ 0.22
Methidathion	8.57 $\pm$ 0.38	ND	2.68 $\pm$ 0.16	4.87 $\pm$ 0.43

ND, not detected; SD, standard deviation.

**Table 4**

Recoveries and RSD for spiked vegetable samples at two different concentration levels of OPPs ( $n=3$ ).

Analytes	Spiked concentration ( $\mu\text{g}/\text{Kg}$ )	Cucumber		Eggplant		Cole		Pepper	
		Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
Phorate	10	91.5	7.87	80.2	6.64	85.4	4.00	84.3	6.78
	50	92.1	4.34	80.4	5.33	86.4	5.17	81.4	11.8
Diazinon	10	90.5	2.72	84.1	5.43	76.7	2.14	86.9	7.82
	50	93.6	3.56	86.1	9.77	79.6	10.9	78.5	11.2
Tolclofos-methyl	5	91.4	1.51	78.2	4.08	76.5	2.81	78.0	4.09
	25	96.4	4.31	86.6	7.54	78.9	8.96	78.9	5.97
Malathion	10	107.7	9.24	83.6	4.69	85.1	2.30	100.7	9.61
	50	106.5	2.94	108.1	2.20	96.1	12.0	99.4	5.68
Chlorpyrifos	20	104.1	4.34	94.6	1.24	77.9	3.14	87.5	1.30
	100	108.4	3.50	82.5	10.5	84.2	7.15	85.2	3.55
Fenthion	10	98.6	4.79	78.9	4.31	79.1	2.88	84.4	13.1
	50	101.1	7.41	84.1	8.43	84.7	7.17	81.3	2.78
Methidathion	10	105.8	4.82	86.0	5.62	86.0	5.44	96.2	8.54
	50	104.5	4.54	109.4	4.61	105.6	12.8	100.9	8.77

**Table 5**  
Comparison with other reported MAE methods for pesticide determination.

Analytes	Sample preparation step	Extraction time (min)	Extraction temperature (°C)	Electric energy/solvent volume (kW h/L)	Recovery (%)	Ref.
OPPs	Vegetable acetonitrile/acetone (50:50, v/v) → extraction → dSPE (PSA, magnesium sulfate) → clean up → analysis hexane/acetone (50:50, v/v) → extraction → ENVI-Carb SPE cartridge → hexane/ethyl acetate (80:20) → elution → GC-ECD → analysis	9	75–80	3	82–114	[33]
OCPs	Vegetable water/acetone (5:95, v/v) → extraction → centrifuge/florisil cartridge → Clean up → hexane/ethyl acetate (80:20) → elution → GC-MS → analysis	12	No control	8	92–132	[30]
OCPs	Seasame seed hexane/acetone (1:1, v/v) → extraction → alumine, florisil, Sop-Pak cartridge → Clean up → hexane/ethyl acetate (80:20) → elution → GC-ECD → analysis	10	100	4	84–105	[40]
OCPs	Plants hexane/ethyl acetate (80:20, v/v) → extraction → GC8, florisil cartridge → Clean up → hexane/ethyl acetate (80:20) → elution → PTV-LIV-GC/MS/MS → analysis	12	95–107	8	50–108	[20]
Multiresidues pesticides	Edible seaweed MAT, hexane → extraction → GC-MS → analysis	20	125	10	–	[41]
OPPs	Vegetable	8	70	2	76.5–109.4	This method

In order to validate the accuracy of MAE procedure, the spiked samples were analyzed in triplicate and the results are shown in Table 4. The recoveries are in the range of 76.5–109.4%, and the relative standard deviations (RSDs) are  $\leq 13.1\%$ . These results could be acceptable. The proposed MAE was proved to be a feasible method in extracting OPPs from vegetable samples with non-polar solvent only.

### 3.5. Safety consideration

In this work, the extraction was carried out with a commercial PTFE microwave closed-vessel. At airtight condition, inflammation could hardly taken place because of the lack of oxygen. The security of microwave absorption tube filled with graphite powder was tested again and again, and no blast happened in the extraction process. The bearable pressure of the PTFE microwave closed-vessel is 200 p.s.i. (ca. 14 bar) [38]. In proposed MAE, the extraction process was carried out using hexane as extraction solvent, the pressures of the solvent inside PTFE microwave closed-vessel can be obtained based on Antoine equation [39] and are about 1.21 bar, 1.63 bar and 2.14 bar at temperatures 70 °C, 80 °C and 90 °C, respectively. So, the proposed method should be safe.

### 3.6. Comparison of the proposed method with other MAE methods

A comparison between the proposed method and other MAE methods reported in literature was made, and the results are shown in Table 5. The results indicate that there is no significant difference in the recoveries obtained by different methods. However, compared with other method is simpler, and has some advantages in extraction time and electricity energy consumption.

In recent report, graphite fiber was the first time used as a sorbent in micro-solid phase extraction, coupled with MAE. The graphite fiber exhibits excellent extraction capability for the extraction of polycyclic aromatic hydrocarbon compounds [25]. A polytetrafluoroethylene/graphite compound (Weflon) was used as a stir bar in Soxhlet extraction assisted by microwave and the extraction of fats and oils was complete and efficient [31]. In this study, the graphite powder was sealed in the glass tube was used as microwave absorbing medium for the extraction of OPPs. The results were satisfactory.

## 4. Conclusions

In this study, by sealing graphite powder in glass tube, microwave absorption tube was made and used to directly heat sample system under microwave irradiation. Microwave absorption tube was harmless and could be reused. With both the assistance of microwave absorption tube and the use of portable microwave extraction apparatus, seven OPPs were extracted from vegetable samples by MAE using single non-polar solvent at 70 °C within 8 min, and the extracts were directly analyzed by GC-MS without any clean-up step. Some experimental parameters of MAE were studied and optimized. The proposed method was proved to be a simple, efficient and feasible method in extracting OPPs from vegetable samples.

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