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Integrated microwave processing system for the extraction of organophosphorus pesticides in fresh vegetables

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ABSTRACT

A simple and efficient integrated microwave processing system (IMPS) was firstly assembled and validated for the extraction of organophosphorus pesticides in fresh vegetables. Two processes under microwave irradiation, dynamic microwave-assisted extraction (DMAE) and microwave-accelerated solvent elution (MASE), were integrated for simplifying the pretreatment of the sample. Extraction, separation, enrichment and elution were finished in a simple step. The organophosphorus pesticides were extracted from the fresh vegetables into hexane with DMAE, and then the extract was directly introduced into the enrichment column packed with active carbon fiber (ACF). Subsequently, the organophosphorus pesticides trapped on the ACF were eluted with ethyl acetate under microwave irradiation. No further filtration or cleanup was required before analysis of the eluate by gas chromatography–mass spectrometry. Some experimental parameters affecting extraction efficiency were investigated and optimized, such as microwave output power, kind and volume of extraction solvent, extraction time, amount of sorbent, elution microwave power, kind and volume of elution solvent, elution solvent flow rate. Under the optimized conditions, the recoveries were in the range of 71.5–105.2%, and the relative standard deviations were lower than 11.6%. The experiment results prove that the present method is a simple and effective sample preparation method for the determination of pesticides in solid samples.

1. Introduction

Organophosphorus pesticides (OPPs) are widely used in agriculture to prevent insect damage to crops [1–3], but the overuse of OPPs has caused problems of pesticide residues in food commodities, water, air, and soil [4–11]. Recently, researchers have found that most human acute toxicity incidents are linked with the intoxication of OPPs, especially OPP residues in vegetables and fruits [12,13]. So development and optimization of reliable and efficient analytical methods for the precise and accurate determination of OPPs in foods and environmental matrices are of great importance.

The step of sample preparation is the key aspect in the determination of OPPs. Various methods have been proposed for the extraction of OPPs from fruits and vegetables, such as Soxhlet extraction [14], solid-phase extraction [15], matrix solid-phase dispersion extraction [16–18], headspace solid-phase microextraction [19], liquid–liquid extraction [20], dispersive liquid–liquid microextraction [21], supercritical fluid extraction [22], ultrasonic extraction [23], accelerated solvent extraction [24–25], microwave-assisted extraction [26–30]. In these methods, microwave-assisted extraction (MAE) has been proved to be a quick, efficient and environment-friendly method. In recent years, many improvements of MAE were reported, such as dynamic microwave-assisted extraction [31–35], solvent-free microwave-assisted extraction [36–37]. The aims of these improvements of MAE were to simplify pretreatment step, improve microwave efficiency and reduce consumption of solvent and time.

Recently, active carbon fiber (ACF) was considered to be one of the most promising adsorbents [38–40] due to its large specific surface area, pore volume, and uniform microporosity. Compared with granular activated carbon (GAC) and any other commercially available adsorbents, ACFs have the advantages of fast adsorption rate and ease of handling. The adsorption capacity of ACF is usually very high due to its high BET surface area and macro-pore size distribution [41–44]. On the other hand, generally ACF may be heated up instantly, because the heating during MAE is based on the direct effect of microwave on molecules by ionic conduction and dipole rotation [45]. The well-distributed micropores of ACF make it easy to adsorb and desorb volatile and semi volatile

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organic compounds. Based on the characteristics of ACF, the dynamic microwave assisted solvent elution based on ACF can be employed for the elution of OPPs.

Microwave heating is not heat transfer process, but a volumetric heating. The efficiency of the microwave heating is higher than that of the traditional heating method [46]. In microwave irradiation, the interaction between the activated carbon fiber and the adsorbed organic pollutants can be reduced, which can promote the adsorbed pollutants transferred to the solvent system quickly and efficiently. At the same time, the temperature of the elution solvent increases rapidly, and the solubility of the organic pollutants in the solution also increases [47].

The microwave-assisted solvent elution technique was reported by Lee and his co-workers [48], and it is effective to extract non-polar and polar from different aqueous matrices with the assistance of an enrichment procedure using membrane disks. However when the extraction process finished, the vessels must be cooled to room temperature before being opened to avoid losses of volatile analytes, thus the overall extraction time increased considerably. In addition, some compounds may be partly decomposed in this condition [49].

In this work, the fresh solvent was continuously pumped through the extraction cell. The analytes were extracted from the sample, transferred from the extraction vessel to the enrichment column and then retained on the ACF. The extraction was performed in a recirculating system which showed obvious advantages in short duration and low consumption of solvent. Due to the ACF has excellent microwave absorbing performance, the usage of microwave heating elution combined with solvent elution, and adopting countercurrent elution can shorten the elution time, and obtain the high efficiency to extract OPPs.

The new extraction method was applied for the extraction of organophosphorus pesticides (OPPs) in vegetables, using dynamic microwave-assisted extraction (DMAE) combined with solid-phase extraction and the dynamic microwave-assisted solvent elution technique. The determination of the analytes was carried out by gas chromatography–mass spectrometry.

2. Experimental

2.1. Chemicals and reagents

Six OPPs, including phorate, diazinon, methyl-parathion, fenitrothion, fenthion and fenamiphos, 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. Hexane, isooctane, petroleum ether, carbon tetrachloride, ethyl acetate, acetonitrile, acetone and methanol were of analytical grade and purchased from Beijing Chemical Factory (Beijing, China). The ACF was treated by Soxhlet extraction with acetone and hexane (1:1) for 4 h and then heated at 100 °C for 2 h before use.

2.2. Apparatus and instruments

The integrated microwave processing system (IMPS) was assembled in our laboratory. It consisted of a modified household microwave oven (NN-MX25WF, Shanghai, China) with the output maximum power of 800 W, two peristaltic pumps (Michem Technology Co., Ltd., Beijing, China). A glass column (8.0 cm × 0.5 cm i.d.) packed with ACF was used to adsorb the analytes, and another glass tube (10.0 cm × 0.5 cm i.d.) was used as extraction vessel. Two peristaltic pumps and two six-way valves (Valve 1 and Valve 2) were used to deliver the extraction solvent and elution solvent, respectively. A metallic sheath which overcovered the enrichment column could move up and down and was connected to the microwave cavity wall. When the metallic sheath was lifted up, the objects in it would be exposed and heated by microwave irradiation. On the contrary, when the metallic sheath was put down, the objects would be packed and avoided to be heated.

2.3. Sample preparation

Fresh vegetable samples (celery cabbage, tomato, green peppers and cucumber) were purchased from local supermarket (Changchun, China). The samples were chopped and homogenized with food processor. The spiked samples were prepared by adding working standard solutions into the samples. The spiked samples were stored for 24 h in the dark place at room temperature. The celery cabbage sample was used in the optimization of experimental conditions. All experiments were performed in triplicate.

2.4. Procedure

2.4.1. Extraction and elution

The extraction vessel and enrichment column are shown in Fig.1. The integrated microwave processing system is shown in Fig. 2. 2.0 g of sample was accurately weighted and mixed with 1.0 g of quartz sand used as dispersant, and then the mixture was placed between two small plugs of glass fiber in the extraction vessel. 0.15 g of ACF was added into the enrichment column. 8 mL extraction solvent and 12 mL elution solvent were added in the solvent reservoirs, respectively.

In extraction step (Fig. 2a), pump A was activated and the extraction solvent was passed through the extraction vessel and ACF column. The metallic sheath was put down. When the extraction vessel was properly filled with the extraction solvent, microwave heating was started. The analytes were extracted, and transferred into the ACF columns directly by extraction solvent at a flow rate of 1.0 mL/min. The extraction solvent was used recirculatly. When extraction was finished, both pump A and microwave irradiation were stopped.

In elution step (Fig. 2b), pump B was activated and the elution solvent was counter-current passed through the ACF column. The metallic sheath was lifted up to make the ACF column exposed under the microwave irradiation. When the ACF column was properly filled with the elution solvent, 250 W microwave heating was started. The OPPs on the ACF were eluted with 12 mL ethyl acetate into flask at a flow rate of 10 mL/min. After elution, the eluate obtained was concentrated to dryness in rotary evaporator at 30 °C. The residue was dissolved in 100 μL of hexane, filtrated through 0.22 μm PTFE filter membrane, and then directly analyzed by GC–MS.

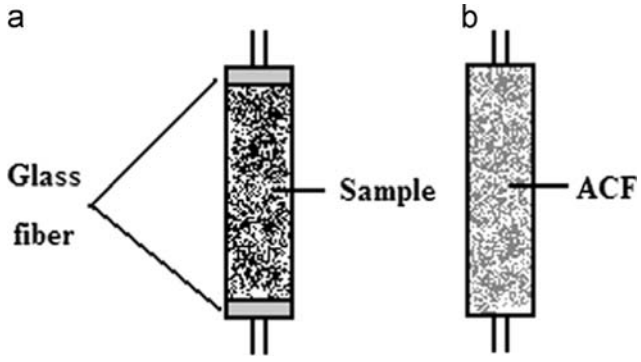

Fig. 1. Extraction vessel (a) and enrichment column (b).

2.4.2. Chromatographic determination

A GC–MS system (GC–MS QP2010 plus, Shimadzu, Kyoto, Japan) was used. Chromatographic separation was conducted with a DB-5 MS capillary column (30 m × 0.25 mm I.D., film thickness of 0.25 μm, J&W Scientific, Folsom, CA, USA). Helium (purity ≥ 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, ramp to 170 °C at a rate of 15 °C/min, held for 9 min, and then ramp to 210 °C at a rate of 15 °C/min, held for 2 min, finally raised to 250 °C at a rate of 15 °C/min, held for 3 min. Injector temperature was maintained at 280 °C, and the injection volume was 1.0 μL in the splitless mode. The ion source and interface temperatures were 200 °C and 250 °C, respectively, and electron impact ionization energy was 70 eV. The mass spectrometer was operated in 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.

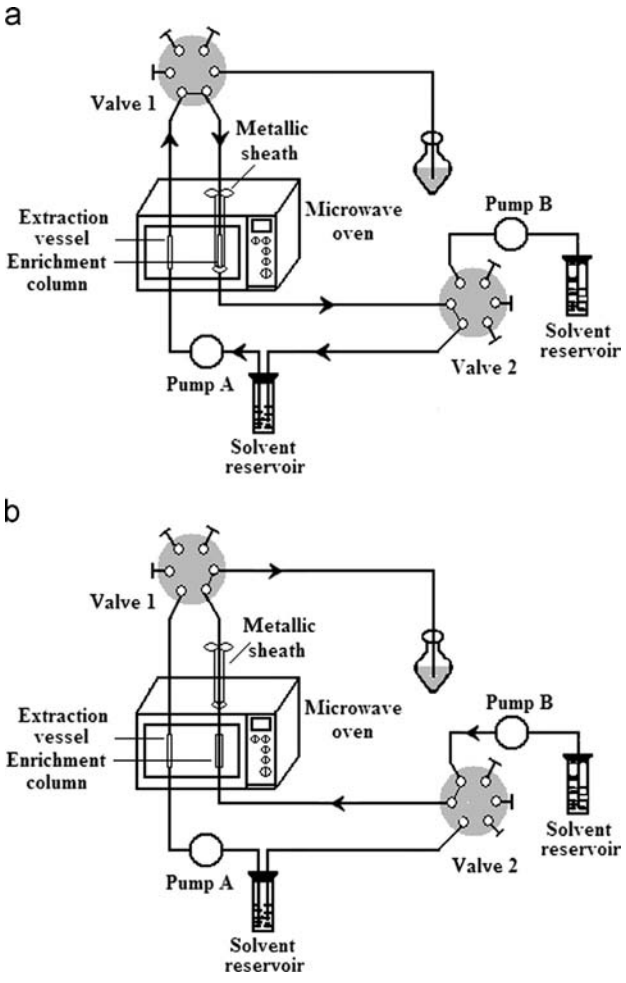

Fig. 2. Integrated microwave processing system.

3. Results and discussion

In this study, non-polar solvent was used as extraction solvent instead of polar solvent or the mixture of non polar and polar solvent, mainly because the fresh vegetables containing a lot of water could absorb microwave. Compared with polar solvent, the usage of non-polar solvent can reduce a lot of undesired interferents to be extracted simultaneously [50–53].

Main variables potentially affecting IMPS were evaluated in order to obtain an efficient extraction. When one parameter was changed, the other parameters were fixed at their optimized values. All the experiments were performed in triplicate. The spiked samples at analyte concentration of 10 μg/kg were used.

3.1. Optimization of DMAE conditions

3.1.1. Microwave output power

The effect of microwave output power on the extraction efficiency was investigated in the range of 60–500 W. It can be clearly seen from Fig.3 that the optimal microwave output power is 300 W. Under the fixed flow of solvent, the temperatures of sample and solvent would rise accordingly with the increase of microwave output power [33]. The high temperature would cause the cell of vegetable rupturing more completely and the target compounds dissolving more quickly. Furthermore, high temperature was also beneficial to the diffusion and mass transfer during extraction, and the improvement of

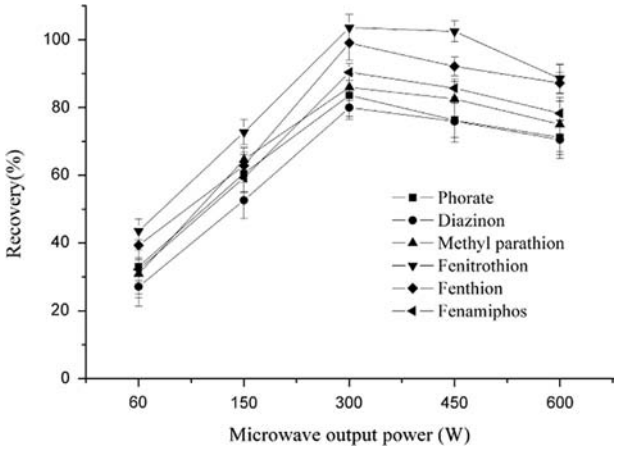

Fig. 3. Effect of microwave output power on extraction recoveries of OPPs. Extraction solvent, hexane; extraction solvent flow-rate, 1.0 mL/min; extraction time, 5 min; extraction solvent volume, 8 mL; amount of sorbent, 0.15 g; elution solvent, ethyl acetate; elution microwave power, 250 W; volume of elution solvent, 12 mL; and elution solvent flow-rate, 10 mL/min.

Table 1
Qualitative and quantitative data of six OPPs.

Analytes	Retention time (min)	Main fragment ion (*m*/*z*)	Calibration curve[a]	Linear range (μg/kg)	*r*	LOD (μg/kg)	LOQ (μg/kg)
Phorate	14.617	260[b], 75, 121, 65	$A=216.35+1577.60c$	0.50–50	0.9991	0.14	0.46
Diazinon	17.850	304[b], 137, 179, 93	$A=91.22+907.14c$	0.50–50	0.9993	0.17	0.58
Methyl-parathion	19.975	263[b], 109, 125, 79	$A=85.46+3181.57c$	1.00–100	0.9982	0.26	0.86
Fenitrothion	21.050	277[b], 125, 260, 79	$A=404.70+9344.66c$	0.50–50	0.9996	0.093	0.31
Fenthion	21.875	278[b], 125, 109, 169	$A=314.22+7300.49c$	0.50–50	0.9994	0.11	0.37
Fenamiphos	24.358	303[b], 154, 80, 217	$A=245.08+3523.58c$	0.50–50	0.9992	0.13	0.42

[a] *A* and *c* are the peak area and concentration of the analytes, respectively.
[b] The ion for quantitative analysis.

dissolving capacity of extraction solvent at the same time. However, high microwave output power could also make the temperature of sample too high which would result in the decomposition of OPPs [54]. The main decomposition reaction of OPPs should be due to the hydrolysis. The group containing phosphorus is easily hydrolyzed. Thus, 300 W was considered as the appropriate microwave output power.

3.1.2. Kind of extraction solvent

The kinds of extraction solvents tested in this study were n-hexane, isooctane, petroleum ether and carbon tetrachloride. The influence of extraction solvent on the extraction efficiency is shown in Fig.4. The results demonstrate that extraction recoveries obtained with n-hexane are higher than those obtained with others. In the extraction process, analytes were first transferred from sample to extraction solvent phase, and then adsorbed by ACF. When n-hexane was used as extraction solvent and ethyl acetate was used as elution solvent, few of polar interferents were detectable by GC–MS, which could consider that n-hexane was the feasible extraction solvent to make the extract clean enough to be analyzed directly. Thus, n-hexane was chosen as the extraction solvent for the further applications.

3.1.3. Extraction time

The effect of extraction time was examined in the range of 2–7 min. The results showed that the recoveries of OPPs did not increase when the extraction time exceeded 5 min. Thus, 5 min was chosen as the extraction time in this study.

3.1.4. Extraction solvent volume

The effect of extraction solvent volume was studied in the range of 5–15 mL. The results revealed that the extraction solvent volume had no significant effect on the extraction recoveries. In order to make the circulatory system of IMPS running well, 8 mL of extraction solvent was used in this study.

3.1.5. Amount of sorbent

ACF was used as sorbent in extraction step. The influences of amount of ACF ranged from 0.05 to 0.25 g were studied, and the results are shown in Fig. S-1 of the Supplementary material. It can be seen that most of recoveries increase slightly when until the amount of ACF larger than 0.15 g. Therefore, 0.15 g of ACF was used.

3.2. Optimization of MASE conditions

3.2.1. Elution microwave power

The effect of microwave irradiation power on the elution efficiency was investigated and the experimental results are shown in Fig.5. The high desorption microwave power was beneficial to the desorption of the target compounds from ACF to the elution solvent. However, the strict control of the power is very significant, because the high power will produce instantaneous high temperature which may cause the decomposition of the analyte. However, the low power is not enough to desorb the analyte from the ACF. The results revealed that the extraction recoveries of the pesticides increased along with the increase of microwave irradiation power from 60 to 250 W, and did not increase when microwave irradiation power was higher than 250 W. The elution solvent was colored when the microwave irradiation power reached 350 W. The probable reason was that the impurities like pigment adsorbed by ACF would be desorbed and can be introduced into the extraction solvent. Thus, the optimized microwave irradiation power was 250 W.

3.2.2. Kind of elution solvent

In this study, five kinds of elution solvents, including ethyl acetate, acetone, n-hexane, methanol and acetonitrile, were tested. The experimental results are shown in Fig. 6. The results demonstrate that the recoveries obtained with ethyl acetate and acetone are higher than those obtained with n-hexane, methanol and acetonitrile. The n-hexane is poor to elute the analyte. The toxic of ethyl acetate is lower than that of acetone and the polarity of ethyl acetate is similar to that of the OPPs. Hence ethyl acetate was chosen as the elution solvent.

3.2.3. Volume of elution solvent

The effect of the volume of elution solvent on extraction efficiency is shown in Fig. S-2 of the Supplementary material. The results reveal that the extraction recoveries of the pesticides increase with the increase of the desorption volume from 6 to 12 mL, and then change slightly. The probable reason is that the small extraction volume is not enough to elute the pesticides from the ACF completely. When the desorption flow rate was unchanged, the small volume means the short microwave irradiation time. Take all those to consideration, 12 mL of the elution

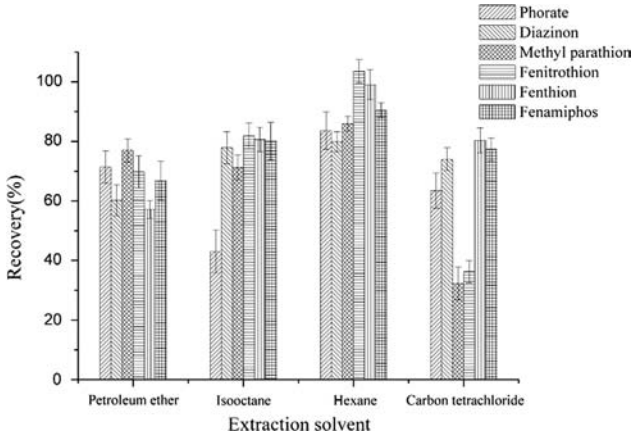

Fig. 4. Effect of kind of extraction solvent on extraction recoveries of OPPs. Microwave output power, 300 W; extraction solvent flow-rate, 1.0 mL/min; extraction time, 5 min; extraction solvent volume, 8 mL; amount of sorbent, 0.15 g; elution solvent, ethyl acetate; elution microwave power, 250 W; volume of elution solvent, 12 mL; and elution solvent flow-rate, 10 mL/min.

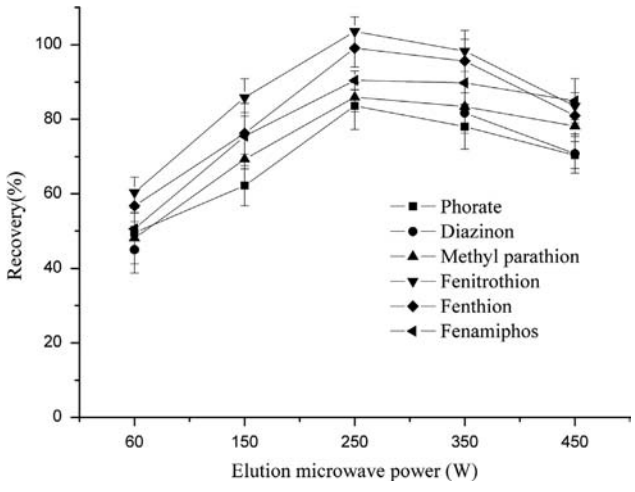

Fig. 5. Effect of elution microwave power on extraction recoveries of OPPs. Extraction solvent, hexane; microwave output power, 300 W; extraction solvent flow-rate, 1.0 mL/min; extraction time, 5 min; extraction solvent volume, 8 mL; amount of sorbent, 0.15 g; elution solvent, ethyl acetate; volume of elution solvent, 12 mL; and elution solvent flow-rate, 10 mL/min.

solvent volume was chosen in this work, which was enough to complete the extraction.

3.2.4. Elution solvent flow rate

The effect of the extraction solvent flow rate from 3 to 20 mL/min on the extraction recovery was investigated. Generally, under the same microwave power, the instantaneous temperature of ACF may be unchanged. The solvent temperature is related to the flow rate of solvent. If the flow rate is too slow, solvent temperature will be too high and the target may be decomposed. So the appropriate flow rate is very important. The results shown in Fig. S-3 of the Supplementary material indicate that when the extraction solvent flow rate is 10 mL/min, the recoveries for all analytes are highest, and the desorption time is only 1 min. Thus, 10 mL/min was chosen as the elution solvent flow rate.

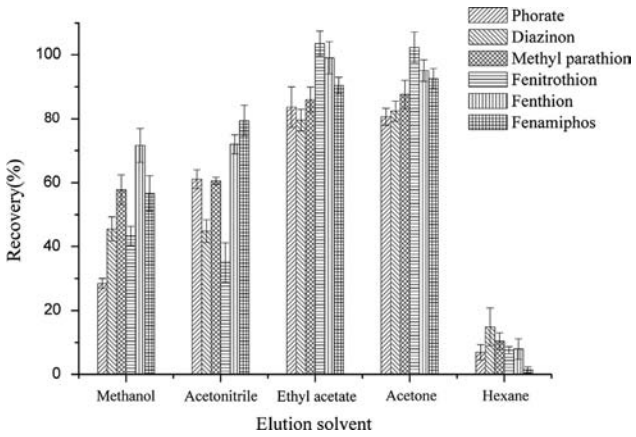

Fig. 6. Effect of kind of elution solvent on extraction recoveries of OPPs. Extraction solvent, hexane; microwave output power, 300 W; extraction solvent flow-rate, 1.0 mL/min; extraction time, 5 min; extraction solvent volume, 8 mL; amount of sorbent, 0.15 g; elution microwave power, 250 W; elution solvent flow-rate, 10 mL/min; and elution solvent volume, 12 mL.

3.3. Validation and application of the method.

3.3.1. Limits of detection and quantification

All pesticides determined by GC/MS were identified by their retention time and fragmentation spectra in the chromatograms. Under the optimal experimental conditions, a series of experiments were performed for obtaining linear ranges, precision. All the experiments were performed in triplicate. The working curves were constructed by plotting the peak areas measured versus the concentrations of analytes. The linear regression equations and correlation coefficients are listed in Table 1. The correlation coefficients (r) ranging from 0.9982 to 0.9996 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 0.093–0.26 μg/kg and 0.31–0.86 μg/kg for all analytes, respectively.

3.3.2. Precision and recovery

The precision of this method was evaluated at the concentration of 10 μg/kg. The intra-day precision was obtained by determining the analytes five times in the same day, and the inter-day precision was obtained by performing the same process in five consecutive days. The recoveries and RSDs are shown in Table S-1 of the Supplementary material. All the RSDs of intra- and inter-day were lower than 8.0% for six target components.

3.3.3. Analysis of real samples

To evaluate the applicability of the present method, some real samples, including celery cabbage, tomato, green peppers and cucumber were analyzed. The typical chromatograms of the blank and spiked celery cabbage sample are shown in Fig. 7. As can be seen, the OPPs in the samples are not detectable. The reason

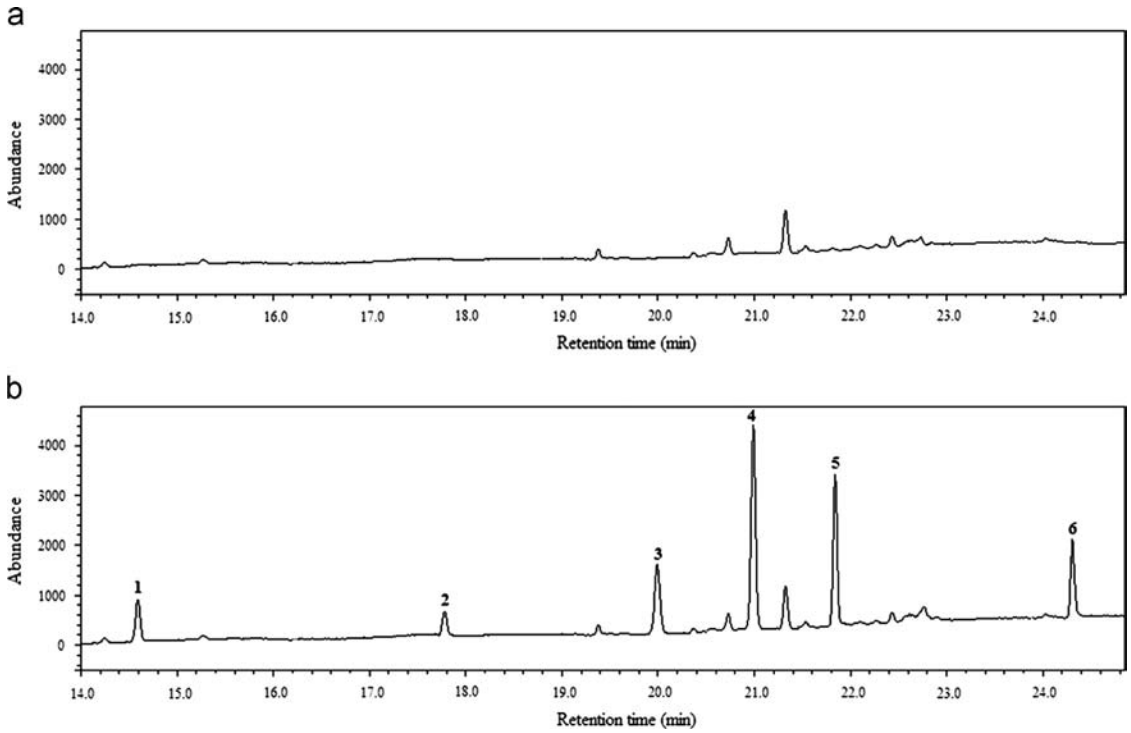

Fig. 7. GC–MS chromatograms of celery cabbage sample (a) and spiked celery cabbage sample (b). *Spiked concentrations: 10 μg/kg for phorate, diazinon, fenitrothion, fenthion and fenamiphos; 20 μg/kg for methyl parathion. 1. phorate, 2. diazinon, 3. methyl parathion, 4. fenitrothion, 5. fenthion, and 6. Fenamiphos.

Table 2
Analytical results for vegetable samples.

Analytes	Spiked concentration (μg/kg)	Celery cabbage		Tomato		Cucumber		Green peppers	
		Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
Phorate	3	86.3	8.2	84.2	7.8	85.3	4.5	88.1	8.6
	20	82.7	9.7	85.1	4.1	85.9	6.3	84.9	5.3
Diazinon	3	77.8	7.7	74.5	8.7	73.8	8.2	71.5	11.6
	20	79.6	5.4	77.7	7.5	75.2	7.1	74.9	5.3
Methyl-parathion	6	81.5	3.7	89.3	4.5	85.4	8.5	82.1	9.7
	40	84.9	6.4	85.7	9.3	88.5	5.8	89.9	4.0
Fenitrothion	3	94.8	4.2	98.3	6.8	96.6	5.6	93.8	2.6
	20	105.2	6.2	103.5	3.1	100.5	2.6	98.9	4.2
Fenthion	3	95.2	3.7	93.5	5.2	94.5	4.7	99.5	9.2
	20	99.6	3.3	101.6	4.3	90.2	8.6	96.7	4.2
Fenamiphos	3	86.7	2.6	88.3	3.2	87.4	5.7	92.3	2.4
	20	90.0	3.8	93.9	5.3	89.1	9.5	85.8	5.3

Table 3
Comparison with other reported methods for pesticide determination in vegetable samples.

Analytes	Method	Extraction solvent	Volume of extraction solvent (mL)	Extraction time (min)	Recovery (%)	Ref.
Organochlorine pesticides	SOE	Hexane–acetone (1:1, v/v)	80	1200	76.0–121.0	[14]
OPPs	HWBE	Hexane	90	20	92.1–103.9	[55]
Multiresidues pesticides	USE	Ethyl acetate hexane:acetone (1:1, v/v)	40	35	83.7–97.9	[23]
Multiresidues pesticides	MAE	hexane/acetone (1:1, v/v)	10	8	73.0–110.0	[26]
Hexachlorocy-clohexane	MSPE	n-hexane–ethyl acetate solvent mixture (70:30; v/v)	20	—	91.0–98.0	[17]
OPPs	Present method	Hexane	8	5	71.5–105.2	This work

should be that vegetable samples were purchased from local supermarket, not from vegetable growers and the number of each kind of sample was limited. No significant interference peaks are found at the retention times of OPPs. In order to validate the accuracy of the procedure, the spiked samples were analyzed in triplicate and the results are shown in Table 2. The recoveries are in the range of 71.5–105.2%, and the relative standard deviations (RSDs) are $\leq$ 11.6%. The results indicate that the present method provides acceptable recoveries and precision for the determination of OPPs in the real samples.

3.3.4. *Comparison of different extraction methods*

Some other methods reported in literature, including Soxhlet extraction (SOE) [14], hot-water bath extraction (HWBE) [55], ultrasonic solvent extraction (USE) [23], microwave-assisted extraction (MAE) [26], and matrix solid-phase dispersion (MSPD) [17] were compared with the present method for pesticide extraction in vegetable samples, and the results are shown in Table 3. It is obvious that there is no significant difference in the recoveries obtained by the methods. However, compared with the reference methods, the present method has some advantages in extraction time and solvent consumption.

4. Conclusion

Integrated microwave processing system, which couples DMAE with MASE, was developed and proved to be a rapid and efficient technique in treatment of the samples. The extraction, separation, enrichment and desorption could be performed in a single step. The ACF was proved to have good adsorption ability and desorption ability. With both the assistant of DMAE and MASE, six OPPs were extracted from vegetable samples, and the extracts were directly analyzed by GC–MS without any other clean-up step. Some experimental parameters were studied and optimized. The present method was proved to be a simple, efficient and feasible method in extracting OPPs from vegetable samples.

Acknowledgments

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Appendix A. Supplementary materials

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.talanta.2014.11.035.

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