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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.

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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 ≥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.

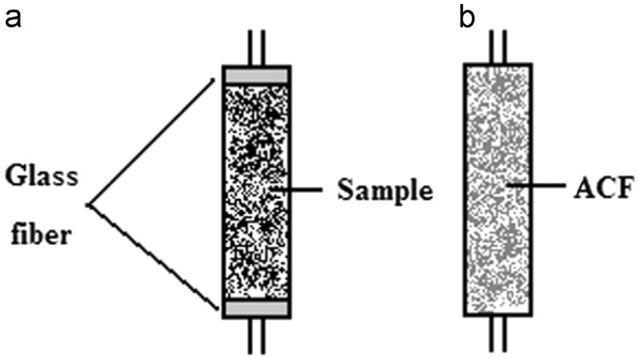

Fig. 1. Extraction vessel (a) and enrichment column (b).

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