



Analytical Methods

A fast, simple and green method for the extraction of carbamate pesticides from rice by microwave assisted steam extraction coupled with solid phase extraction



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ABSTRACT

This paper presented a fast, simple and green sample pretreatment method for the extraction of 8 carbamate pesticides in rice. The carbamate pesticides were extracted by microwave assisted water steam extraction method, and the extract obtained was immediately applied on a C₁₈ solid phase extraction cartridge for clean-up and concentration. The eluate containing target compounds was finally analysed by high performance liquid chromatography with mass spectrometry. The parameters affecting extraction efficiency were investigated and optimised. The limits of detection ranging from 1.1 to 4.2 ng g⁻¹ were obtained. The recoveries of 8 carbamate pesticides ranged from 66% to 117% at three spiked levels, and the inter- and intra-day relative standard deviation values were less than 9.1%. Compared with traditional methods, the proposed method cost less extraction time and organic solvent.

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1. Introduction

Carbamate pesticides are widely used in rice cultivation owing to their broad spectrum of biological activity (Ni, Qiu, & Kokot, 2005). The residues of carbamate pesticides in rice would threaten the human health and the environment safety (Lim & Lee, 2011). European Union requires that the maximum residue limits (MRLs) of several carbamate pesticides in rice are in the range of 0.02–1 µg g⁻¹ (Regulation (EC) No. 396/2005), while in China, the MRLs of six carbamate pesticides in cereals are in the range of 0.01–0.1 µg g⁻¹ (GB/T 5009.145, 2003). Therefore, sensitive and selective methods are desirable in the determination of carbamatepesticides in rice Pareja et al., 2011.

For the sensitive detection of carbamate pesticides in food products, gas chromatography (GC) and gas chromatography–mass spectrometry (GC–MS) were often used but not desirable due to the low thermal stability of carbamate pesticides (Delgado, Barros, Fernández-Tostado, & Polo-Díez, 2001; García-Rodríguez, Cela-Torrijos, Lorenzo-Ferreira, & Carro-Díaz, 2012). High performance liquid chromatography (HPLC) (Wu et al., 2009) and liquid chromatography–mass spectrometry (LC–MS) have been developed for the detection of carbamate pesticides, which were supposed

to be more sensitive, reliable and suitable (Masahiko, 2001; Sinha, Vasudev, & Rao, 2012).

Sample preparation is a crucial step during the whole analysis process especially in the analysis of complex solid samples. Traditional extraction methods for carbamate pesticides from rice are Soxhlet extraction and solvent extraction, but both methods suffer from the disadvantages of time-consuming and need large volumes of samples or toxic organic solvents (Zhang, Chu, Cai, An, & Li, 2006; Zhou, Xiao, & Li, 2012). Recently, “green extraction” methods have been reported for the extraction and concentration of carbamate pesticides such as cloud point extraction (Chen, Zhao, Liu, Zhou, & Yang, 2009), dispersive liquid–liquid microextraction (Lin et al., 2011) and hot water extraction (Bogialli, Curini, Corcia, Nazzari, & Tamburro, 2004). In these methods, the consumption of toxic organic solvents was decreased and the extraction efficiency was satisfactory.

Microwave assisted extraction (MAE) was a commonly used sample preparation technique for the extraction of organic contaminants from solid matrix (Satpathy, Tyagi, & Gupta, 2011). With the development of “green extraction” methods, “green microwave extraction” methods have been frequently reported such as solvent-free microwave extraction (SFME) (Li et al., 2012), microwave hydrodiffusion and gravity (MHG) (Bousbia et al., 2009) and microwave-assisted hydrodistillation (MAHD) (Golmakani & Rezaei, 2008). These methods have been applied on the isolation of volatile

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compounds from natural plants. In these methods, the extraction procedure was carried out utilising the water inside the plant issues, which was heated up and caused the cell rupture and dropping of the extracts (Li et al., 2012). The remarkable benefits of these methods were rapidity and low organic solvent consumption. However, these methods were not suitable for the extraction of compounds from samples in which the moisture content was low. To overcome the restriction, microwave assisted steam distillation (MASD) (Numata, Yarita, Aoyagi, & Takatsu, 2003) and microwave-assisted water steam extraction (MASE) (Song et al., 2012) were developed. For MASD, the sample soaked up water in a few minutes before extraction and nonpolar organic solvents were used to absorb the analytes. For MASE, the analytes could be extracted without soaking the low-moisture content samples before extraction, and the benefits of rapidity and low organic solvent consumption were maintained.

This work presented a simple and green method for the extraction of the carbamates from dry rice samples. The proposed method combined MASE with solid phase extraction (SPE). Parameters affecting extraction efficiency were investigated and optimised. The results obtained by the optimised MASE–SPE were compared with those obtained by traditional steam extraction and Soxhlet extraction. Finally, this proposed method was applied on determination of rice samples which were obtained from different production areas.

2. Materials and methods

2.1. Materials

Standards of carbamates (Methomyl, Aldicarb, Metolcarb, Propoxur, Carbofuran, Carbaryl, Diuron, Isoprocarb), all 98% or higher purity, were obtained from the Dr. Ehrenstorfer (Augsburg, Germany). Stock solutions of each compound were prepared in methanol at a concentration level of 500 mg L⁻¹. Work standard solutions were prepared by diluting certain amount of each stock solution. All stock and work solutions were stored in darkness at 4 °C. Chromatographic grade acetonitrile was obtained from Fisher (Pittsburgh, PA, USA), analytical grade quartz sands were obtained from Sinopharm Chemical Reagent (Shanghai, China). The ultra-purity water was obtained from a Milli-Q water system (Millipore, Billerica, MA, USA), the resistivity was 18.2 MΩ at 25 °C. All other reagents used were of analytical grade.

Rice samples were purchased from the OuYa Supermarket (Changchun, China), and the producing areas were Dehui, Mehekou, Yushu and Wanchang of Jilin province and Wuchang of Helongjiang province in China, respectively. All the samples were powdered using a cyclone mill and passed through a 40 mesh sieve. The blank samples were spiked with carbamate standards at 1 μg g⁻¹ for optimisation and validation experiments.

2.2. Microwave-assisted water steam extraction

The schematic diagram of MASE–SPE system was illustrated in Fig. 1. A microwave oven (NM-GS575WX, Panasonic, Japan) with 1000 W maximum microwave power was applied as microwave source. A glass tube (20 mL) immersed in ice water was used to collect the extract. A 50 mL filtering flask with a side tube was settled in the oven as a water steam generator. The extraction and collection parts of MASE system were connected by a slender tube (Teflon, 55 cm long; i.d. 3 mm). In a typical MASE procedure, 1 g rice powder mixed with certain quartz sands was transferred to an extraction vessel (polyethylene, 30 mm long; i.d. 15 mm), and then 500 μL methanol was added. The extraction vessel was settled in the microwave oven. Water steam, which was also generated by microwave heating, flowed through the extraction vessel. The extract containing the target compounds was condensed in the collection tube. Afterwards the condensed extract was submitted to SPE section for clean-up and concentration.

2.3. Steam extraction with electromagnetic heating

The steam extraction (SE) procedure was carried out with the same apparatus used in MASE except that water steam was generated by electromagnetic heating instead of microwave heating. Moreover, the transmission of steam (from the generator to the extraction vessel) and the extraction vessel were kept warm during SE process. Other conditions were consistent with those in MASE. Then the condensed extract was submitted to SPE section for clean-up and concentration.

2.4. Solid phase extraction for clean-up and concentration

In a typical SPE procedure, an Oasis HLB cartridge (10 mg, Waters, USA) was conditioned with 2.0 mL acetonitrile and 10.0 mL Milli-Q water, successively. Then the aqueous extract obtained in MASE or SE procedure was loaded onto the cartridge,

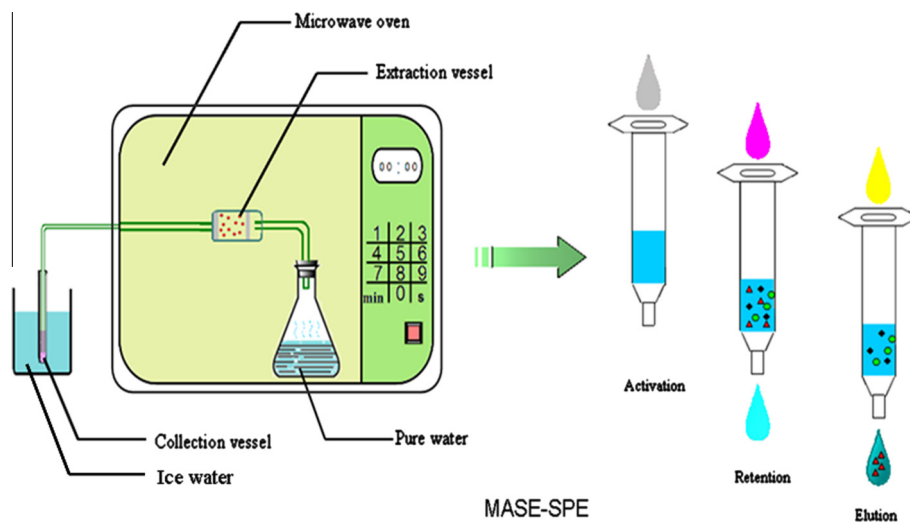


Fig. 1. Schematic diagram of MASE–SPE system.

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