



Coupling of multi-walled carbon nanotubes/polydimethylsiloxane coated stir bar sorptive extraction with pulse glow discharge-ion mobility spectrometry for analysis of triazine herbicides in water and soil samples



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ABSTRACT

An analytical method based on stir bar sorptive extraction (SBSE) coupled with pulse glow discharge-ion mobility spectrometry (PGD-IMS) was developed for analysis of three triazine pesticide residues in water and soil samples. An injection port with sealing device and stir bars hold device were designed and constructed to directly position the SBSE fiber including the extracted samples into the heating device, making desorption and detection of analytes proceeded simultaneously. The extraction conditions such as SBSE solid phase material, extraction time, extraction temperature, pH value and salt concentration were optimized. Mixture of MWCNTs-COOH and PDMS were shown to be effective in enriching the triazines. The LODs and LOQs of three triazines were found to be 0.006–0.015 $\mu\text{g kg}^{-1}$ and 0.02–0.05 $\mu\text{g kg}^{-1}$, and the linear range was 0.05–10 $\mu\text{g L}^{-1}$ with determination coefficients from 0.9987 to 0.9993. The SBSE-PGD-IMS method was environmentally friendly without organic solvent consumption in the entire experimental procedures, and it was demonstrated to be a commendable rapid analysis technique for analysis of triazine pesticide residues in environmental samples on site. The proposed method was applied for the analysis of real ground water, surface water and soil samples.

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1. Introduction

Triazine herbicides have been extensively used as selective herbicides for the control of broadleaf and grassy weeds in many agricultural crops, including corn, soybeans and sugarcane over the past years. However, triazine herbicides have some toxicity and may persist in the environment [1]. Moreover, atrazine is considered as a group to be endocrine-disrupting chemicals by the US Environmental Protection Agency [2]. The main analytical techniques reported for extraction methods of triazine pesticides from different samples were solid phase extraction (SPE) [3–5], solid-phase micro-extraction (SPME) [1,6,7], dispersive liquid-liquid

microextraction [8,9], stir bar sorptive extraction (SBSE) [10,11], and coupled with various instruments, such as gas chromatography [6,8], high performance liquid chromatography [1,3,7,11] and capillary electrophoresis [12], and so on. SBSE, which was developed in 1999 by Baltussen and co-workers [13], possesses many advantages, such as high sensitivity, high adsorption capacity, operation simply and organic solvents free, and has shown great potential in pesticides analysis in environmental samples [11].

Multiwalled carbon nanotubes (MWCNTs) are novel carbon nano materials with ultra-high specific surface areas. Based on unique physical and chemical properties, MWCNTs have been used as adsorbents in SPE, SPME or SBSE for either non-polar or polar compounds [14–16]. MWCNTs have been successfully applied as the SPME or SBSE coatings for the determination of organic pollutants in environmental samples including pesticides [15,17],

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polycyclic aromatic hydrocarbons (PAHs) [18,19], drugs [20], phenols [11,21] and organometallic compounds [22].

Ion Mobility Spectrometry (IMS) is a vanguard analytical instrument. Qualitative analysis capability of IMS relies on the specific mass, charge and calculated collision cross section of the analytes. IMS is well suited for large-scale, trace-level investigation and monitoring programs where sophisticated laboratory instrument is not available. A number of literatures have demonstrated the applications of IMS in analysis of explosives [23,24], drugs [25], pharmaceuticals [26], pesticides [27] and other agricultural or industrial contaminants [28].

A variety of sample-introduction systems are currently in use to introduce gas, liquid and solid samples into IMS instruments, including permeation tubes [29], membrane inlets [30], headspace samplers [31], SPME [32], SBSE [33], thermal desorption units [34], chromatographic columns [35], and so on [36]. SPME has been directly coupled to IMS instruments with ^{63}Ni or corona discharge as the ionization source [37,38]. Mohammad T. [32] designed a new desorption chamber to have the direct introduction of SPME fiber into the electrospray solvent flow, making direct introduction of liquid samples into IMS possible.

The objective of our work is to prepare a carboxylic multi-walled carbon nanotubes/polydimethylsiloxane (MWCNTs-COOH/PDMS) SBSE coating and direct combine SBSE with pulse glow discharge-ion mobility spectrometry (PGD-IMS) to develop a new method of MWCNTs-COOH/PDMS-SBSE-PGD-IMS for the determination of triazine herbicides in environmental samples. Some parameters affecting the extraction efficiency such as the effects of extraction time, extraction temperature, pH value and salt concentration were studied. Finally, this method was successfully applied to analyze the triazine herbicides in ground water, surface water and soil samples.

2. Experimental

2.1. Instrumentation and parameters

An IMS-KS-100 ion mobility spectrometer with pulse glow discharge (PGD) ion source used in this work was constructed by Wuhan Syscan Technology Limited Company (Wuhan, China). The device is composed of three main parts: A PGD ion source with an ion trap, an IMS drift tube and other utilities. The PGD ion source comprises a power supply, a discharge body and an ion extraction electrode, and it realizes the adjustment and control of the pulse ion flow intensity and the pulse width. Meanwhile, the problem that ions adhere to the ion source tube wall is solved, the ion loss is reduced, and the ion extraction rate is improved [39]. In this work, an appropriate new interface arranged on the introduction device was designed and constructed, helping us to have the direct introduction of SBSE fiber into the sample introduction mechanism. The schematic diagram of the device is illustrated in Fig. 1. When being used, the sealing device is connected with an introduction hole in a sealed manner. The injection port is formed into an opened type feeding hole, namely, the sealing device is not arranged at the injection port but is arranged on the injection device so as to seal the detection equipment injection port. Therefore, the contaminations are avoided, which caused by the fact that a partitioning pad needs to be punctured when a conventional injection device is used for injection samples. Furthermore, the interference of data is avoided, and the substance detection limit is effectively reduced. Using this interface, the extracted sample could be desorbed in sample introduction mechanism with a heating device and then moved toward the ionization source of PGD-IMS.

A stir bar hold device was designed, and the schematic diagram of the device is illustrated in Fig. 2. The device comprises a handle, a

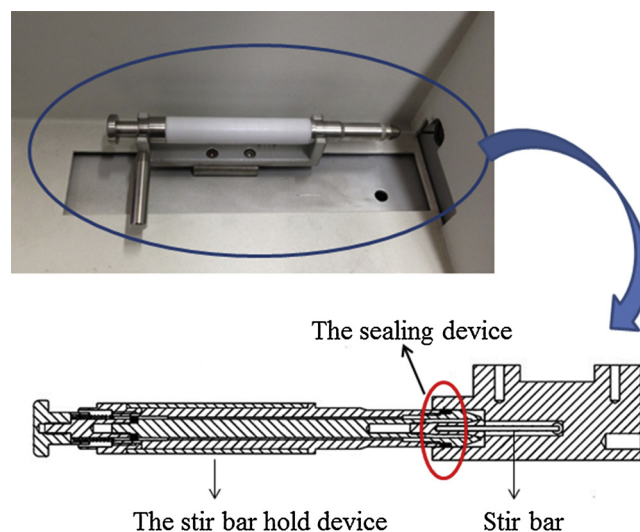


Fig. 1. The schematic diagram of PGD-IMS.

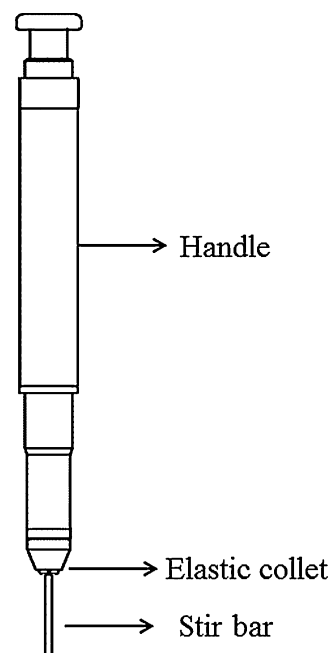


Fig. 2. The schematic diagram of the stir bar hold device.

stir bar and an elastic collet. The elastic collet is arranged at one end of the handle and is linked with the handle. When the elastic collet is opened by operating the handle, a stir bar can be taken out from or put into the elastic collet; and when the elastic collet is shrunk by operating the handle, the stir bar is clamped by the elastic collet.

The experimental parameters for obtaining the ion mobility spectra of pesticides are summarized in Table 1, and the conditions listed herein are adopted for all experiments outlined in the present work.

2.2. Chemicals and reagents

Pesticide standards of atrazine, simazine and ametryn were received from Pesticide Analysis Laboratory, China Agricultural University (Beijing, China). The purities of atrazine, simazine and ametryn were 99.0%, 98.5% and 99.8%, respectively. Individual stock standard solutions of pesticide standards were prepared in acetonitrile (1000 mg L^{-1}) and stored at -20°C . Individual working

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