



Determination of triazine herbicides in vegetables by ionic liquid foam floatation solid phase extraction high performance liquid chromatography



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ARTICLE INFO

Article history:

Received 20 August 2013

Received in revised form 21 January 2014

Accepted 10 February 2014

Available online 19 February 2014

Keywords:

Ionic liquids

Foam floatation

Triazine herbicides

Solid phase extraction

Vegetable

ABSTRACT

The ionic liquid foam floatation solid phase extraction was established and applied to the extraction of six triazine herbicides, including desmetryn, secbumeton, terbumeton, terbuthylazine, dimethametryn and dipropetryn, in vegetable samples. To obtain the optimized experimental parameters, the effects of pH value of sample solution, the type and concentration of ionic liquid, the flow rate of carrier gas, foam floatation time, the type of solid phase extraction cartridge, the type and volume of elution solvent on the recoveries of the analytes were examined. The high performance liquid chromatography was applied to the determination of the analytes. Under the optimized experimental conditions, the linearities for determining the analytes were satisfactory and the limits of detection for desmetryn, secbumeton, terbumeton, terbuthylazine, dimethametryn and dipropetryn were 2.50, 1.75, 2.76, 1.87, 1.36 and 1.44 $\mu\text{g kg}^{-1}$, respectively. The recoveries of the analytes ranged from 78.64% to 104.37% and the relative standard deviations ranged from 1.44 to 6.45%. The real samples were analyzed and the results were satisfactory.

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1. Introduction

Triazine herbicides are extensively used around the world by virtue of their properties of inhibiting photosynthetic reactions to help control the growth of grasses and broadleaf weeds [1]. However, they may be dangerous for human health. A large number of researches have proven the toxicity of triazine herbicides, which have been suspected of giving rise to cancer, congenital defect and so on [2–4]. Triazine herbicides and their metabolites and degradation products can pollute the crop itself and the environment of crop growth, such as soil and natural water. When these harmful substances pollute the foodstuffs, they can seriously endanger human health.

Many countries and regions have established the criteria of maximum residue limits (MRLs) of triazine herbicides. The Environmental Protection Agency (EPA) has provided that the MRLs of triazine herbicides in most products are 0.25 mg kg^{-1} , while the European Union (EU) dictated that the MRL of terbuthylazine in vegetables is 0.05 mg kg^{-1} . The simple, rapid and sensitive method

for extraction, separation and determination of the triazine herbicides in vegetable samples is required.

In case of the complex matrix sample and low analyte content, the extraction and concentration should be more important than the determination. In recent years, more and more extraction methods have been developed, such as liquid–liquid extraction (LLE) [5–7], solid phase extraction (SPE) [8–11], solid phase micro-extraction (SPME) [12], pressurized microwave-assisted extraction (PMAE) [13], dynamic microwave-assisted extraction-solidification of floating organic drop (DMAE-SFOD) [14], cloud point extraction (CPE) [15], matrix solid phase dispersion (MSPD) [16], molecularly imprinted solid phase extraction (MISPE) [17], dispersive solid-phase extraction (DSPE) [18] and pressurized liquid extraction (PLE) [19,20]. Recently, magnetic nanoparticles (MNPs) were used as the adsorbents in many fields and successfully used for the pre-concentration and removal of some toxic and hazardous pollutants from water, soil and biological samples [21–31].

The methods based on ionic liquids (ILs) were developed for the extraction of triazine herbicides from different liquid samples (milk, honey, yogurt), such as microwave-assisted ionic liquid micro-extraction (MAILME) [32], dispersive liquid–liquid micro-extraction (DLLME) [33] and ionic liquid foaming-based solvent

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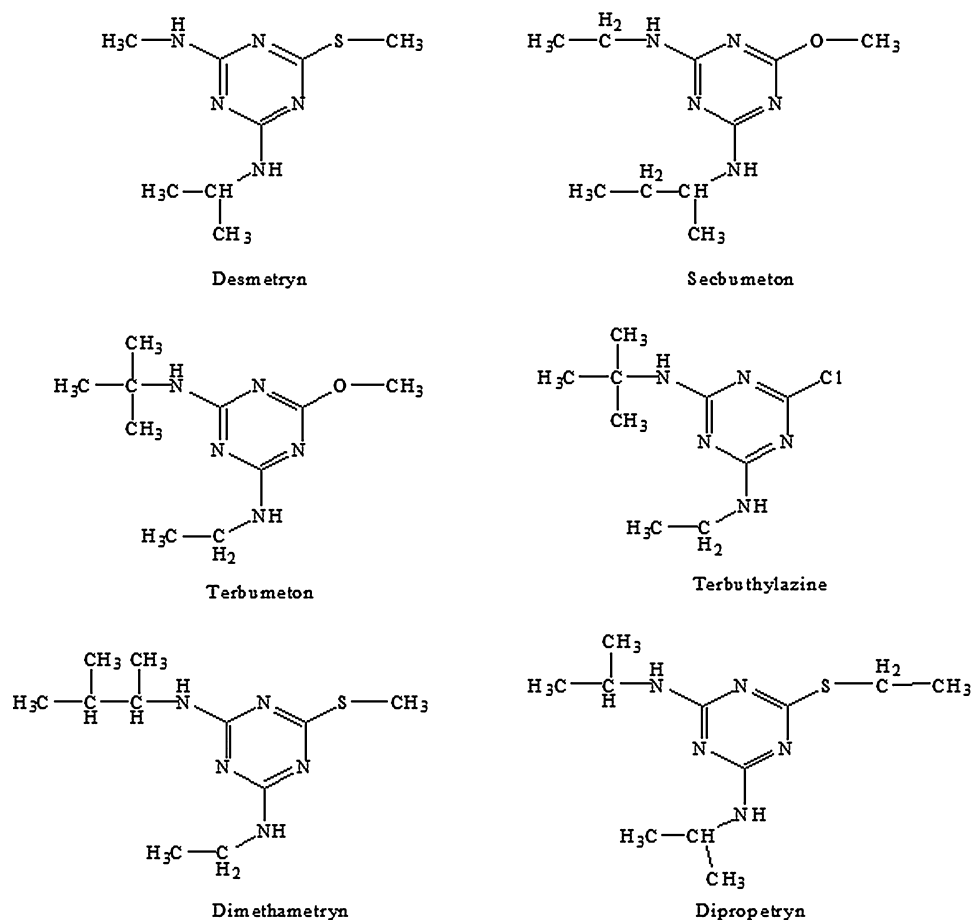


Fig. 1. Structures of triazine herbicides.

floatation (ILF-SF) [34]. Determination of triazine herbicides in water and food samples, especially in vegetable samples, was predominantly performed by high performance liquid chromatography (HPLC) [20], gas chromatography (GC) [35], HPLC mass spectrometry (HPLC-MS) [36], GC-MS [37] and micellar electrokinetic capillary chromatography (MECC) [38].

In this paper, the extraction and determination of triazine herbicides in vegetable samples by ionic liquid foam floatation solid phase extraction (ILFF-SPE) high performance liquid chromatography (HPLC) was developed. This method was based on the fact that IL has the characteristics of surfactant [39]. The ILFF-SPE was applied to the extraction of triazine herbicides in vegetables. IL-based FF-SPE was applied to the extraction of steroid hormones in water sample [39]. There is no report about application of ILFF-SPE in triazine herbicides in vegetable samples. In the present study, the ILFF-SPE was applied directly to solid and large volume sample. At the same time, sample pretreatment steps and extraction time were reduced because of the combination of extraction method and enrichment method.

2. Materials and methods

2.1. Chemicals and reagents

Desmetryn, sebumeton, terbumeton, terbutylazine, dimethametryn, and dipropetryn were obtained from National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China). The structures of the triazine herbicides are

shown in Fig. 1. 1-Ethyl-3-methylimidazolium tetrafluoroborate ([C₂MIM][BF₄]), 1-butyl-3-methylimidazolium tetrafluoroborate ([C₄MIM][BF₄]), 1-hexyl-3-methylimidazolium tetrafluoroborate ([C₆MIM][BF₄]), 1-Octyl-3-methylimidazolium tetrafluoroborate ([C₈MIM][BF₄]), 1-butyl-3-methylimidazolium hexafluorophosphate ([C₄MIM][PF₆]), 1-hexyl-3-methylimidazolium hexafluorophosphate ([C₆MIM][PF₆]), and 1-octyl-3-methylimidazolium hexafluorophosphate ([C₈MIM][PF₆]) were obtained from Chengjie Chemical Co. Ltd. (Shanghai, China).

Chromatographic grade methanol and acetonitrile were purchased from Fisher Scientific Company (UK). All other reagents of analytical grade were purchased from Beijing Chemical Factory (Beijing, China). Pure water was obtained with a Milli-Q water purification system (Millipore Co., USA).

2.2. Apparatus

HPLC analysis was carried out on a LC chromatographic system equipped with a binary pump (LC-20AB) and UV detector (Shimadzu Corporation, Kyoto, Japan). Chromatographic separation of target analytes was performed on Agilent Eclipse XDB-C18 column (150 mm × 4.6 mm i.d., 3.5 μm, Agilent, USA). Oasis HLB (3 mL, 100 mg) and MCX (3 mL, 100 mg) extraction cartridges were purchased from Waters (Milford, MA, USA). RE-52AA vacuum rotary evaporator (Yarong, Shanghai, China) were used. SPE cartridges, including SuperClean Al₂O₃ (3 mL, 100 mg), SuperClean C18 (3 mL, 100 mg), and SuperClean Si (3 mL, 100 mg), were purchased from Supelco (Bellefonte, PA, USA).

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