



Preparation of stir cake sorptive extraction based on polymeric ionic liquid for the enrichment of benzimidazole anthelmintics in water, honey and milk samples



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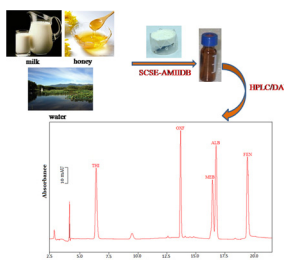
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HIGHLIGHTS

- A new polymeric ionic liquid-based monolith was prepared.
- The monolith was used as the extractive medium of stir cake sorptive extraction.
- The SCSE-AMIIDB can extract benzimidazole anthelmintics (BAs) effectively.
- A combination of SCSE-AMIIDB-LD-HPLC/DAD was developed.
- The combination was applied to monitor trace BAs in water, milk and honey samples.

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, a new stir cake sorptive extraction (SCSE) using polymeric ionic liquid monolith as sorbent was prepared. The sorbent was obtained by in situ copolymerization of an ionic liquid, 1-allyl-3-methylimidazolium bis[(trifluoro methyl)sulfonyl]imide (AMII) and divinylbenzene (DB) in the presence of *N,N*-dimethylformamide. The influence of the content of ionic liquid and the porogen in the polymerization mixture on extraction performance was studied thoroughly. The physicochemical properties of the polymeric ionic liquid were characterized by infrared spectroscopy, elemental analysis, scanning electron microscopy and mercury intrusion porosimetry. The usefulness of SCSE-AMIIDB was demonstrated by the enrichment of trace benzimidazole anthelmintics. Several parameters affecting the extraction efficiency were investigated, and under the optimized conditions, a simple and effective method for the determination of trace benzimidazoles residues in water, milk and honey samples was established by coupling SCSE-AMIIDB with high performance liquid chromatography/diode array detection (SCSE-AMIIDB-HPLC/DAD). Results indicated that the limits of detection ($S/N=3$) for target compounds were $0.020\text{--}0.072\ \mu\text{g L}^{-1}$, $0.035\text{--}0.10\ \mu\text{g L}^{-1}$ and $0.026\text{--}0.076\ \mu\text{g L}^{-1}$ in water, milk and honey samples, respectively. In addition, an acceptable reproducibility was achieved by evaluating the repeatability and intermediate precision with relative standard deviations (RSD) of less than 9% and 11%, respectively. Finally, the established AMII-SCSE-HPLC/DAD method was successfully applied for the determination of benzimidazoles residues in milk, honey and environmental water samples. Recoveries obtained for the determination of benzimidazole anthelmintics in spiking samples ranged from 70.2% to 117.6%, with RSD below 12% in all cases.

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1. Introduction

Benzimidazole anthelmintics (BAs) are broad-spectrum drugs that include thiabendazole, oxfendazole, mebendazole, albendazole and fenbendazole, etc. BAs have been widely used to control gastrointestinal nematodes and lungworms, and are also commonly used in agriculture, aquaculture, and veterinary practices for protecting and treating these parasitic infections [1–3]. Improper uses of these BAs by inappropriate period and excess administration may result in their residues to be present in environmental water, edible tissue and animal-derived food products such as milk and honey [4]. Several toxic effects have been associated with a chronic exposure to benzimidazole compounds, such as congenital malformations, polyploidy, diarrhea, pulmonary edemas, and necrotic lymphadenopathy [5]. Therefore, to control the amount of BAs that can be used under certain circumstances and the maximum amounts that can be tolerated in drinking water or food, maximum concentration level (MCL) or maximum residue limits (MRLs) have been set by governments and organizations. For example, the European Water Framework Directive has established a MCL of $0.1 \mu\text{g L}^{-1}$ for most benzimidazole compounds present in natural waters [6]. European Community legislation has set the MRLs for oxfendazole and mebendazole in milk were 10 and $50 \mu\text{g L}^{-1}$, respectively [7]. The Japanese “Positive List” system set the MRL for mebendazole in honey was $20 \mu\text{g L}^{-1}$ (<http://www.mhlw.go.jp/english/topics/foodsafety/positivelist060228/index.html>). Therefore, it is vital to develop simple and sensitive method to monitor the residues of BAs in water and food.

Among the monitoring tools of BAs residues, high performance liquid chromatography coupled with ultraviolet (UV) detector [8–12] or mass spectrometry (MS) [13–20] is the most popular analytical technology. However, sample preparation is necessary prior to the HPLC analysis. In the sample preparation procedure, the target analytes can be enriched and the interferences in sample matrix will be diminished. So far, several sample preparation techniques such as solid-phase extraction (SPE) [8–10,16,17], stir bar sorptive extraction (SBSE) [11], liquid–liquid extraction (LLE) [18], dispersive liquid–liquid microextraction (DLLME) [21], cloud-point extraction (CPE) [22] and supercritical fluid extraction (SFE) [23] have been applied for the analysis of BAs. However, most of these methods have some limitations including low extraction capacity, being time consuming and using toxic organic solvents. Hereby, there is still a necessary to develop sample pretreatment method with simplicity, high extraction capacity and environmental friendliness for the monitoring of BAs residues in complicated samples.

In our previous studies, a promising extraction and enrichment methodology named stir cake sorptive extraction (SCSE) with porous monolith as sorbent was developed [11,24,25]. The SCSE integrates extraction, enrichment and clean-up into one step. There are some features of SCSE including simple operation, high cost-efficiency, friendly to environment and excellent longevity of extractive medium. At the same time, SCSE possesses expected extraction capacity because more sorbent can be utilized. However, longer time should be spent in order to reach extraction equilibrium. It is worthy to be noted that SCSE is very flexible. According to the character of target analytes, the extraction medium-monolithic cake can be easily designed and prepared to realize effective extraction of analytes. In this study, five BAs were selected as target analytes. According to the structural characters of BAs, there are hydrophobic groups–phenyl groups and strongly polar amide and imidazole groups (Table S1). An ionic liquid, 1-allyl-3-methylimidazolium bis[(trifluoro methyl)sulfonyl]imide (AMII) was used as monomer to copolymerize with divinylbenzene to form monolithic cake (AMII–DB). In the monolith, the aromatic

ring can interact with analytes through π – π conjugation, the amino groups can produce hydrogen-bond and dipole–dipole interactions with amide and imidazole groups of BAs. Hereby, it is reasonable to expect that SCSE–AMII/DB can extract BAs effectively. After the optimization of polymerization and extraction conditions, a convenient and sensitive methodology combining SCSE–AMII/DB and liquid desorption (LD), followed by high performance liquid chromatography with diode array detection (SCSE–AMII/DB–LD–HPLC/DAD) for the direct analysis of trace BAs in water, milk and honey samples was developed.

2. Experimental

2.1. Chemicals

1-Allyl-3-methylimidazolium bis[(trifluoro methyl)sulfonyl]imide (AMII) (98%) was purchased from Cheng Jie Chemical Co. Ltd. (Shanghai, China); divinylbenzene (DB) (80%) was supplied by Alfa Aesar Ltd. (Tianjin, China); azobisisobutyronitrile (AIBN) (97%, recrystallized before use), *N,N*-dimethylformamide (DMF) (98%), trifluoroacetic acid (TFA) (99%) and formic acid (FA) (97%) were purchased from Shanghai Chemical Co. (China); HPLC-grade acetonitrile (ACN) and methanol were purchased from Tedia Company (Fairfield, USA); water used throughout the study was purified using a Milli-Q water purification system (Millipore, USA).

Fenbendazole (FEN), thiabendazole (THI), mebendazole (MEB), albendazole (ALB) and oxfendazole (OXF) were supplied by National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China). The chemical properties of the above analytes are shown in Table S1. Water samples were collected from Xiamen city and filtrated through $0.45 \mu\text{m}$ membranes. Milk and honey samples with different brands were purchased from locally retail markets. All samples were stored at -4°C before use.

Individual stock solutions of BAs were prepared at a concentration of 10.0mg L^{-1} by dissolving methanol and renewed monthly. Working solutions (containing a standard mixture for each benzimidazole) were prepared at a concentration of $100 \mu\text{g L}^{-1}$ to validate the method.

2.2. Instruments

HPLC analyses were carried out on a LC chromatographic system (Shimadzu, Japan) equipped with a binary pump (LC-20AB) and a diode array detector (SPD-M20A). Sample injection was carried out using a RE3725i manual sample injector with a $20 \mu\text{L}$ loop (Rheodyne, Cotati, CA, USA), all experiments were performed at room temperature.

The morphologies of monolithic materials were examined by a Model XL30 scanning electron microscopy (SEM) instrument (Philips, Eindhoven, The Netherlands). The pore size distribution (PSD) of the monolith was measured on mercury intrusion porosimeter Model PoreMaster-60 (Quantachrome instruments, Florida, USA). Elemental analysis (EA) was carried out on PerkinElmer (Shelton, CT, USA) Model PE 2400. FT-IR was performed on an Avatar-360 FT-IR instrument (Thermo Nicolet, Madison, WI, USA).

2.3. Chromatographic conditions

The separation of BAs was performed on a Hypersil C18 column ($5 \mu\text{m}$ particle size, $250 \text{mm} \times 4.6 \text{mm}$ i.d.). Optimum separation was obtained with a binary mobile phase composed of Milli-Q water (solvent A, with 0.1% formic acid) and ACN (solvent B). The gradient elution program was as follows: 0–6.0 min = 20% B, 6.0–12.0 min = 20% B–45% B and kept to 16 min, 16.0–17.0 min = 45%

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