Determination of benzimidazole residues in animal tissue samples by combination of magnetic solid-phase extraction with capillary zone electrophoresis

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ARTICLE INFO

Article history:
Received 2 October 2011
Received in revised form 13 December 2011
Accepted 14 December 2011
Available online 20 December 2011

Keywords:
Benzimidazole drugs
Magnetic solid-phase extraction
Fe3O4/SiO2/poly (MAA-co-EGDMA) magnetic microspheres
Capillary zone electrophoresis
Field-amplified sample stacking

ABSTRACT

Benzimidazole drugs (BZDs) comprise a large number of synthetic anthelmints, which are widely used in food-producing animals for prophylactic and therapeutic purposes. To protect consumers from the risks related to BZDs residues, a simple, rapid, and efficient method for simultaneous determination of ten BZDs in animal tissues samples was developed. This analytical procedure involved extracting samples with magnetic solid-phase extraction (MSPE) using magnetite/silica/poly (methacrylic acid-co-ethylene glycol dimethacrylate) (Fe3O4/SiO2/poly (MAA-co-EGDMA)) magnetic microspheres, and determination by capillary zone electrophoresis (CZE). To improve the sensitivity of the method, we employed the electrokinetic injection with field-amplified sample stacking technique (FASS). Berbine solution was used as internal standard to minimize the fluctuation of analytical results. Under the optimized extraction conditions, good linearities were obtained for the ten BZDs with the correlation coefficients ($R^2$) above 0.9920. The limits of detections (LODs) for ten BZDs were 1.05–10.42 ng/g in swine muscle and 1.06–12.61 ng/g in swine liver, respectively. The intra- and inter-day relative standard deviations (RSDs) of the developed method were less than 13.6%. The recoveries of the ten BZDs for the spiked samples ranged from 81.1% to 105.4% with RSDs less than 9.3%.

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1. Introduction

Benzimidazole drugs (BZDs) comprise a large number of synthetic anthelmints, which are widely used in food-producing animals for therapeutic and prophylactic purpose [1–3]. Improper use of BZDs, such as inappropriate withdrawal period and excessive administration, may result in their residues in animal tissues and animal-derived food products. A chronic exposure to BZDs has been associated with several toxic effects such as teratogenicity, congenic malformations, polyptoidy, diarrhea, anemia, pulmonary edemas, or necrotic lymphadenopathy [4]. To protect consumers from the risks related to BZDs residues, maximum residue limits (MRLs) for marker residue (the sum of a parent drugs and/or their metabolites) in animal products have been set by Codex Alimentarius Commission (CAC) and China [5,6], and the values usually range from 10 to 5000 μg/kg according to the different compounds and biological matrix. For example, both of the MRLs for thiabendazole (TBZ)-related drugs (sum of TBZ and 5-OH-TBZ) have been set at 100 μg/kg for muscle and liver. Due to the complexity of samples and the differences of lipophilicity and pKa values among BZDs, the multi-residue determination of BZDs in biological matrices is still a challenge. Therefore, the development of sensitive, rapid, and inexpensive analytical method is required [3].

Hereo, the combination of high performance liquid chromatography (HPLC) and mass spectrometry (MS) has become the favored technique for monitoring a wide range of BZDs for highly sensitive and selective determination in edible animal tissues and animal-derived products [1,7–12], while the MS detections are very expensive and not widely applicable for a common laboratory. Gas chromatography (GC) was also applied while extra derivatization step of residues to sufficiently volatilize was required [3]. As a powerful complementary separation technique, capillary electrophoresis (CE) has also been applied in the separation and determination of BZDs due to its unique advantages such as rapid analysis, excellent separation efficiency, versatile separation mode, low cost, and less need for sample and organic solvents [13–15]. However, analytical sensitivity by CE is inferior to HPLC due to both the small injection volumes and the short optical path length.
81.1–105.4% with RSDs less than 9.3% (as shown in Table 4). These results demonstrated that the proposed method was reliable for the determination of the BZDs residues in swine tissue samples. In addition, the Fe₃O₄/SiO₂/poly (MAA-co-EGDMA) magnetic microspheres can be recycled and no significant change was observed on extraction efficiency after repeated extraction/desorption cycles.

### 4. Conclusion

A simple, fast and sensitive method for the determination of ten BZDs in swine muscle and liver samples was established by combining Fe₃O₄/SiO₂/poly (MMA-co-EGDMA) magnetic microspheres with FASS–CZE technique. The result showed the magnetic microspheres had obvious preconcentration ability for the ten BZDs. Satisfactory results were obtained with regard to sensitivity, accuracy and precision. Compared with other previously reported methods, the MSPE–FASS–CZE method is inexpensive and applicable for a common laboratory detection of BZDs. Furthermore, this method is effective on removing the potential interferences in edible animal tissue since no interference was observed for the quantification of BZDs. Therefore, the MSPE–FASS–CZE method was adequate for the routine residue monitoring of BZDs in swine tissue samples, and the combination of MSPE and FASS–CE provides a practical tool for veterinary multi-residue determination.

### Acknowledgements

This work was partly supported by grants from the Fundamental Research Funds for the Central Universities, the National Natural Science Foundation of China (91017013, 31073027, 21005057) and the Science Fund for Creative Research Groups (No. 20921062), NSFC.

### References