VERSITA

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Simultaneous determination of 32 antibiotics in aquaculture products using LC-MS/MS

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An analytical multiclass, multi-residue method for the determination of antibiotics in aquaculture products was developed and validated. A fast, cheap, and straightforward extraction procedure followed by liquid chromatography–tandem mass spectrometry analysis was proposed. This method covers 32 antibiotics of different classes, which are frequently used in aquaculture. Three different extraction procedures were compared, and the extraction with acetonitrile (0.1 vol. % formic acid) showed the best results. The selected extraction procedure was validated at four different fortification levels (10 µg kg^{-1}, 25 µg kg^{-1}, 50 µg kg^{-1}, and 100 µg kg^{-1}). Recoveries of the tested antibiotics ranged from 70 % to 120 %, with the relative standard deviation (RSD) of triplicates lower than 20 %. The limits of quantification (LOQ) ranged from 0.062 µg kg^{-1} to 4.6 µg kg^{-1}, allowing for the analysis of trace levels of these antibiotics in aquaculture products. The method was applied to the analysis of selected antibiotics in fish and shrimp meat available in the Czech market.
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Introduction

Aquaculture is a fast-growing food production sector. The increasing demand for fish products has promoted the intensification of aquaculture production in many countries (Cañada-Cañada et al., 2009) which has led to the wide application of antibiotics, used for both prevention and treatment of bacterial diseases. Excessive use of antibiotics in industrial aquaculture results in the presence of residual antibiotics in commercial fish and shellfish products (Cabello, 2006). Unintentional consumption of antibiotics can lead to the development of antibiotic resistance in bacteria that are pathogenic to humans (Greenlees, 2003). The maximum residue limits (MRLs) for antibiotics in different tissues are set in the annexes of the European Union (EU) directive No 37/2010 (European Commision, 2010).

To monitor the occurrence of these undesirable residues in edible tissues, a simple and reliable analytical method is required. Currently, liquid chromatography (LC) techniques coupled with mass spectrometric (MS) detection are used (Li et al., 2011; Peters et al., 2009; Romero-González et al., 2007; Yu et al., 2011). Most of the published methods are focused on one or two antibiotic groups (Dasenaki & Thomaidis, 2010; Jo et al., 2011; Johnston et al., 2002; Nakazawa et al., 1999). However, the current analytical strategy is shifting towards multi-residue and multiclass methods, which save time because all the target antibiotics are analyzed in one run. At present, such methods are uncommon. The extraction process is the limiting factor of any multi-residual method, since it should provide acceptable recoveries of analytes with a broad range of physicochemical properties. Moreover, it should be simple, fast and cheap, to increase the sample throughput.

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