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A single-step solid phase extraction for the simultaneous determination of 8 mycotoxins in fruits by ultra-high performance liquid chromatography tandem mass spectrometry

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Abstract

A simple and rapid extraction procedure for the simultaneous determination of eight mycotoxins (*Alternaria* toxins, ochratoxin A, patulin, citrinin) in a variety of fruit matrices has been developed using ultra high performance liquid chromatography coupled to tandem mass spectrometry. The procedure involves a one-step cleanup using homemade solid phase extraction (SPE) cartridges. By comparative evaluation among six various adsorbents (C18, PSA, HLB, MCX, Silica, NH_2), the combination of MCX and NH_2 was found to provide the most effective cleanup, removing the greatest number of matrix interferences and also allowing the quantification of all analyzed mycotoxins in fruits. The optimized extraction conditions including acidified aqueous acetonitrile and an additional salt-out step using NaCl were employed before SPE cleanup. Method validation was performed by analyzing samples spiked at three levels (LOQ, 2 LOQ and 10 LOQ). Four fruits including apple, sweet cherry, tomato and orange fruits were selected, and accuracy (recovery%), precision (RSD%), limits of quantification (LOQ), linearity and matrix effect were evaluated during validation. Matrix-matched linearity with correlation coefficients $\geq$0.9921 was established in the range of 5-200 ng mL^{-1} for patulin and 1-200 ng mL^{-1} for other mycotoxins, respectively. Recoveries between 74.2% and 102.4% and relative standard deviations lower than 4.7% were obtained for all tested fruits. The matrix effect observed was low ($\leq \pm$17%) in all three fruit matrixes with the exception of orange, for which strong ion suppression was observed for alternariol (25.3%), ochratoxin A (31.6%) and citrinin (40.3%). Therefore, matrix-matched calibration was used for a correct quantification in order to compensate for matrix effect. The limits of quantification (LOQ), ranging from 1 to 5 μg kg^{-1} depending on mycotoxins type, were always lower than maximum permitted levels for every regulated mycotoxin by the current European legislation.

Keywords

Mycotoxin, SPE cleanup, UPLC-MS/MS, Fruits.

1. Introduction