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18  
19 Abstract

20 A simple and rapid extraction procedure for the simultaneous determination of  
21 eight mycotoxins (*Alternaria* toxins, ochratoxin A, patulin, citrinin) in a  
22 variety of fruit matrices has been developed using ultra high performance  
23 liquid chromatography coupled to tandem mass spectrometry. The procedure  
24 involves a one-step cleanup using homemade solid phase extraction (SPE)  
25 cartridges. By comparative evaluation among six various adsorbents (C18,  
26 PSA, HLB, MCX, Silica, NH<sub>2</sub>), the combination of MCX and NH<sub>2</sub> was found  
27 to provide the most effective cleanup, removing the greatest number of matrix  
28 interferences and also allowing the quantification of all analyzed mycotoxins  
29 in fruits. The optimized extraction conditions including acidified aqueous  
30 acetonitrile and an additional salt-out step using NaCl were employed before  
31 SPE cleanup. Method validation was performed by analyzing samples spiked  
32 at three levels (LOQ, 2 LOQ and 10 LOQ). Four fruits including apple, sweet  
33 cherry, tomato and orange fruits were selected, and accuracy (recovery%),  
34 precision (RSD%), limits of quantification (LOQ), linearity and matrix effect  
35 were evaluated during validation. Matrix-matched linearity with correlation  
36 coefficients  $\geq 0.9921$  was established in the range of 5-200 ng mL<sup>-1</sup> for patulin  
37 and 1-200 ng mL<sup>-1</sup> for other mycotoxins, respectively. Recoveries between  
38 74.2% and 102.4% and relative standard deviations lower than 4.7% were  
39 obtained for all tested fruits. The matrix effect observed was low ( $\leq \pm 17\%$ ) in  
40 all three fruit matrixes with the exception of orange, for which strong ion  
41 suppression was observed for alternariol (25.3%), ochratoxin A (31.6%) and  
42 citrinin (40.3%). Therefore, matrix-matched calibration was used for a correct  
43 quantification in order to compensate for matrix effect. The limits of  
44 quantification (LOQ), ranging from 1 to 5  $\mu\text{g kg}^{-1}$  depending on mycotoxins  
45 type, were always lower than maximum permitted levels for every regulated  
46 mycotoxin by the current European legislation.

47  
48 Keywords

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

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51 1. Introduction

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