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Review

Trace mycotoxin analysis in complex biological and food matrices by liquid chromatography–atmospheric pressure ionisation mass spectrometry

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Abstract

Mycotoxins are toxic secondary metabolites produced by filamentous fungi that are growing on agricultural commodities. Their frequent presence in food and their severe toxic, carcinogenic and estrogenic properties have been recognised as potential threat to human health. A reliable risk assessment of mycotoxin contamination for humans and animals relies basically on their unambiguous identification and accurate quantification in food and feedstuff. While most screening methods for mycotoxins are based on immunoassays, unambiguous analyte confirmation can be easily achieved with mass spectrometric methods, like gas chromatography/mass spectrometry (GC/MS) or liquid chromatography/mass spectrometry (LC/MS). Due to the introduction of atmospheric pressure ionisation (API) techniques in the late 80s, LC/MS has become a routine technique also in food analysis, overcoming the traditional drawbacks of GC/MS regarding volatility and thermal stability. During the last few years, this technical and instrumental progress had also an increasing impact on the expanding field of mycotoxin analysis. The aim of the present review is to give an overview on the application of LC–(API)MS in the analysis of frequently occurring and highly toxic mycotoxins, such as trichothecenes, ochratoxins, zearalenone, fumonisins, aflatoxins, enniatins, moniliformin and several other mycotoxins. This includes also the investigation of some of their metabolites and degradation products. Suitable sample pre-treatment procedures, their applicability for high sample through-put and their influence on matrix effects will be discussed. The review covers literature published until July 2006.
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Keywords: Mycotoxin; Metabolite; Degradation product; Bioadduct; Bioconjugate; Trichothecene; Ochratoxin; Zearalenone; Zeranol; Fumonisin; Aflatoxin; Patulin; Moniliformin; Enniatin; Beauvericin; Mycophenolic acid; Mass spectrometry; High-performance liquid chromatography; LC/MS; Electrospray ionisation; Atmospheric pressure ionisation; Atmospheric pressure chemical ionisation; Atmospheric pressure photoionisation; ESI; APCI; APPI

Abbreviations: 3-/15-AcDON, 3-/15-acetyldesoxynivalenol; ACN, acetonitrile; AcOH, acetic acid; AFB_{1-2}, aflatoxin B_{1-2}; AFG_{1-2}, aflatoxin G_{1-2}; AFM_1, aflatoxin M_1; AFQ_1, aflatoxin Q_1; AFP_1, aflatoxin P_1; APCI, atmospheric pressure chemical ionisation; API, atmospheric pressure ionisation; APPI, atmospheric pressure photo ionisation; BEA, beauvericin; CID, collision induced dissociation; CRM, consecutive reaction monitoring; DAS, diacetoxyscirpenol; DOM-1, deepoxydesoxynivalenol; DON, desoxynivalenol; EA, EA_1, enniatin A, enniatin A_1; EB, EB_1, enniatin B, enniatin B_1; ELISA, enzyme-linked immuno sorbent assay; ESI, electrospray ionisation; FAB, fast atom bombardment; FA_{1-3}, fumonisin A_{1-3}; FAK_1, fumonisin AK_1; FB_{1-5}, fumonisin B_{1-5}; FC_{1-4}, fumonisin C_{1-4}; FD, fumonisin D; FP_{1-3}, fumonisin P_{1-3} = 3-hydroxypyridinium FC_{1-3}; FDA, food and drug administration; FL, fluorescence detection; FUS, fusaproliferin; F-X, fusarenone X; GC–ECD, gas chromatography–electron capture detection; GC/MS, gas chromatography/mass spectrometry; HFB_1, fully hydrolysed fumonisin B_1; HT-2, HT-2 toxin; IAC, immunoaffinity chromatography; LC/MS, liquid chromatography/mass spectrometry; LOD, limit of detection; LOQ, limit of quantification; MAS, monoacetoxyscirpenol; MeOH, methanol; MON, moniliformin; MPA, mycophenolic acid; MS/MS, tandem mass spectrometry; NanoESI, nano electrospray ionisation; NEO, neosolaniol; NIV, nivalenol; OTA, ochratoxin A; OTB, ochratoxin B; OTC, ochratoxin C; OTα, ochratoxin α; OTβ, ochratoxin β; PB, particle beam; $PHFB_1$, partially hydrolysed FB_1; QTrap, combination of triple quadrupole and ion trap mass analyser; RP, reversed-phase; SFC, supercritical fluid chromatography; SFE, supercritical fluid extraction; SIM, selected ion monitoring; SPE, solid-phase extraction; SRM, selected reaction monitoring; T-2, T-2 toxin; TFA, trifluoro acetic acid; TLC, thin-layer chromatography; TSP, thermospray; VER, verrucarol; WHO, World Health Organisation; ZAN, zearalanone; α-ZAL, α-zearalanol = zeranol; β-ZAL, β-zearalanol = taleranol; α-ZOL, α-zearalenol; β-ZOL, β-zearalenol; ZON, zearalenone

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Contents

1. Introduction

Mycotoxins are toxic secondary metabolites produced by about 200 identified filamentous fungi, as e.g. *Fusarium*, *Aspergillus* and *Penicillium* species, growing under a wide range of climatic conditions on agricultural commodities (grains, spices, fruits, coffee, nuts, etc.) in the field and during storage [1,2]. Their occurrence in food, beverages and feed has

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