

Dispersive liquid-liquid microextraction DLLME and LC-MS/MS analysis for multi-mycotoxin in rice bran: method development, optimisation and validation

ABSTRACT

Rice bran, a by-product of the rice milling process, has emerged as a functional food and being used in formulation of healthy food and drinks. However, rice bran is often contaminated with numerous mycotoxins. In this study, a method to simultaneous detection of aflatoxins (AFB1, AFB2, AFG1, and AFG2), ochratoxin A (OTA), deoxynivalenol (DON), fumonisins (FB1 and FB2), sterigmatocystin (STG), T-2 toxin, HT-2 toxin, diacetoxyscirpenol (DAS) and zearalenone (ZEA) in rice bran was developed, optimized and validated using dispersive liquid-liquid microextraction (DLLME) and liquid chromatography-tandem mass spectrometry (LC-MS/MS). In DLLME, using a solvent mixture of methanol/water (80:20, v/v) as the dispersive solvent and chloroform as the extraction solvent with the addition of 5% salt improved the extraction recoveries (63–120%). The developed method was further optimized using the response surface methodology (RSM) combined with Box–Behnken Design (BBD). Under the optimized experimental conditions, good linearity was obtained with a correlation coefficient (r^2) \geq 0.990 and a limit of detection (LOD) between 0.5 to 50 ng g⁻¹. The recoveries ranged from 70.2% to 99.4% with an RSD below 1.28%. The proposed method was successfully applied to analyze multi-mycotoxin in 24 rice bran samples.

Keyword: Dispersive liquid-liquid microextraction; Multi-mycotoxin; Liquid chromatography mass spectrometry; Response surface methodology; Rice bran