

DEVELOPMENT AND VALIDATION OF A MULTI-METHOD FOR THE ANALYSIS OF 36 MYCOTOXINS AND 149 PESTICIDES IN MAIZE USING UPLC-MS/MS (ESI⁺)

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Maize represents one of the most produced crops in the world. In 2007, 800 million tons were produced, and the USA was responsible for almost half of this amount. Brazil is the 3rd largest world producer and the largest in Latin America¹. Maize is widely used as primary base for human and animal consumption products, which makes necessary a strict control of residues and contaminants in this crop. One of the main risk factors is the presence of mycotoxins, potentially carcinogenic substances produced by certain species of fungus, which are commonly present in maize¹. The main mycotoxins present in maize are fumosin and aflatoxin types, which are very toxic to human been.

The purpose of this work is to develop and validate a method capable to extract a large number of mycotoxins, and concurrently also pesticides. The analytical detection method performed was UPLC-MS/MS (ESI⁺). An acetonitrile-based extraction (modified QuEChERS²) procedure was used and no clean-up step was applied. A slurry containing maize flour and water (1:1.5 w/w) was made and then extracted with acetonitrile/acetic acid (1%), followed by addition of MgSO₄ for partitioning. The mycotoxins were divided in two groups for the fortification step: Group 1 with 15 mycotoxins (1, 2 and 10 µg kg⁻¹ spike levels), and Group 2 with 18 mycotoxins (50, 100 and 400 µg kg⁻¹ spike levels). The pesticides were spiked at the levels of 10, 20 and 50 µg kg⁻¹. Accuracy (as % recovery), precision (RSD %), and method LOQ, as well as instrument LOD and matrix effects, were determined.

Group 1 showed 2, 3 and 6 mycotoxins with acceptable method performance for quantification (recoveries between 70-120% and RDS ≤ 20%), for the spike levels 1, 2 and 10 µg kg⁻¹, respectively. Group 2 presented 4, 5 and 5 mycotoxins which fulfilled the requirements at the 50, 100 e 400 µg kg⁻¹ spike levels, respectively. The matrix effects were, for most mycotoxins, insignificant or acceptable and did not influence the results.

The numbers of pesticides that showed acceptable method performance for quantification at spike level 10, 20 and 50 µg kg⁻¹ were 104, 14 and 4, respectively. The matrix effects for pesticides were also insignificant /acceptable and did not influence the results.

Due to the possibility to extract not only mycotoxins, but also pesticides within the same procedure, the combined method proved to be very efficient in routine survey analysis. Thus, the results obtained were completely satisfactory for the project's purpose.

[1] FAO Corporate Document Repository. *Micotoxinas em grãos*; <http://www.fao.org/wairdocs/X5012O/X5012o00.HTM>. July 10th, 2009.

[2] Pizzutti, I. R., De Kroon, M., Prestes, O. D., Rensen, P., De Kok, A., 7th European Pesticide Residue Workshop, Berlin, Book of Abstracts, 2008, 213.