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MYCOTOXIN ANALYSIS IN 10 SEC? USE OF LASER DIODE THERMAL DESORPTION ION SOURCE COUPLED WITH TANDEM MASS SPECTROMETRY (LDTD-MS/MS) IN DETERMINATION OF DEOXYNIVALENOL AND ZEARELENONE IN ANIMAL FEED

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The mass spectrometry coupled with novel interfaces able to direct analysis of the sample became popular alternative for separation methods. Direct analysis of samples does not include chromatographic separation, and thus reducing the time involved in sample preparation and analysis and reducing costs by eliminating the need for chromatographic columns and mobile phase solvents.

One of the approaches is Laser Diode Thermal Desorption Ion Source (LDTD) introduced by Phytronix. The aim of this study was to evaluate LDTD-MS/MS technique in determination of two important mycotoxins: deoxynivalenol (DON) and zearalenone (ZEN) in animal feed.

The feed samples (solvent standard, blank feed sample, spiked blank samples and certified reference material) were prepared with two sample preparation procedures. First was based on sample extraction with acetonitrile: water: formic acid solution. Second procedure was expanded with clean-up step with immunoaffinity columns.

The 5 µl of extracts were diluted with desorption solution and 5 µl of sample were spotted on LazWell plate and evaporated to dryness. The spots were analyzed on LDTD interface coupled with Sciex Qtrap 5500 mass spectrometer. The laser pattern was 10 s while mass spectrometer worked with MRM mode (two transition per one analyte). Deoxynivalenol (MRMs: 297/247, 297/203) and zearalenone (319/185, 319/187) were used in positive mode.

The results show sufficient sensitivity of the technique for both analytes (LOD for DON=50 µg/kg, ZEN=10 µg/kg) and good repeatability of the signal. The carryover effects were negligible. The linearity of the signal was on the satisfactory level ($r > 0.99$). The repeatability of the results was characterized by relatively low CV (<25%). Based on the data collected during quantitative analysis, the LDTD-MS/MS overall results demonstrated good precision and accuracy.

The results show that LDTD-MS/MS can be interesting alternative in the mycotoxin analysis and potential for further applications in this area.

Keywords: mycotoxin, deoxynivalenol, zearalenone, laser diode thermal desorption, feed

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INCIDENCE OF MYCOTOXINS, PATHOGENIC FUNGI AND INJURED GRAINS IN CORN STORAGE IN FAMILY FARMERS LOCATED IN THE CENTRAL REGION OF MINAS GERAIS

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The corn is a cereal that has significant importance in human and animal feeds. Small farms in the state of Minas Gerais, Brazil, adopt corn storage in spikes, in small cribs, that allow the attack of pests and the spread of toxigenic fungi, which may result in the production of mycotoxins. Mycotoxins are secondary fungal metabolites that can cause serious damage to human and animal health, besides great economic losses, because they make the grains unfit for consumption. This study evaluated the mycotoxins incidence (total fumonisins and zearalenone), injured grains and the fungi prevalence in 44 maize samples collected during two consecutive years in 11 family farmers located in the Central Region of Minas Gerais. The pathology test of grains was carried out employing the method of freezing filter paper and the percentage of injured grains was determined visually. The fumonisins was determined in fluorimeter, after purification in immunoaffinity columns and zearalenone in high efficiency liquid chromatography associated with mass spectrometry. The samples had a low incidence of injured grains with levels between 0,03% and 0,75% and between 0,02% and 0,14% in the first and second year of the study, respectively. There was a prevalence of fungus of the *Fusarium* genus, which presented up to 100% of occurrence, followed by *Penicillium*, *Aspergillus* e *Stenocarpella*. All corn samples analyzed were contaminated with fumonisin, with levels ranging from 31 to 4650 µg kg⁻¹. Results showed that 43,18% of the samples were contaminated with levels above the 1000 µg kg⁻¹, limit established by the European Union for human consumption. However, none of the samples were above the maximum limit established by the Brazilian mycotoxin regulations for unprocessed maize (5000 µg kg⁻¹). Only traces of zearalenone were found in the analyzed samples. The results showed a high incidence of fungi and mycotoxins, although the majority of the samples showed levels of mycotoxins below the maximum limit according to the Brazilian legislation. Therefore, it is suggested the adoption of good agricultural practices and adequate storage, to minimize the contamination of the grains, aiming at good sanitary quality of the corn.

Keywords: fumonisins, zearalenone, fluorescence, fusarium

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BOOK OF ABSTRACTS

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