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Non-destructive detection of mycotoxins in maize kernels using diffuse reflectance spectroscopy

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ABSTRACT

Deoxynivalenol is one of the most abundant contaminants in crops worldwide. Its presence in food and feed products causes health risks and induces economic losses. To date, deoxynivalenol is mostly detected by sample-based, time-consuming chemical analyses. To obtain a non-destructive detection of deoxynivalenol in unprocessed, solid maize kernels, we investigate UV–Vis–NIR diffuse reflection spectroscopy. Specifically, we propose a two-stage spectroscopic measurement procedure, allowing the characterization and identification of deoxynivalenol in ungrounded maize kernels. The first stage focuses on the characterization of the optimum illumination and detection wavelengths, by the measurement of the mean contamination of a collection of maize kernels, using a 250 mm-reflection integrating sphere. The second measurement stage enables an accurate classification of contaminated maize grains, by the measurement of the local contamination on individual kernels, using a 30 mm-reflection integrating sphere. We first characterized and compared the reflection spectra of low (150 ppb) and high (1388 ppb) natural deoxynivalenol-contaminated maize kernels. Moreover, we validated these measurement results by the characterization of a reference deoxynivalenol-contaminated maize powder (1840 ± 30 ppb). A spectral contrast between the low and high contaminated samples could be observed between 700 nm and 1400 nm. Furthermore, a detection criterion could be defined, allowing us to successfully classify a contaminated maize batch into a high (18184 ppb) and low (654 ppb) contaminated subsample. As a result, this verifies the two-stage measurement approach and illustrates the use of diffuse reflection spectroscopy as a valuable tool for the measurement of deoxynivalenol concentrations in maize, paving the way for real-time non-destructive industrial scanning-based detection.

1. Introduction

The presence of mycotoxins, secondary metabolites of toxic fungi, in agricultural commodities is worldwide a major problem. According to the Food and Agricultural Organization (FAO) estimates, 25% of the world's food crops are affected by mycotoxin producing fungi (Rasch, Kumke, & Löhmannsröben, 2010). The most important mycotoxins in food and feed production, that pose a major threat to public health and agro-economy, include aflatoxins, deoxynivalenol (DON), ochratoxin A, fumonisin, zearalenone, patulin and T-2 toxin (Miller, 1995; Traar, 2013).

DON is the most prevalent mycotoxins and is mostly produced by the moulds *Fusarium graminearum* and *Fusarium culmorum*. It frequently occurs on cereal commodities, like wheat, maize, barley, oats and rye, which can be infected before or after the harvest (Sobrova et al., 2010). Moreover, because DON cannot be destroyed during food processing, like cooking, freezing and roasting, it appears both in the raw and processed products. The ingestion of DON-contaminated products can cause acute and chronic health effects such as diarrhea, nausea, immunosuppression and neurotoxicity (Abysique, Tardivel, Troadec, & Félix, 2015; Pestka, 2007). The detection of DON is an important issue in the food industry, because it is present in more than 90% of all mycotoxin-contaminated cereal samples and its occurrence is considered to be an indicator of the presence of other mycotoxins (Ran et al., 2013).

In this paper, we focus on the presence of DON in natural contaminated maize kernels, since DON is an important

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contaminant of maize (Pleadin et al., 2012) and maize is the staple food in many countries. Currently, the presence of DON in food and feed products is strictly regulated in most regions of the world. Regarding raw maize kernels, the European Commission states the maximum allowed DON concentration to be 1750 ppb, while in the USA and China a limit of 1000 ppb of DON is imposed (EFSA., 2013; European Commission, 2007). To fulfill these limits, the presence of DON is nowadays mostly detected by the use of chemical analyses, like liquid chromatography – tandem mass spectrometry (LC-MS/MS) and enzyme-linked immunosorbent assays (ELISA). However, these analytical techniques are time-consuming, expensive and destructive (Ran et al., 2013). Due to the uneven presence of the toxin in both the food products and the crops, these sample-based analyses often give a limited view on the degree of contamination. Therefore, we are interested in the use of non-destructive spectroscopic methods that can be used to screen individual, solid maize kernels.

Spectroscopic detection techniques are already widely used in agriculture and chemical industries for the determination of organic compounds in matter, like proteins, moisture, starch and pigments (Baye, Pearson, & Settles, 2006; Volkers, Wachendorf, Loges, Jovanovic, Taube, 2003; Meulebroeck & Thienpont, 2012). To date, there is a high interest to apply the spectroscopic detection techniques for the identification of DON. The use of Fourier-transform near- and mid-infrared (FT-NIR and FT-MIR) spectroscopy for the detection of DON in wheat and maize is already widely discussed (Abramović, Jajić, Abramović, Ćosić, & Jurić, 2007; De Girolamo, Cervellieri, Visconti, & Pascale, 2014; Kos, Lohninger, & Krska, 2003). However, current published measurements use homogeneously contaminated, grinded samples and require the use of chemometrics to classify the samples into their various contamination levels. Moreover, due to its vibration sensitivity, Fourier-transform spectroscopy can hardly be implemented in an industrial environment. In contrast, we pursue a spectroscopic setup that enables the measurement of the localized contamination in ungrounded, individual maize kernels. Furthermore, we want to develop a technology which can be used for in-line industrial applications.

We investigate ultraviolet–visible–near infrared (UV–Vis–NIR) diffuse reflection spectroscopy for the detection of DON in individual, solid maize kernels. Specifically, we present a two-stage measurement methodology, enabling to efficiently monitor the local DON-contamination on a large amount of maize kernels. The first stage focusses on the characterization of the optimal detection wavelengths, by the measurement of the mean reflection spectrum of a collection of maize kernels with the use of a 250 mm-reflection integrating sphere. The second stage studies the localized contamination on the kernel's surface, by measuring the reflection spectra of individual kernels with a 30 mm-reflection integrating sphere.

Our proposed two-stage approach was first tested on a low and high naturally contaminated maize sample, with an a-priori known DON concentration of 150 ppb and 1388 ppb respectively. Studying the reflectance spectra, an optical contrast between 700 nm and 1400 nm could be observed for both measurement stages. Based on the reflectances at 830 nm, 940 nm and 1220 nm, an optical detection criterion could be defined. This detection criterion was first validated by the measurement of a reference DON-contaminated maize powder. Secondly, it was used to successfully split a contaminated batch, with a-priori unknown DON-concentration, in a low and high contaminated subsample, of which subsequent chemical analyses indicated a DON-concentration of 645 ppb and 18184 ppb respectively.

Generally, this paper presents the use of diffuse reflection spectroscopy as a non-destructive optical detection technique for the identification of DON in solid maize kernels. Section 2 gives an overview of the examined maize samples and discusses the two-stage measurement technique, using the 250 mm- and 30 mm-reflection integrating spheres. In section 3, we investigate the UV–Vis–NIR reflection spectra of low and high naturally contaminated maize kernels. We study the spectral differences and validate our results by the measurement of reference DON-contaminated maize powder. Subsequently, the application of our classification criterion to a contaminated maize batch is presented. Finally, we quantitatively evaluate the optical identification of DON-contaminated maize kernels.

2. Materials and methods

To accurately study the UV–Vis–NIR diffuse reflection spectra of low and high DON-contaminated maize kernels, reliable samples and a repeatable measurement procedure are indispensable. We used natural contaminated samples, taking the typical inhomogeneous DON-contamination into account. Moreover, because maize kernels show a large internal variation in density, texture and substituents concentrations, we investigated different independent samples to minimize the influence of the maize type and the cultivation environment. Considering the spectroscopic measurement configuration, we pursue a measurement methodology which enables a fast, accurate and non-destructive DON-detection that is suited for implementation in industrial in-line, scanning machines. Because of the inhomogeneous DON-contamination, the ability to monitor the contamination of individual maize kernels is indispensable to increase food safety and to reduce economical losses.

In this section, we first give an overview of the investigated maize samples, after which we discuss the diffuse reflection measurement setups.

2.1. Sample preparation

We consider two different natural contaminated maize batches. One batch was obtained from France, while the other one was imported from Poland. The French batch contained a low and high contaminated sample, of each 500 g, that was presorted by the supplier. Before the start of our measurements, 100gr of the low and high contaminated sample was chemically analysed by the use of the LC-MS/MS method, indicating a DON-contamination of 150 ppb and 1388 ppb respectively. No visible differences could be observed between these two French samples. We used the French batch for the optimization of our measurement procedure and to characterize the spectral contrast. The Polish batch contained one DON-contaminated sample with a-priori unknown contamination, which we used to validate our optical classification method. We applied our developed measurement methodology to classify these maize kernels into a low and high contaminated subsample. After classification, the contamination of these subsamples was confirmed by the CODA-CERVA, the Belgian Reference Laboratory for Mycotoxins, who measured a contamination level of 654 ppb and 18184 ppb respectively, by the use of the LC-MS/MS analytical technique.

To approve the reliability of our measurements, we also investigated the optical characteristics of a matrix certified DON-contaminated maize powder. We purchased 55 g reference DON-contaminated maize powder of the Biopure Series from Romer Labs, which contained a contamination level of 1840 ± 30 ppb (Romer Labs, 2011). This contamination level is just above the European limitations, which makes it suited to evaluate our detection performance.

All samples, both the reference maize powder as the French and