Detection of datura seeds containing alkaloids in buckwheat production by NIRS and NIR hyperspectral imaging.

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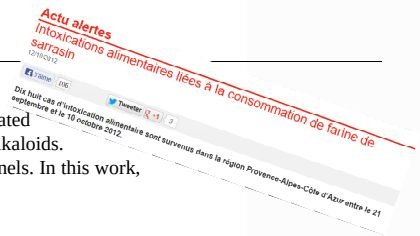
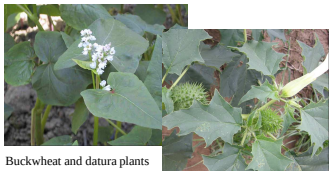
Introduction

The development of organic food leads to a reemergence of the presence of poisonous plants. Recent intoxications in France were linked to the consumption of bakery products made using organic buckwheat flour that was contaminated with *Datura stramonium*, a wild-growing plant found in several crops and well known for the high content in toxic alkaloids. Rapid and non-invasive methods are needed to manage risk and detect the presence of contaminants in seeds and kernels. In this work, NIR and NIR hyperspectral imaging spectroscopy have been assessed for this purpose.



Material and methods


Buckwheat and datura plants

For this work, datura and buckwheat seeds issued from different sources have been collected. Then, seven mixtures of buckwheat grains contaminated with 0.01% to 1% (i.e. 100 to 10 000 ppm) of *Datura stramonium seeds* were prepared. The samples (pure kernels and mixtures) were measured using two NIR instruments: a classical NIR spectrometer and a NIR hyperspectral line scan imaging system combined with a conveyor belt, both active in the 1100-2400 nm range.


Datura seeds in buckwheat grains

Classical NIR spectrometer using a small spot setup


Classical NIR instrument

For this study, a calibration set (30 datura seeds spectra and 30 buckwheat kernels spectra) and a validation set (20 datura seeds spectra and 30 buckwheat kernels spectra) have been used from the database by selecting, for the validation set, samples from different sources than the calibration set. Figure 1 shows the mean raw spectra for datura and buckwheat respectively. The detection of datura seeds in a lot of buckwheat was investigated. It was shown that by using Composite NIR analysis, it was possible to obtain different spectral profile in relation to the presence of datura seed in the buckwheat. The data were preprocessed by the Standard Normal Variate transform followed by 1st derivative Savitzky-Golay treatment (5,2,1). PCA allowed discriminating between pure datura, buckwheat kernels and mixture of both specimens. (Figure2). Wavelength selection was performed to better chemically understand the differences between datura and buckwheat kernels. For this, the Fisher criterion was applied, among others, to select the most important wavelengths. Figure 3 shows the Fisher coefficient calculated on preprocessed data for the wavelength range of the classical NIR instrument. Two wavelengths, 1726 nm and 2282 nm were selected, based on the specific spectral region of the datura seeds (lipid content) and on the Fisher coefficient value. It was shown that based on these 2 wavelengths, pure datura, pure buckwheat kernels and mixture of both specimens can be discriminated (Figure 4).

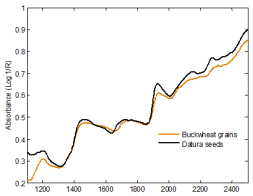


Figure 1: Datura (black line) and buckwheat (brown line) mean NIR spectra.

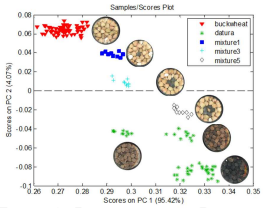


Figure 2: Discrimination Datura vs. Buckwheat based on full spectral range.

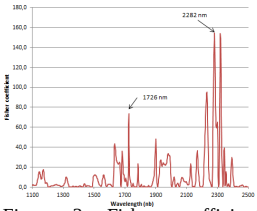


Figure 3: Fisher coefficient calculated from preprocessed spectra.

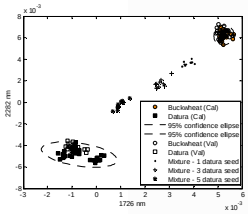
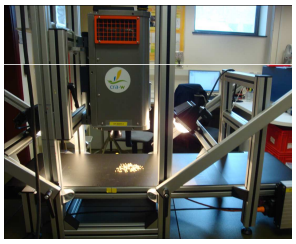


Figure 4: Discrimination Datura vs Buckwheat based on 2 wavelengths (1726 nm/ 2282 nm).

NIR hyperspectral line scan imaging system using a conveyor belt


NIR hyperspectral line scan camera

NIR hyperspectral line scan imaging system was also assessed for the identification and quantification of datura in buckwheat. The figure 5 shows the typical spectra for buckwheat kernels and datura seeds. Partial Least Squares Discriminant Analysis (PLSDA) was used as classification method for the construction of the discrimination model. It was applied to all the individual pixels in the images of the adulterated buckwheat samples in order to isolate and quantify the number of pixels detected as datura (Figure 6).
The results showed a correlation higher than 0.99 between the reference and the predicted values using PLSDA model applied on samples contaminated with 0.1 g/kg to 10 g/kg (Figure 7) . The LOD and LOQ were estimated to be 146 mg/kg and 392 mg/kg respectively. These results indicated that the LOD and LOQ of the methodology were easily capable of meeting the current EU regulatory limits for cereals (500 and 1000 mg/kg for food and feed respectively).
It was shown also that datura flour can be identified in buckwheat sample using NIR hyperspectral imaging (Figure 8).

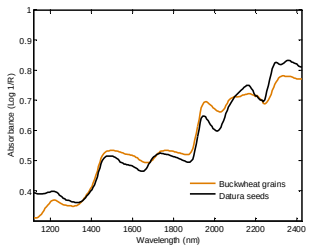


Figure 5: Datura (black line) and buckwheat (brown line) mean NIR hyperspectral spectra.

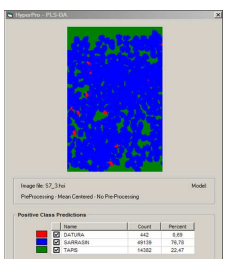


Figure 6: Image showing the results of the PLSDA model for an adulteraded buckwheat grains sample on the conveyor belt.

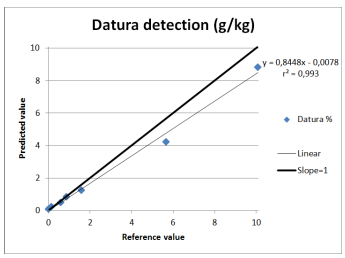


Figure 7: Results of datura quantification in buckwheat using PLSDA model.

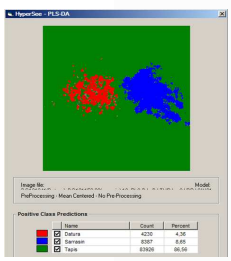


Figure 8: Image showing the results of the PLSDA model for an adulteraded buckwheat flour sample on the conveyor belt.

Conclusions

Until now, all the published studies dealing with the detection of datura during harvest use macroscopic, optical microscopic or HPLC-MS methods. In this study it is demonstrated that non-destructive techniques as NIR and NIR hyperspectral imaging spectroscopy can be used as alternative methods for detection and quantification of undesirable substances in crop kernels.

Reference

Vermeulen P., Fernández Pierna J.A., Van Egmond H., Dardenne P. & Baeten V. (2012). *On-line detection and quantification of ergot bodies in cereals using near infrared hyperspectral imaging*. Food Additives & Contaminants, 29 (2), 232-240.
Vermeulen P., Fernández Pierna J.A., Van Egmond H., Zegers J., Dardenne P. & Baeten V. (2013). *Validation and transferability study of a method based on near infrared hyperspectral imaging method for the detection and quantification of egot bodies in cereals*. Analytical and Bioanalytical Chemistry, CONffIDENCE Special issue, in press.

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