

Inline HSI food inspection and concentration measurements of pharmaceuticals – a report from an industrial environment

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Abstract Current food diagnostic measurement techniques tend to suffer from unreliable chemometric quantification and poor spatial resolution. Chemical components in food such as sugar, fat, water and dry substance are today inferred from destructive, lab based, techniques that measure chemical composition albeit with limited statistical accuracy and insufficient precision for food quality monitoring. Technologies such as NIR hyperspectral imaging (HSI) and FTNIR offer the potential for accurate, fast and efficient evaluation of chemical components accurately over the whole food production cycle. In this regard, the disentanglement of spectral endmembers from a mixed detector response due to background and multiple chemical constituents is both crucial and scientifically challenging for a reliable signal detection in terms of illumination technique, data pre-processing and multivariate analysis. In this talk various inline applications employing NIR hyperspectral imaging using the HELIOS camera system will be presented, including examples and results from a prototype project in pharmaceuticals and a joint study in which HSI quantitative analysis of chemical concentration gradients found in potatoes and wheat grains are discussed.

1 Introduction

Near infrared spectroscopy (NIRS) has proven to be one of the most powerful techniques for (quantitative) analysis and classification of materials as well as continuous controlling the quality of e.g. food and feed. NIRS uses normally a spectral range from 780-2500 nm, where mainly overtones and combinations of vibrational modes from C-H, O-H and N-H chemical bonds are located, carrying most of the information of the chemical composition of organic materials, food, feed and beverages [1]. The advantage of NIRS is the fact that no sample preparation is necessary and there are several ways to collect spectra in a non-destructive way like transmittance, interactance, transreflectance, diffuse transmittance, and diffuse reflectance. In addition the advantage of NIRS in comparison to mid infrared spectroscopy is the higher energy due to shorter wavelengths penetrating the products to get information from under the surface like the ripeness of fruits [2, 3]. Attention must be paid for the calibration models which can be a very time consuming and costly procedure. Changes of the composition of the target product like new harvest or new varieties must be taken into account and the training set should be updated and checked on a regular base [4]. Beside water several other components like fat, carbohydrates and proteins can be quantified using NIRS to optimize quality parameters from raw materials to the finished product. Moreover, beside the determination of the chemical composition of the (food) material even other parameters like authenticity [5] or sensory properties [6] are inferable. Problems can arise from interferences from uneven surfaces during online monitoring or when the chemical information is not evenly distributed in the sample.

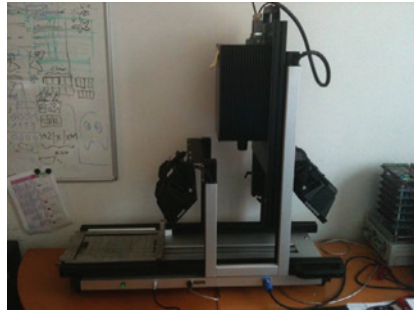
In the following we describe two different applications of NIRS from the pharmaceutical as well as the food industry employing near infrared hyperspectral imaging (NIR HSI) and/or Fourier transform near infrared (FTNIR) spectroscopy cameras.

1.1 The camera systems

The EVK HELIOS camera is a hyperspectral imaging system operating in push broom mode yielding spatially resolved NIR spectra from a moving sample [7]. It features an $320(\text{spectral}) \times 256(\text{spatial})$ InGaAs



(a) HELIOS CORE camera.



(b) Setup including HELIOS hyperspectral camera, halogen light source and driving unit.

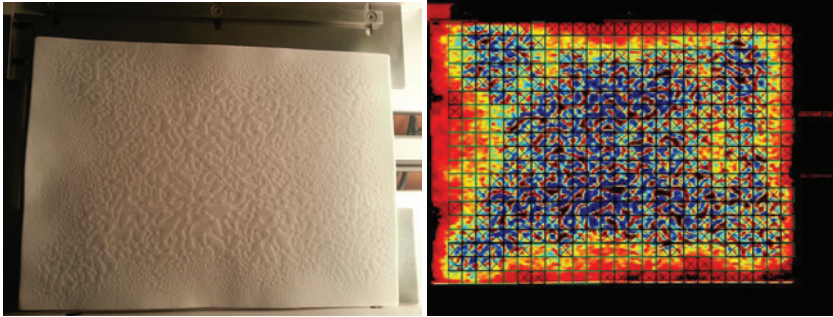
Figure 13.1:

sensor array and covers a spectral range of $0.9\text{--}1.7\ \mu\text{m}$. A powerful FPGA unit is part of the camera and allows for integrated real time data processing and analysis (Fig. 13.1 (a)). With a sampling rate of up to 330 Hz (full frame) the camera is suitable for fast inline applications with bulk flows of several m/s of input material.

The i-RED FTNIR spectrometer operates from 900–2600 nm and acquires up to 100 spectra per second with DSP based high speed data processing [8]. Light reflected from the sample surface is directed to a spectrograph with 0.15 nm bandpass at 2600 nm via one channel fibre optics. The data are streamed to a PC where further (real time) analysis of the spectra is possible.

2 HSI concentration monitoring

In the following we describe the inference of concentration gradients of thin layers of dry chemicals from HSI data. Figure 13.2 (a) shows a patch covered with a thin layer made from baking a white pharmaceutical powder. The problem in related pharmaceutical quality moni-



(a) Patch covered with baked white powder (b) Hyperspectral concentration gradient showing concentration gradients. (colour coded) of dry chemicals.

Figure 13.2:

toring is the reliable measurement of concentration gradients due to ill deposition of the dry chemical prior to the baking process. The data analysis discussed here is based on measurements using the $1.7\ \mu\text{m}$ HE-LIOS CORE camera as described in sec.1.1 and multivariate analysis techniques as e.g. described in [9].

2.1 The model

The underlying quantitative model is the *partial least squares regression* (PLS) a standard technique where the spectral data is optimized with respect to the target data to be inferred (see e.g. [9] and [10]). The set of equations

$$X = tp^T + E \quad (13.1)$$

$$Y = uq^T + F \quad (13.2)$$

represents the decomposition of the spectral and chemical concentration data, X and Y , respectively, into loadings and scores according to standard *principal component analysis* (PCA). The t , u and p , q denote the *scores* and *loadings* vectors of X and Y , respectively, and E and F are the residual variance matrices. The PLS regression adapts the chemical concentrations with respect to the spectral data during the optimization

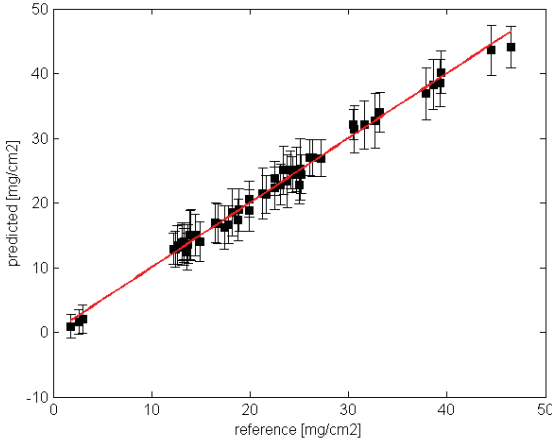


Figure 13.3: PLS regression of 53 NIR spectra of dry chemicals using two PLS components. Shown are nominal vs. measured concentrations in units of mg/cm^2 .

process via the relations [9]

$$Y = X^T B + B_0 \quad (13.3)$$

$$B = W(p^T W)^{-1} q^T \quad (13.4)$$

$$B_0 = Y^T - X^T B \quad (13.5)$$

$$W = X^T u / \sqrt{(X^T u)(X^T u)^T} \quad (13.6)$$

Such, information from the target data influences the PCA of the spectral data and vice versa. Figure 13.3 shows the outcome of a two component PLS regression of 53 NIR spectra of dry chemicals recorded with the HELIOS camera. The fit shows the measured against the nominal concentration values in units of mg/cm^2 . The correlation between predicted and nominal concentrations is $R^2 = 0.99$. The inferred (normalized) PLS coefficients B along with the teach spectra are shown in Fig. 13.4.

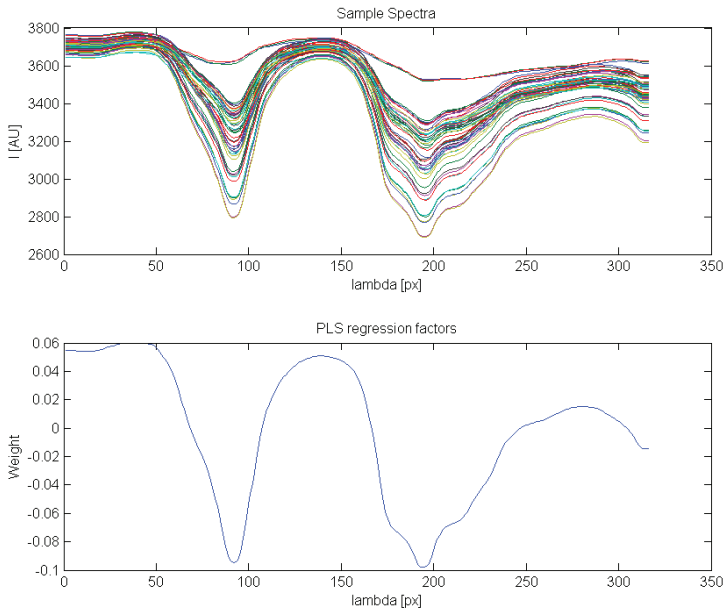
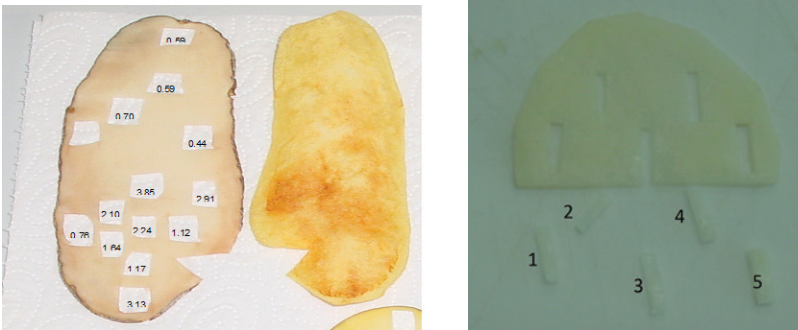


Figure 13.4: NIR teach spectra for the PLS regression model (upper panel). PLS coefficients B for a two component regression (lower panel).

2.2 Measurement

The sample sheets coated with different concentrations of a dry chemical (Fig. 13.2) were scanned with the HELIOS camera in *push broom* mode using a driving unit (see Fig. 13.1). Using the model described in sec. 2.1 it is thus possible to assign a concentration value to every scanned sample pixel containing a full spectrum. An instructive example of the spatially resolved colour coded concentration gradient is shown in Fig. 13.2. The already to the naked eye visible inhomogeneous deposition of the investigated chemical substance can be reproduced with high accuracy quantitatively in the NIR regime (see Fig. 13.2) and help identifying quality issues in the production process following statistical bench marks.



(a) Chemical reference analysis of dry substance combined with brown reactions. (b) Chemical reference analysis of sugar content.

Figure 13.5:

3 Quantitative analysis of potatoes and wheat grains

Objective of this study was the feasibility of measuring chemical constituents concentrations in foods using NIR technology with the ultimate goal of constructing an inline food analyser in the near future. In particular, we focussed on the investigation of concentration gradients found in potatoes and wheat grains. For potatoes a crucial parameter for subsequent food processing is the distribution of water vs. dry substance and sugar, since gradients of e.g. sugar can have unwanted effects during the frying stage present e.g. in the French fries production. Concerning wheat grains, the parameters of interest are the baking properties of flour usually inferred from an amylograph which displays the viscosity of dough as a function of (linearly increased) temperature. In order to be able to correlate hyperspectral information with the wanted target parameters, such as sugar content in potatoes and gluten found in flour, chemical reference analysis is needed.

3.1 Potatoes

Slices of different potato varieties were investigated using FTNIR, HSI and chemical reference analysis. The latter provided both the spatial information of sugar content as well as unwanted correlated browning

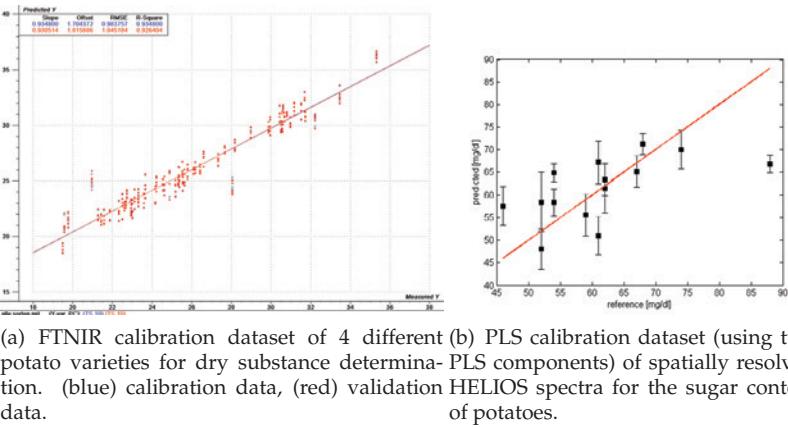


Figure 13.6:

due to Maillard’s reaction [11] during frying as shown in Fig. 13.5. The amount of glucose at different locations across the potato slide was extracted using a highly sensitive blood sugar test (tab.13.1). Such the reference analysis yields glucose concentrations as target data Y being input for a PLS regression of NIR spectra X along the lines of sec. 2.1.

Table 13.1: Chemical reference analysis of glucose content in mg/dl across potato slices. Sample locations are depicted in Fig. 13.5 (b).

Slice #	1	2	3
Sample #	[mg/dl]		
1	61	62	-
2	62	67	54
3	52	61	59
4	54	52	46
5	74	88	68

The regression of a calibration data set for dry substance inferred from 4 different potato varieties is depicted in Fig. 13.6 (a) for FTNIR spectral data measured with a *Thermo Nicolet Antaris* analyzer. The spectral data show a very high correlation with the inferred reference analysis independent of the potato variety. Since an inline food analyser should

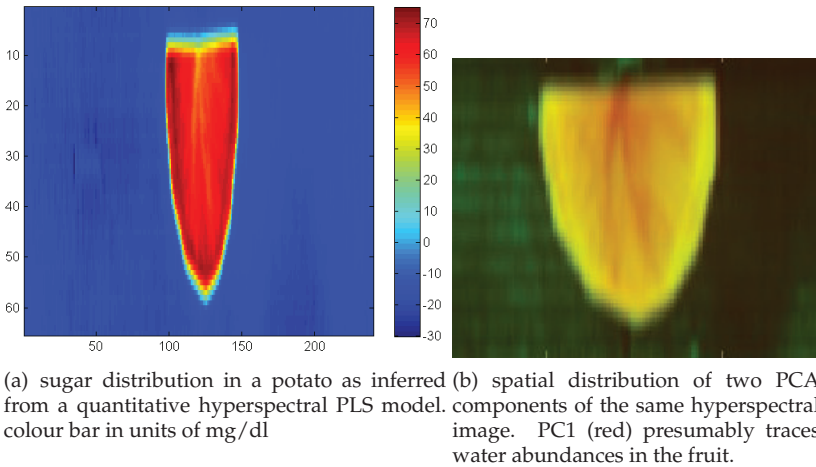


Figure 13.7:

ultimately deliver spatially resolved dry substance and sugar gradients the analysis was also performed using the HELIOS hyperspectral imager.

Figure 13.6 (b) shows the calibration curve inferred from PLS regression using spatially resolved NIR spectra for the sugar content. There is only a moderate correlation of $R^2 = 0.42$ between the reference analysis and the spectral prediction. While the FTNIR usually features better spectral statistics based on averaging over the whole spatial sample domain, the hyperspectral approach suffers from reduced spectral statistics but gains spatial information of sugar gradients in the potato. Such it was already possible to detect so called *sugar ends* [12] in whole potatoes as shown in [13]. But rather than merely classifying different constituents such as sugar, peel or dry substance it is interesting to get more quantitative information as well in order to gain quality and process analysis data for the production. Figure 13.7 (a) shows the quantified sugar gradient for one of the analysed potato slices. It appears that glucose increases towards the rim of the potato while the inner parts are dominated by fine channel like structures presumably tracing water (Fig. 13.7 (b)).

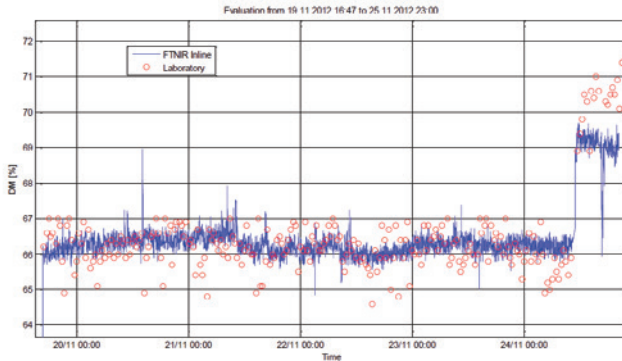
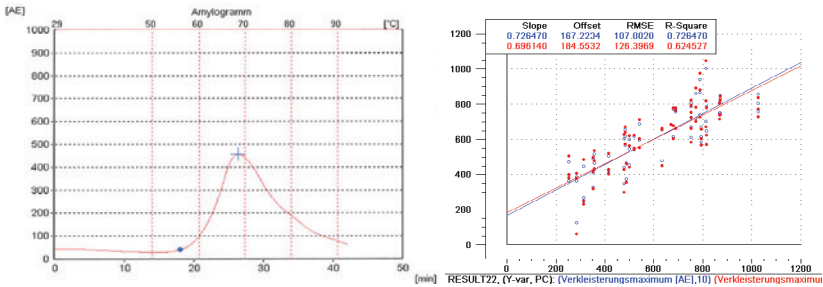


Figure 13.8: Dry substance content of French fries vs. time in a production process. (red) lab references (blue) inline FTNIR measurements.

An inline measurement of the dry substance content of French fries integrated in the production process was performed employing the i-RED FTNIR process spectrometer system and compared with lab references inferred from the material stream at the same time. The measured values from the FTNIR data based on PLS regression show an overall good agreement with the lab references over time following the same global trend while featuring reduced scatter (Fig. 13.8).

3.2 Wheat grains

Along the same lines, i.e. correlating chemical reference data with FT-NIR spectral data, the baking properties of whole wheat grains as well as flour was modelled. The relevant baking parameters are the start/end of agglutination and its maximum in terms of dough viscosity as shown in the amylogram Fig. 13.9 (a). The according data was correlated with the agglutination maximum similarly as for the potato dry substance content (Fig. 13.9 (b)). A correlation coefficient of roughly $R^2 \sim 0.7$ for predictions inferred from whole wheat grain spectra vs. viscosity maxima as measured with the amylograph represents again a promising result with respect to an inline food analysing application.



(a) Amylogram of wheat showing dough viscosity vs. temperature and time, respectively. The blue spot denotes the beginning of agglutination and the cross its maximum. Taken from [14].

(b) Correlation of whole wheat grain color vs. agglutination maxima taken from amylograph data.

Figure 13.9:

4 Conclusion

Quantitative analysis of material parameters such as chemical concentrations, constituent gradients and food components using inline NIR technologies is possible and ready for industrial application. Hyperspectral imaging and FTNIR cameras provide sufficient spectral (and in case of HSI spatial) resolutions at fair data processing speeds to tackle industry relevant process analysis problems at typical bulk flow velocities of several m/s. Albeit comprising limited statistical accuracy compared to chemical lab methods with respect to the individual sample test the great benefit of quantitative inline analysis comes with the enormous increase of the sample size, ultimately monitoring the entire input stream on-line. The prototype studies for a dry chemical concentration as well as inline food analyser presented in this article show that quantitative results inferred from NIR spectra are compatible with lab references within their statistical accuracy. Further work has to be done to increase the methods accuracies mainly with respect to quality (S/N) and selection criteria of relevant NIR spectra for the model building involved.

Acknowledgments

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