Portable Near-Infrared Spectroscopy for Analysis of Crops

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ABSTRACT

In this article, the tools needed for applied near-infrared (NIR) spectroscopy in field analysis of crops are discussed. An overview of the hardware and mathematical modeling needed for the realization of such instruments is provided, and, as an example, the performance of a hand-held analyzer (GrainSense) with common wheat is described.

Protein content is a key quality factor for many crops worldwide, but it is known to vary considerably even within an individual field. Initially, analytical chemical analysis methods for analyzing protein content were used, but in general, they are impractical and too time-consuming for large-scale analysis. Therefore, near-infrared (NIR) spectroscopic methods were introduced and commercialized in the 1980s. At first, NIR spectroscopy was primarily used to analyze silage for the livestock sector, but it use was soon expanded worldwide for a variety of crops (4). Currently, NIR spectroscopy is the most widely used method by the global cereals industry for the assessment of compositional characteristics such as protein, moisture, and lipid contents, which are all essential parameters in the food industry (1).

To analyze grain quality in the field, GrainSense has developed portable NIR spectroscopy-based technology. In addition to developing a hand-held NIR solution, the collected data are immediately analyzed and presented to users in a user-friendly format. The device can be used by anyone, anywhere, and at any time once kernels are ripe. The data is sent from the device to the GrainSense app with GPS coordinates via a Bluetooth connection. This enables subsequent data analysis through GrainSense cloud services, either indirectly in the app or using the GrainSense dashboard. GrainSense allows growers, breeders, collectors, and other members of the food value chain to collect data and turn it into immediately available information to improve their business operations based on actual data.

Applied NIR Spectroscopy

Energy carried by photons can be transferred to materials via photon–matter interactions, in which the photon disappears and the energy is absorbed by the material. In general, this can happen only in material-specific resonant frequencies. Depending on the photon energy range (and material), absorption can take place between nuclear, electronic, vibronic, or rotational resonances. In the case of NIR spectroscopy, at wavelengths of about 800 to 2,500 nm, the resonances are in the (ro-)vibronic range, where it is possible to further focus on the fundamental frequencies, overtones, or so-called combination bands. Since photon absorption probability for the first absorption is dramatically higher than for the overtones, different energy ranges penetrate matter to different depths and, thus, provide a way to make NIR instruments sensitive to surface or bulk properties (described in more detail in Pasquini [2] and the references therein). In organic material the most important bonds that absorb energy in the NIR energy region are O-H (water), C-H (fats), and N-H (protein), which provide the means for determining absolute or relative moisture, protein, and lipid contents in the material.

In industrial applications, NIR spectroscopy is usually applied as an indirect correlative technique. In practice, it means that the measured spectra alone are not sufficient to provide direct information about the studied properties. Instead, the spectra are analyzed using multivariate (chemometric) means (5). In this process, the spectral data are correlated against reference measurements obtained using chemical analytical methods. With sufficient amounts of data, a spectroscopist is able to build a calibration model that can predict properties of unknown samples. Several types of methods can be used for this task. Traditional approaches include, for example, different linear regression models, such as principal components regression and partial least squares regression. More recently, neural networks and machine learning have also been successfully applied for the task (6).

The general process for obtaining and using a multivariate calibration is illustrated in Figure 1. The model is initially fed by calibration spectra with external references that are expected to be correct. A model using a selected mathematical framework is calculated from a set of calibration measurements and tested against independent validation data. If the model has sufficient predictive power for the validation data, it can then be applied to spectra from samples that do not have external references.

Portable NIR Spectrometer

The project that was developed and eventually became GrainSense Ltd. began in the VTT Technical Research Centre of Finland in 2015. The aim of the project was to bring an application of traditional, bulky NIR lab equipment to an economical hand-held format. The project was realized through the key innova-

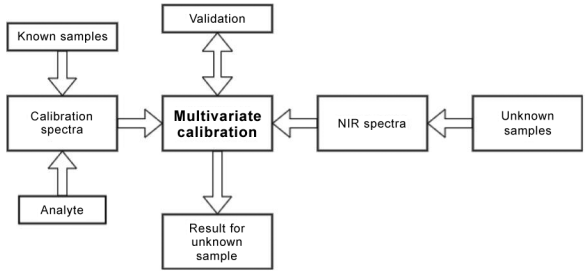

Fig. 1. Flowchart of multivariate calibration process.

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tion of using an integrating sphere in combination with absorbance spectroscopy, as well as the development of a low-noise and low-power consumption NIR photodetector for ambient temperatures.

An integrating sphere is a construct that contains a reflecting sphere in the desired photon energy range, a light source, and a small hole for the light to exit the sphere for detection. The main benefit of this combination is that when the sphere is illuminated, a single photon can bounce around hundreds of times before finding its way to the hole and, eventually, to the detector. Therefore, if a single layer of matter is placed inside the sphere, it is penetrated by light hundreds of times and from all angles. consequently, the layer is effectively seen as hundreds of layers, which increases the sensitivity of the device by orders of magnitude, which in turn makes a handheld NIR device possible in practice. The increase in sensitivity is so large, in fact, that these devices could be used to predict the properties of crops down to the single seed level. A comparison of the integrating sphere method and common NIR bench-top lab devices is provided in Figure 2.

The GrainSense hand-held analyzer is capable of rapidly measuring key parameters of crops: protein, moisture, oil, and carbohydrate contents. Protein and moisture contents are essential parameters for cereal crops that need to be measured in order to improve daily operations from harvest to storage. For oilseeds, oil content is often the main parameter for commercial trade (3). Carbohydrate content is often less important, but it is nevertheless provided for the sake of completeness. It should be noted that the term carbohydrate includes minerals and other minor components. Carbohydrates are calculated using proximate analysis, subtracting the other measured components, resulting in a carbohydrate measurement. Since the amount of gluten in wheat is known to correlate with total protein content, the device also measures gluten as an additional derived parameter.

The GrainSense calibration models are constructed on an absolute scale, which means that the values obtained from the spectra are not percentages, but real masses of different components (in milligrams). It is also worth noting that since it is the component masses that are defined, the device is actually a stand-alone NIR spectroscopy-based scale that is sensitive and selective for protein, moisture, oil, and carbohydrates. When the masses are known, it is a straightforward process to provide the percentages for any desired moisture basis, adjusted to local reporting standards. For example, protein is reported on a dry matter basis (i.e., 0% fixed moisture) for wheat in Germany, whereas protein values in the United States are reported on a fixed 12% moisture basis. Currently, grain data can be obtained using the GrainSense analyzer through seven base calibrations: wheat (whole kernels), barley (whole kernels), oats (whole kernels), rye (whole kernels), rapeseed (whole seeds), maize (ground kernels), and soybean (ground seeds).

GrainSense Calibration Performance for Common Wheat

For a better understanding of the GrainSense calibrations, the common wheat calibration is discussed in more detail. In practice, a multivariate calibration for complex systems such as grains and seeds is never complete, but the more data the calibration has, the more reliable it becomes. Ideally, the model should automatically "adapt" to differences resulting from varying climate conditions, color and size variations from different subspecies, year-to-year changes in temperature and rainfall, and so on. Therefore, companies working in this field fine-tune their calibrations on a yearly basis, and GrainSense is no exception. Currently, the external laboratory references for protein are obtained using the Dumas method (Gerhardt Dumatherm, C. Gerhardt GmbH & Co. KG, Königswinter, Germany) and for moisture and oil with NMR spectroscopy (Bruker mq-one, Bruker Optik GmbH, Ettlingen, Germany). These reference methods have the benefit that the values are not affected by the aforementioned variations, which allows immediate analysis of any seeds from anywhere in the world. In the current wheat calibration, 807 wheat samples from different harvest years and more than 10 countries were used. The analytical performance is presented in Table I. In addition, scatter plots of reference versus predicted results from measurements for protein, moisture, and carbohydrates are depicted in Figure 3.

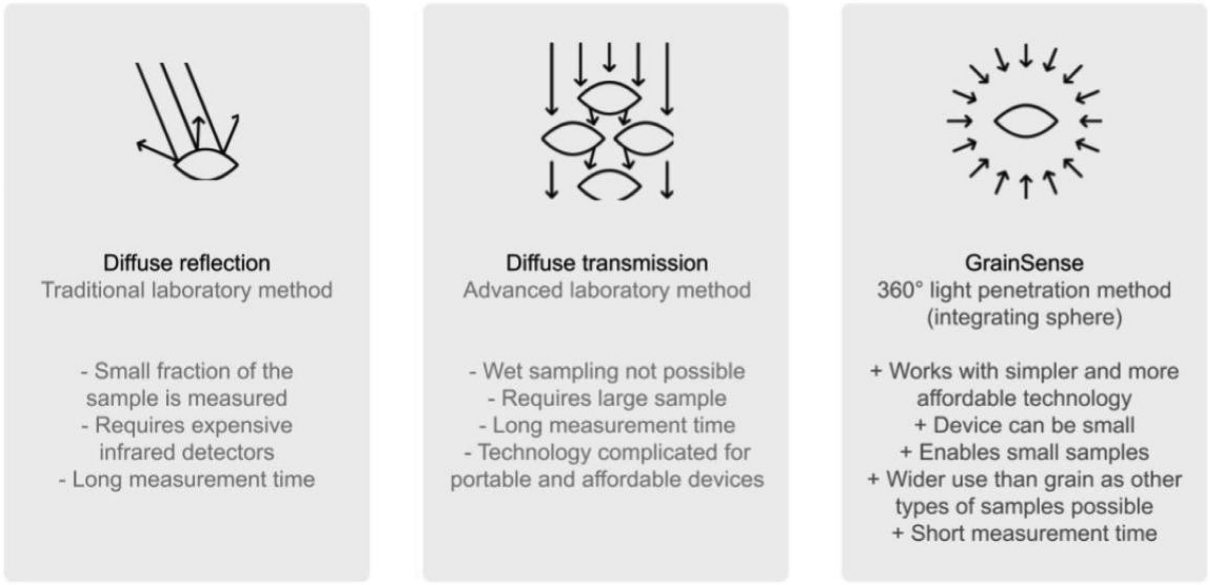

Fig. 2. Comparison of optical methods showing advantages of an integrated sphere.

Table I. Performance evaluation based on 807 independent measurements[a]

Component	SEP (%)	R^2	Min Ref (%)	Max Ref (%)
Protein	0.52	0.93	8.4	19.5
Moisture	0.24	0.98	8.4	19.2
Carbohydrate	0.64	0.90	78.5	89.6

[a] The percentages for protein and carbohydrate are calculated on a dry basis. Minimum (Min) and maximum (Max) reference (Ref) moisture contents are for the samples on a wet basis. SEP: standard error of predictions; R^2: coefficient of determination.

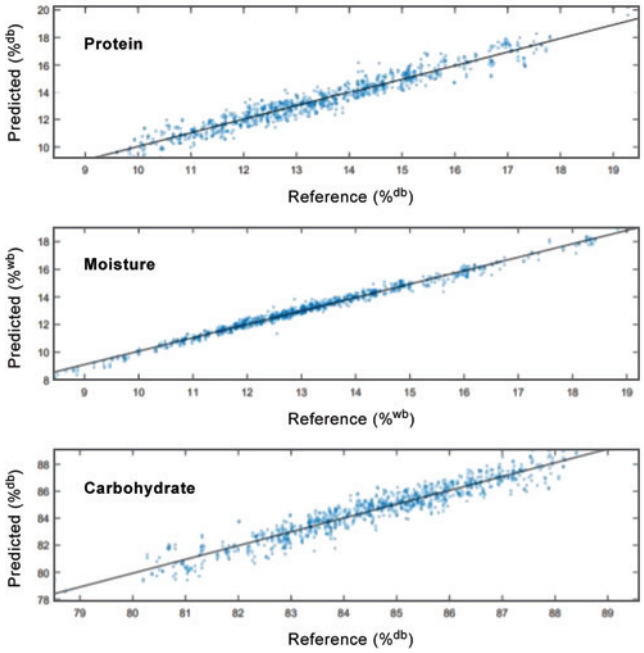

Fig. 3. Reference versus predicted results for common wheat. Protein and carbohydrate are measured on a dry basis, and moisture is measured on a wet basis. The data consist of samples from 13 countries; the black lines show least-square fits to the data.

The Future of Portable NIR

Technical innovation and practical applications of collected data resulted in GrainSense creating an in-field portable solution for collecting key quality parameters of grains and oilseeds. This technology can be applied to a variety of organic substances with the appropriate calibration work. Continued innovations from companies focused on economical NIR solutions will allow more and quicker access to quality data in the field, thereby giving crop producers the ability to analyze their ion more quickly using more precise data.

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Melvin Robert Huber III, M.A., works in the GrainSense Commercial Team based in Berlin, Germany. This team focuses on bringing GrainSense technology to end-users, as well as supporting improvements in the application of GrainSense's portable NIR solution to the agricultural sector. He is also a current Ph.D. student, focusing his research on agricultural ethics.

Kari Jänkälä, Ph.D. (physics), is currently the chief technical officer of GrainSense. His role in the company is to lead the R&D actions, and he is responsible for the development of spectral analysis and mathematical frameworks applied in GrainSense devices. Before joining the GrainSense team in 2018, he had worked for 13 years in the university sector. He specializes in computational modeling of nanoscale materials and data analysis and has done scientific research applying various photon-induced spectroscopic techniques from the NIR region up to hard X-rays. He has authored and co-authored about 60 peer-reviewed scientific articles in the fields of atomic and chemical physics.