Application Note

Grain Study Demonstrates MicroNIR Applicability for Quality Analysis

Food safety has been a global topic in recent years stemming from a wide range of incidents related to foodborne illnesses and food adulteration. According the U.S. Food and Drug Administration (FDA), 48 million people fall ill every year in the United States from foodborne illnesses; this is 1 in 6 people[1]. Economically motivated adulteration has cost many lives and is estimated to cost the industry $10 to $15 billion per year, affecting approximately 10% of all commercially sold food products, according to the Grocery Manufacturers Association (GMA)[2].

In an effort to take proactive and preventive steps rather than reactive responses to these problems, the United States passed into law in early 2011 the Food Safety and Modernization Act (FSMA). The FDA is tasked with creating the rules and regulations for the industry to follow, and has proposed five standards that establish the food safety framework. These are:

- Produce Safety Standards

- Preventive Controls for Human Food

- Foreign Supplier Verification Program

- Preventive Controls for Animal Food

- Accredited Third Party Certification

Even though the complete rules and standards remain under construction and new deadlines are being established for the FDA to complete the safety standards, there has been quite a bit of information shared by the FDA on the FSMA scope[3].

At the heart of FSMA is 'prevention' that is supported by inspection, enhancing product traceability, preventing adulteration or contamination, and ensuring import safety, to list just a few. Of note, imports of food products in the US are significant with 75% of seafood and 50% of fruits being imported, for example[3].

Analytical methods for testing and inspecting against adulteration or contamination will play a key role in helping the industry comply with the expected FSMA regulations. Near-infrared (NIR) spectroscopy is a well-known technique that has been used in the food, feed, and agriculture industries for decades for the compositional analysis of grains, forage, and cereals, and more recently, for fruit ripening and the authentication of food products. The FSMA's rules on preventive control of animal food include a requirement for nutritional balance of animal feed, whereby NIR spectroscopy is an established and effective tool in providing the nutritional content of feed and feed ingredients. With the increasing miniaturization and affordability of NIR instruments, in the manner that been accomplished with the VIAVI Solutions MicroNIR™ spectrometer, taking measurements in the field, at the farm, or anywhere throughout the supply chain will be crucial in democratizing the technology and enabling a safer food supply.

Objective

In order to assess the capability of the MicroNIR spectrometer for the fast and non-destructive chemical analysis of grains, a study was done by Prof. Charles R. Hurburgh's group at Iowa State University (ISU) at the Grain Quality Laboratory (GQL).

Introduction

The ISU GQL provides grain quality analysis services for genetic trials and commercial uses. GQL analyzes grain samples for a variety of grain quality factors, including composition and physical traits. The GQL near-infrared moisture calibrations are approved for trade by the National Institute of Standards and Technology, and the corn constituent calibrations are being used by corn traders. Dr. Hurburgh is the professor-in-charge of the Grain Quality Laboratory. Dr. Hurburgh's research interests include the physical and chemical properties of biological materials, chemical and electronic instrumentation, near-infrared reflectance analysis and sensors, chemometrics, metrology, and statistics of very large databases.

The MicroNIR Spectrometer

The MicroNIR is an ultra-compact spectrometer designed to be used in diffuse reflection, transflection, or transmission. The MicroNIR uses a linear variable filter (LVF) component mounted over a diode array detector that separates incoming light into individual wavelengths. The spectrometer integrates the light source and readout electronics in a small construction. Key attributes of the MicroNIR 1700 spectrometer are summarized in Table 1.

Figure 1. The MicroNIR spectrometer

Table 1. Key performance attributes of the MicroNIR 1700 spectrometer

Parameter	Specification
Weight	60 g
Dimensions	45 mm diameter x 42 mm height
Spectral range	950 – 1650 nm
Number of pixels	128 pixels, 125 point standardized grid
Optical resolution	<1.25% of center wavelength (for example, at 1000 nm wavelength, resolution is <12.5 nm)
Geometric resolution	6.25 nm per pixel
Power requirement	USB powered, <500 mA at 5 V
Operating temperature	−20°C to 40°C

Experimental Procedure and Model Development

In the first part of a two-part study, calibration and cross-validation models were created from 278, 203, 212, 120, and 171 samples of soybean, soybean meal, corn, bakery meal, and dry distilled grains (DDG) respectively, representing multi-year crops, some dating back to year 1997 until 2012. The models were created to determine the concentration of moisture, protein, oil, fiber, and starch. The reference values for each constituent were obtained from laboratory measurements using either a benchtop NIR instrument or wet chemistry analytical techniques. In the second part of the study, an independent validation of the model with new samples is being conducted. In this application note, we will provide a summary of the first part of the study using the soybean results as a representative example to demonstrate the capability of the MicroNIR spectrometer.

The MicroNIR 1700 spectrometer was placed under a 4" (100 mm) diameter Plexiglas® cup that contained the material to be measured. The cup was not rotating in this study. The spectrometer was used in diffuse reflection mode. The integration time of the MicroNIR 1700 was set to 11,500 microseconds. The total number of scans taken per measurement was set at 50. Three measurements per sample were taken.

Plexiglas cup

Figure 2. An empty Plexiglas cup with a MicroNIR spectrometer positioned below the plastic surface of the cup

Figure 3. Image of a calibration sample set for soybeans from year 2012 crop

The range of moisture, protein, oil, and fiber present in the soybean calibration sets is summarized in Table 2 below.

Table 2. Moisture (M), protein (P), oil (O), fiber (F); P, O, F basis 13% moisture

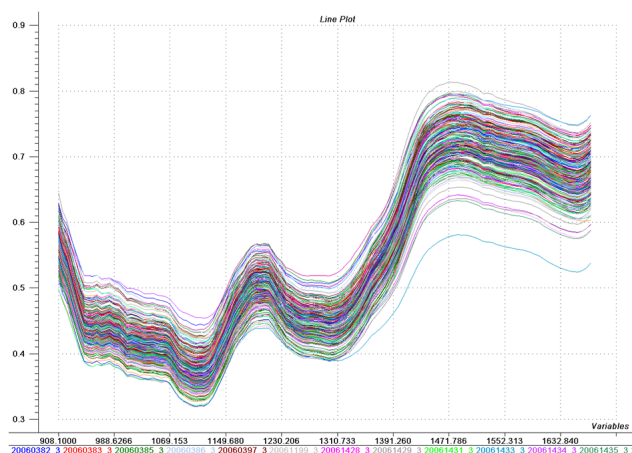| Product	Soybeans			
Const.	M	P	O	F
Range (%)	[6.2; 15.30]	[24.72; 47.17]	[11.85; 26.44]	[3.68; 5.64]
Mean (%)	9.18	36.96	18.15	4.58
Sdev (%)	1.52	4.88	2.86	0.33

Figure 4. Raw spectra of the soybean samples: absorbance vs. wavelength in nm

After data collection, several data processing methods were evaluated. The best results were obtained with a combination of pre-treatment methods: multiplicative scatter correction, averaging, and Savitzky-Golay smoothing. The calibration models were developed with partial least square (PLS) regression using Unscrambler® 9.8 software (commercialized by CAMO Software, Oslo, Norway).

Calibration and Cross Validation Results

Table 3 summarizes each constituent's PLS calibration model.

Table 3. Calibration models used

Constitute	Treatment(s)	Elements	NB PCs	% Outliers	R²	SEC	SECV
Moisture	None	269	5	3.2	0.94	0.36	0.37
Protein	Smooth. Moving Average	276	7	0	0.96	0.91	0.99
Oil	Smooth. Moving Average	274	9	1.1	0.95	0.60	0.65
Fiber	SNV + Smooth. S. Golay	275	10	0.7	0.91	0.10	0.11

The corresponding PLS regression curves for the predicted vs. measured values of each constituent are provided in the figures. Blue font and symbols represent calibration data, red colors represent cross-validation data, the x-axis represents 'reference' data, and the y-axis represents predicted data with the MicroNIR spectrometer.

Calibration and cross validation results of soybean meal, corn, bakery meal, and DDG show comparable results to soybean as well, the details of which can be found in the original report published by Iowa State University.
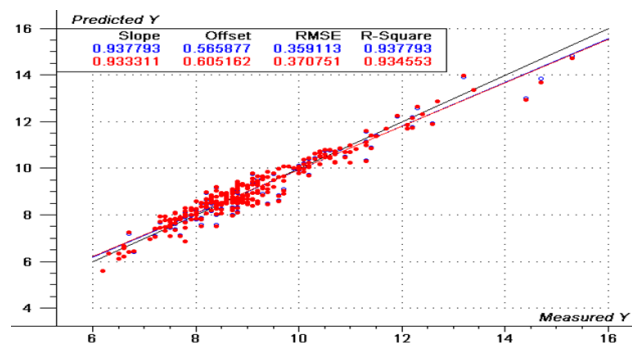

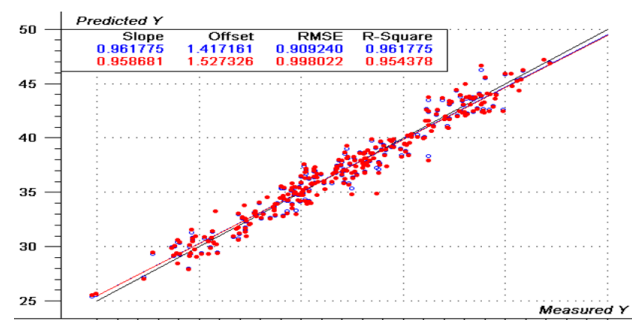Figure 5. PLS regression results for % moisture in soybean

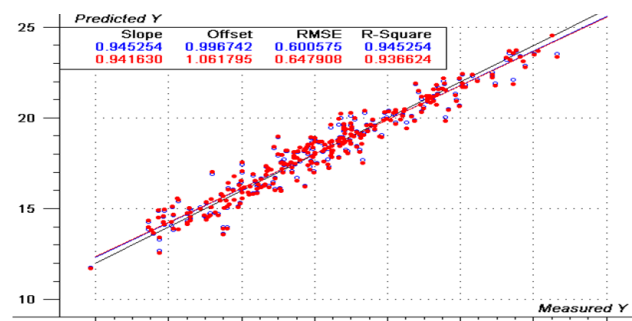Figure 6. PLS regression results for % protein in soybean

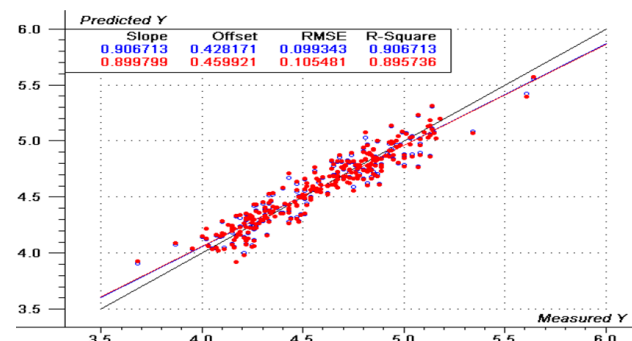Figure 7. PLS regression results for % oil in soybean

Figure 8. PLS regression results for % fiber in soybean

Conclusions

This preliminary study shows that the MicroNIR spectrometer is capable of accurate chemical analysis of grains. One can expect improved results with the use of a rotating cup and in avoiding measurements through a plastic surface. Nevertheless, the performance results are very promising for the food and feed industry. The small size of the MicroNIR spectrometer, cost-effectiveness, and performance will enable grain testing in the field during harvest season, for supplier verification, or at the point of trading.

The independent validation results will be published by the end of summer 2014.

Other food-related projects pertaining to food adulteration are currently being pursued.

Acknowledgments

This application note is an abbreviated version of a larger report and a study conducted by Ms Clémence Biller who was a visiting scientist from Ecole Supérieure dAgriculture de Purpan. Ms Biller was doing an internship at Iowa State University, working in the Grain Quality Lab during the summer of 2013. Special thanks go to Clémence and Prof. Hurburgh for giving us permission to summarize the results here.

References
1. U.S. FDA website, http://www.fda.gov/Food/GuidanceRegulation/FSMA/ucm249243.htm#primer
2. 'Food Fraud and "Economically Motivated Adulteration" of Food and Food Ingredient,' by Renee Johnson, A Congressional Research Service report, 7-5700, www.crs.gov, R43358.
3. U.S. FDA website, http://www.fda.gov/Food/GuidanceRegulation/FSMA/ucm359436.htm

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