Feasibility study of hand-held, light-emitting diode-based, short-wave near infrared transmittance whole grain analyser

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Introduction

Rapid and accurate determination of oil, moisture and protein of grains and oilseeds on the field or at the grain-receiving stations has been a dream for a long time. In Canada, for example, the export of wheat is based on guaranteed minimum protein contents of various protein levels.[1] Accurate protein measurement is essential to the binning and segregation of wheat into the various protein levels. In Europe, the importance of accurate protein analysis of wheat stems from the fact that the price differentiation relies on the protein content of grain.[2]

Near infrared (NIR) spectroscopy has been widely used for determining the moisture and protein content of grains. Karl Norris and his co-workers started work with NIR analysis of grain in the 1960s.[3] In the early days of NIR, they used transmission spectroscopy, but the high scattering of samples combined with non-maturity of technologies and calibration techniques restricted the usefulness of the transmission technology. They then tried using reflectance spectroscopy in the 1800–2500 nm region. Reflection spectroscopy showed its usefulness and it has been widely used to measure wheat protein content. The drawback of this technique is that it requires grinding of the grain. In the 1980s, new instruments and partial least squares (PLS) calibration methods enabled NIR transmittance measurement in the 800–1100 nm region, which eliminated the need for grinding grain samples, while producing results which were as accurate as reflectance instruments.[4]

Although laboratory instruments exist, there is a still need for true field instruments. The field application presents tough challenges to instrument technology. Factors such as varying environmental parameters (for example, temperature and humidity), size and weight requirements, small power consumption, easy calibration and calibration transfer, set specific demands for the analyser technology in field use. Furthermore, the grain analyser should be inexpensive and it should not require any special training for performing the measurements. These requirements are hard to meet with "old" technologies, however well they work in a laboratory environment.

VTT Electronics has a long tradition of developing rugged and reliable on-line and hand-held analysers for various applications. In this work, we have studied the feasibility of the light-emitting diode (LED)-based NIR spectrometer module technology in hand-held grain analysers. The most important results of the wheat grain measurements with the developed hand-held analyser will be presented in this paper.

Maters and methods

Samples

A total of 47 wheat samples, representing different varieties (HRWW, HRSW, SWW, Club, SWSW and SWWW), constituted the data set. These samples came from the United States. The moisture content was analysed just before shipping. Protein had been initially analysed with the Kjeldahl method in two laboratories, with a standard deviation of 0.14 for the two measurements. A plastic sample cup was filled with approximately 300–400 g of whole grain wheat and a fork-type analyser probe was inserted into the sample. No attempt was made to standardise the power needed to insert the probe into the sample. Without removing the sample the probe was inserted into the sample ten times to measure a larger volume and to minimise the sampling error.

Instrumentation

Samples were measured with a GrainGun, the hand-held whole grain analyser prototype (Figure 1) developed at VTT Electronics. The device covers the wavelength range 830–1075 nm, giving a total of 32 data points for each spectrum. The analyser employs a 32 channel short-wave near infrared (SWNIR) spectrometer module based on LEDs, which has been developed by VTT Electronics[5,6] It has a solid glass construction with no air–glass boundaries inside the module. Therefore, it minimises stray light and is highly resistant to moisture condensation, dust and temperature changes. The principle of operation in a typical sample transmission measurement application is illustrated in Figure 2. During operation, the control electronics sequentially drive a linear LED array, electrically scanning the precalibrated wavelength scale of the spectrometer. The radiation from a single LED at a time is directed to a fixed grating monochromator, positioned to give the precalibrated wavelength scale and resolution. A narrow wavelength band of the LED spectrum is transmitted through an optical fibre, which also serves as an exit slit for the spectrometer, to sample optics illuminating the sample. After interacting with the sample, the optical signal is detected and processed into transmittance readings. Each spectrum comprised an average of 64 measurements and the total measurement time was about one second for one subsample. As a background spectrum, we measured the light intensity without a

Figure 1. Photograph of GrainGun, the developed hand-held grain analyser.

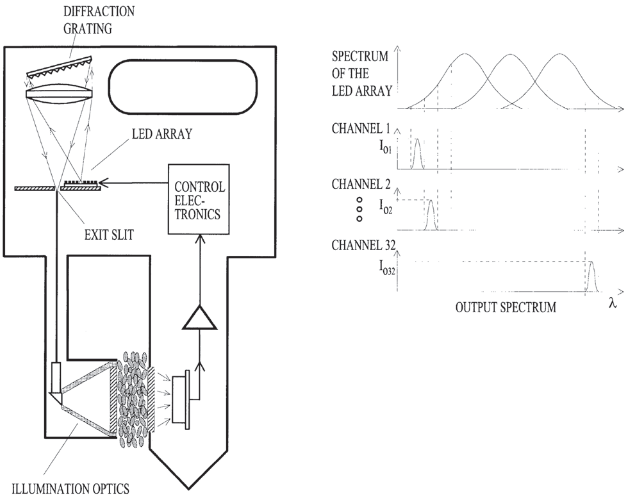

Figure 2. Principle of the LED array spectrometer operation in a typical sample transmission measurement application.

sample between each sample. The transmittance was converted to absorbencies through the following formula

$$A = -\log_{10}\left(\frac{I_i}{I_{0i}}\right) \quad (1)$$

where I_{0i} was the background measurement and I_i the sample measurement with the subscript i representing the wavelength. The thickness of the sample could be varied and the wheat sample thickness in these measurements was set to approximately 18 mm.

In this project, the electronic components were not yet totally integrated inside the handheld probe. Only the electronics needed for controlling the LEDs, detectors and preamplifiers were integrated into the probe. Power supplies, AD converters and the microcontroller were placed in a separate electronics unit, which was connected to a PC through a serial port and data was stored on the PC hard disk.

Software and calibration

All the spectra obtained with the GrainGun were stored in Matlab format and analysed with Data Analysis Toolbox. The NIR transmittance measurements for moisture were made with the first shipped sample set, which was quite small, comprising only 30 samples. Due to the small number of samples, a cross-validation test was used to test the moisture measurement applicability of the hand-held analyser. Absorbency spectra were first mean centred, after which partial least squares (PLS) were used for calibration. For protein measurement (47 samples), we divided the sample set into three different sets and used two of these sets for calibration development and one for testing the calibration. We used each set as a test set and the two remaining sets for calibration, thus coming up with three independent calibrations and tests. In protein calibration, the longest wavelengths were removed from the spectra, because they were very noisy. Therefore, the wavelength range used for protein cali-

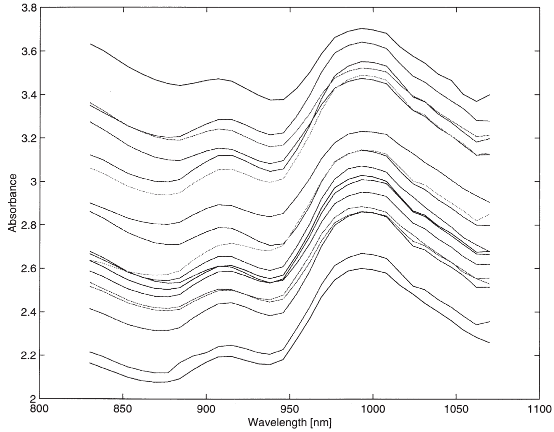

Figure 3. A set of wheat spectra measured with GrainGun.

bration was limited to the range 840–1030 nm. The data was then mean centred and calibrated with PLS. The performance of the calibration was evaluated in terms of standard error of prediction (*SEP*) and standard error of calibration (*SEC*).

Results and discussion

A set of wheat spectra is plotted in Figure 3. As seen in the figure, there is no clear individual absorption band for moisture or protein. The absorption bands are overlapping, thus making it impossible to use any single wavelength calibration. The major wheat constituents absorb in the following wavelengths: moisture at 970 nm, protein at 910 nm and 1020 nm (stronger) as well as starch at 990 nm.[4] The results of the calibrations are presented in Table 1.

As can be seen from the results, the accuracy of moisture measurement is good, whereas the protein measurement accuracy is not yet satisfactory. The main reason for the insufficient performance for protein was the poor signal-to-noise ratio (SNR) at wavelengths over 1000 nm. Unfortunately, the protein absorption maximum is in this region.

High optical output is an essential requirement for the LED spectrometer to achieve a good SNR in the transmission measurements on high-absorbency samples. In this prototype we used an "old" design of the LED spectrometer module. We have, however, improved the design.[5] Figure 4 presents a comparison of the optical output of the developed new prototype unit with the corresponding data of

Table 1. Whole grain wheat calibration of moisture and protein.

	Range	*SEC*	*SEP*	Note
Moisture	7.5–11%		0.137	Cross-validation
Protein set 1	9.7–17%	0.264	0.328	
Protein set 2	9.7–17%	0.229	0.327	
Protein set 3	9.7–17%	0.234	0.319	

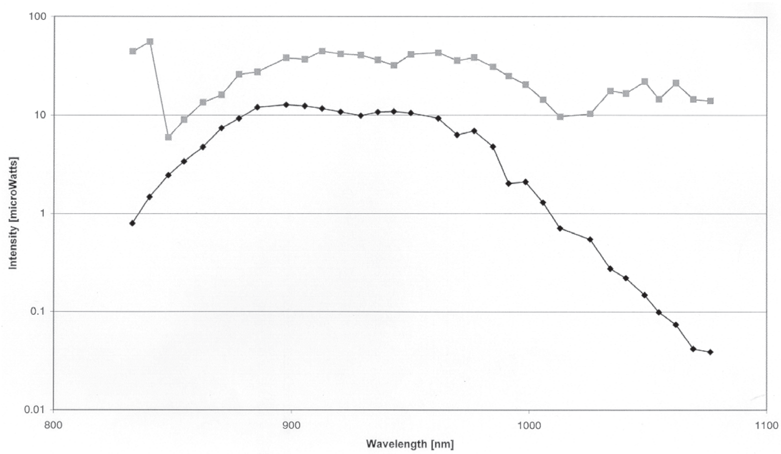

Figure 4. LED spectrometer module output power data, prototype of new design compared with an older design used in this project (—◆— old design —■— new design).

the design [6] used in this project. The improvement in the measured output is remarkable, approximately two decades at the short-wave and long-wave ends of the scale and by a factor of 2 to 5 for the central bands. The improved SNR will have a remarkable effect, especially on the protein measurement accuracy.

Conclusion

In this project we have proved that the handheld LED-based analyser is capable of measuring whole grain wheat moisture accurately. Furthermore, with the improved design, it will also be capable of measuring the protein content. Other potential applications are:

- fruit sugar content
- whole grain moisture, protein, oil
- vegetable maturity
- moisture measurement
- octane measurement

A fixed precalibration of the wavelength scale is expected to improve the possibilities for transferring generic multivariate calibrations to a large number of analysers, thus minimising the calibration costs per application. A solid glass construction is rugged and reliable. Apart from hand-held applications, potential applications are expected to be found in spectroscopic sensors for process automation.

Modifying the spectrometer design to cover further wavelength regions (400–2500 nm) can be used to widen the scope of applications, not only to colorimetry, but also to the whole wavelength region used in NIR spectroscopy. The sample presentation geometry can also be modified for various transmittance, reflectance, transflectance or interactance arrangements, depending on application requirements. We expect that after the first successful demonstrations there will be a rapidly increasing market for small, rugged, easy-to-use and low-cost analysers for a variety of applications in, for example, the food industry, agriculture, pulp and paper industry, petrochemical industry and environmental monitoring.

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