Collaborative development of near infrared calibrations for quality testing of wheat and barley breeding material: 3. Ultra-rapid quality testing of wheat, flour and dough using near infrared diode array spectrometry

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Introduction

Near infrared (NIR) spectroscopic analysis of whole grain has fulfilled the needs for quality testing of early generation plant breeding material to select the most promising lines for quality characteristics. Increasingly, continuous spectra instruments have been used for this purpose in order to extend the range of quality characteristics measured. Recently, a new diode array spectrometer became available. This instrument, the Perten Instruments DA-7000, offers exceptionally fast continuous spectra data acquisition, hence the potential for significant gains in sample turnaround time and throughput. Currently, three of the collaborating laboratories in the Australian Grain Industries Centre for NIR have a DA-7000 instrument, one in up-view mode (Figure 1) and two in down-view mode. We have studied its application in both modes to wheat quality testing including protein, moisture, hardness, flour yield, flour water absorption and dough rheological properties.

The up-view sample presentation option of the DA-7000 lends itself to other uses in addition to whole grain analysis. Powdered samples could be presented to the instrument in an analogous way but cleaning of the platform between samples would be highly inconvenient. However, satisfactory calibrations have been derived when plant tissue samples have been presented to the DA-7000 in up-view mode in plastic bags (A.B. Blakeney, personal communication). We have extended this concept to flour samples.

The DA-7000 with a fibre optic attachment has also been used to record the spectra of dough samples during mixing and thus determine the optimum mixing requirement of a flour sample.[1] This provides an opportunity to streamline one of the most time-consuming aspects of wheat quality assessment.

Materials and methods

Samples

360 wheat samples were collected at grain receival silos in Western Australia, South Australia, Victoria and New South Wales during the 1996/97 harvest. Kjeldahl protein ($N \times 5.7$) and oven moisture determinations on the wheat were performed at BRI.

219 wheat samples were provided by the SARDI Grain Quality Laboratory together with laboratory data for flour yield, grain hardness (particle size index, PSI), Farinograph water absorption, dough extensibility and resistance as measured by the Extensograph and RVA peak height. Kjeldahl protein ($N \times 5.7$) and oven moisture determinations on the wheat were performed at BRI. The wheats represented a set of breeders' lines identified as NIR calibration samples over five years at Roseworthy, South Australia, to represent the range of quality types relevant to the gene pool in that breeding programme. They were grown in 1996 and 1997 in close proximity under the same conditions as the six row plot early generation samples that were screened using the calibrations developed from them.

101 flour samples were prepared using a Bühler MLU 202 Laboratory Mill from wheat samples provided by six regional breeding programmes in Australia. The samples, grown in 1997, represented advanced lines likely to be released together with their controls. Kjeldahl protein ($N \times 5.7$) determinations on the flour samples were performed at BRI.

NIR spectra

NIR spectra were measured as log $1/R$ over the range 400–1700 nm, at 5 nm intervals, using two DA-7000 Diode Array Spectrometers (Perten Instruments, Springfield, IL, USA) located in different laboratories. Wheat was packed in a plastic ring (Instrument 1: BRI) or plastic tray (Instrument 2: SARDI) of 140 mm diameter to a depth of 25 mm and spectra were recorded in the up-view mode (see Figure 1) or down-view mode, respectively, as the average of 30 spectra × 5 re-packs. Typical analysis time was 15 s per re-pack.

Two methods were used to present flour samples to Instrument 1. In the first method, each sample was contained in a plastic bag approximately 150 mm × 85 mm. Since the area of illumination was a circle of diameter 125 mm, a mask was cut from a sheet of matt black filter paper to prevent light pass-

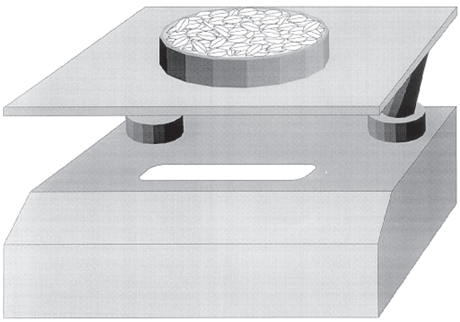

Figure 1. Perten DA-7000 diode array instrument in up-view mode with a grain sample packed in a ring.

ing around the sample when placed on the platform in the up-view mode. A wooden block, covered with matt black filter paper, was used to compress the sample. In the second method, a fibre optic reflectance probe was inserted into the bag containing the flour sample. In each case, data were recorded as the average of 30 spectra.

Calibration development

The raw log 1/*R* data in the range 800–1700 nm were corrected for the effects of scatter using *SNV* and detrend and transformed into second derivative (40 nm gap). Calibrations were derived by modified PLS regression. The optimum number of factors was determined by the point at which no further improvement was made to the standard error of cross-validation (*SECV*). The SECV was obtained by sequentially removing one sample at a time from the calibration set, calibrating using the remaining samples and predicting the one removed. This represents the best estimate of accuracy obtainable from the calibration set.

Results

The results of calibrations derived using the three sets of samples on the two instruments are given in Tables 1–3.

Discussion

The calibration and validation results for both protein and moisture, using the two DA-7000 instruments in either up-view and down view mode (Tables 1 and 2), were all within the guideline *SECV*s given in the Official Australian Method for protein in whole wheat by NIR.[2]

Table 1. Calibration results for wheat receival samples using Instrument 1.

Constituent	*SECV* (%)
Protein	0.24
Moisture	0.21

Table 2. Calibration results for wheat samples on two instruments.

Constituent	Instrument 1 (1997 samples)		Instrument 2 (1998 samples)	
	R^2	*SECV*	R^2	*SECV*
Protein	0.95	0.20%	0.90	0.21%
Moisture	0.86	0.18%	—	—
Flour yield	0.18	1.2%	0.48	1.0%
PSI	0.68	1.6	0.82	1.6
Water abs	0.59	1.5%	0.77	1.4%
Extensibility	0.13	85 mm	0.38	50 mm
Resistance	0.35	1.7 BU	0.54	0.9 BU

Table 3. Calibration results for flour samples using Instrument 1.

Sample presentation	R^2	*SECV* (%)
Plastic bag	0.99	0.19
Fibre optic probe	0.98	0.23

The results of calibrations for a number of wheat quality parameters given in Table 2 show that there was good agreement in the *SECV*s between two different sets of samples measured on two different instruments. The 1997 samples were measured on the BRI instrument in the up-view mode and the 1998 samples were measured on the SARDI instrument in down-view mode. The apparent improvement in the 1998 results may have been due to the fact that the reference analyses were carried out in duplicate and the average results used for calibration. Allen *et al.*[3] reported a calibration for flour yield by NIR measurements on whole grain using a Grainspec with an *SEC* of 1.4% and *SEP* of 1.62%. The DA-7000 results (Table 2) are considerably better than this. Pawlinsky and Williams[4] reported the following *SEP* values for whole wheat calibrations derived using a monochromator which recorded data over the range 400 to 2500 nm: protein—0.15%; particle size index (PSI)—1.75%; water absorption—1.43%; Extensibility—85 mm. The results given in Table 2 for the DA-7000 are equal to, or better than, these figures yet the DA-7000 can enable a sample throughput of up to 400 samples per hour.

The results for protein in white flour (Table 3) show that the accuracy based on presentation of samples in plastic bags was within the limit specified in ICC Recommendation No. 202.[5] On the other hand, the accuracy obtained using the fibre optic probe was significantly worse ($p < 0.05$) according to a t-test.[6] However, this is probably due to the much smaller area of illumination and could no doubt be improved by basing the analysis on the average of multiple measurements.

Typical spectra of whole wheat recorded using the DA-7000 in up-view mode are shown in Figure 2. The DA-7000 has both a visible and an NIR array. The spectra in Figure 1 represent the data produced with the NIR array covering the wavelength range 800 to 1700 nm with a data interval of 6 nm. Thus, the amount of data is less than that recorded using grating monochromator instruments. Never-

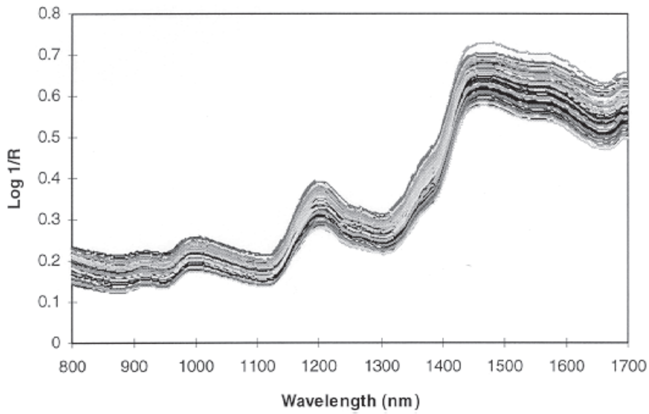

Figure 2. NIR spectra of whole wheat grain samples.

theless, the performance of the DA-7000 over a range of wheat quality tests has been found to be equivalent to results reported in the literature.[4]

Conclusion

This study has demonstrated that a single NIR instrument can be used for analysis of whole wheat, flour and dough very rapidly and cost-effectively.

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