Whole wheat grain hardness measurement by near infrared spectroscopy

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Introduction

Empirical calibrations using near infrared (NIR) spectroscopy are often performed without knowing what is measured or understanding the basis of the measurement. In other words, the NIR spectrophotometer is often used as a “black box”.

Successful NIR hardness measurements on ground wheat grain are based on light scattering. If NIR hardness measurements on whole wheat grain are also based on light scattering this scattering effect could be used to predict hardness on whole grain. Potentially useful techniques not previously applied to whole grain are:

- multiplicative scatter correction (MSC)
- area under the second derivative curve (Area)
- principal component analysis (PCA)

Materials and methods

Samples

A set of 104 wheat samples comprising both UK and Canadian varieties was assembled. The ground samples were prepared using a UDY Cyclone mill and a Model 3100 Falling Number hammer mill both fitted with 1 mm screens. The wheat hardness was assessed by particle size index (PSI)[1] and air jet sieve (AJS)[2] methods, respectively. After sorting the samples in order of increasing PSI values, the 104 samples were divided into a calibration set (63 samples) and a validation set (41 samples) by assigning the first three samples into the calibration set, the following two into the validation set until all the samples had been allocated.

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NIR reflectance spectroscopy

Spectra of the ground and whole wheat grain samples were recorded as log l/*R* at 2 nm intervals from 1100–2500 nm using an NIRSystems Model 6500 monochromator and the standard and coarse sample cells, respectively.

Calibrations

Empirical calibrations

Infrasoft International (ISI) software was used to derive and validate NIR calibrations for wheat hardness by PSI and AJS on ground and whole wheat grain using partial least squares regressions (PLS). The best equation was selected based on the lowest standard error of performance (SEP).

Alternative calibrations

The scatter effect was separated from the spectra using three different methods:

- multiplicative scatter correction (MSC)
- the area between the second derivative curve and the wavelength axis (Area)
- principal component analysis (PCA)

Linear regressions were performed in each case to predict wheat hardness by PSI and AJS.

Results and discussion

The calibration results obtained are shown in Tables 1 and 2 for PSI and AJS, respectively, in terms of standard error of calibration (*SEC*), correlation coefficient (*r*) and standard error of performance (*SEP*).

The results in Tables 1 and 2 indicate that these alternative calibrations do not show any significant improvement ($p < 0.05$) in the *SEP* results compared to the empirical calibrations. Althought not significant at $p < 0.05$, the 1st and 2nd principal components for whole wheat grain reflectance did show an improvement in *SEP* compared to the empirical calibrations.

It is known that the plot of the loadings of the 1st PC normally has the shape of the mean spectrum[3] indicating that the scatter represents the largest single source of variation in the data set. This was observed for the ground wheat grain spectra where the spectral pattern of the 1st PC was similar to the mean spectrum with the 2nd PC indicating variation due to moisture. However,

Table 1. Calibration and validation results for PSI.

	Empirical	MSC	Area	PCA[a]
Ground grain reflectance	*SEC* = 1.74	*SEC* = 2.80	*SEC* = 2.90	*SEC* = 3.02
	r = 0.97	*r* = 0.92	*r* = 0.91	*r* = 0.91
	SEP = 1.94	*SEP* = 3.55	*SEP* = 2.92	*SEP* = 2.98
Whole grain reflectance	*SEC* = 3.31	*SEC* = 5.45	*SEC* = 7.00	*SEC* = 5.24
	r = 0.88	*r* = 0.64	*r* = 0.17	*r* = 0.68
	SEP = 3.96	*SEP* = 4.90	*SEP* = 5.52	*SEP* = 3.84

[a]1st PC for ground grain reflectance and 1st and 2nd PCs for whole grain reflectance.

Table 2. Calibration and validation results for AJS.

	Empirical	MSC	Area	PCA[a]
Ground grain reflectance	*SEC* = 1.37	*SEC* = 2.61	*SEC* = 3.02	*SEC* = 2.62
	r = 0.98	*r* = 0.93	*r* = 0.90	*r* = 0.93
	SEP = 1.45	*SEP* = 2.59	*SEP* = 2.24	*SEP* = 2.08
Whole grain reflectance	*SEC* = 3.01	*SEC* = 5.08	*SEC* = 6.68	*SEC* = 4.89
	r = 0.90	*r* = 0.68	*r* = 0.26	*r* = 0.71
	SEP = 3.76	*SEP* = 4.40	*SEP* = 4.70	*SEP* = 3.53

[a]1st PC for ground grain reflectance and 1st and 2nd PCs for whole grain reflectance.

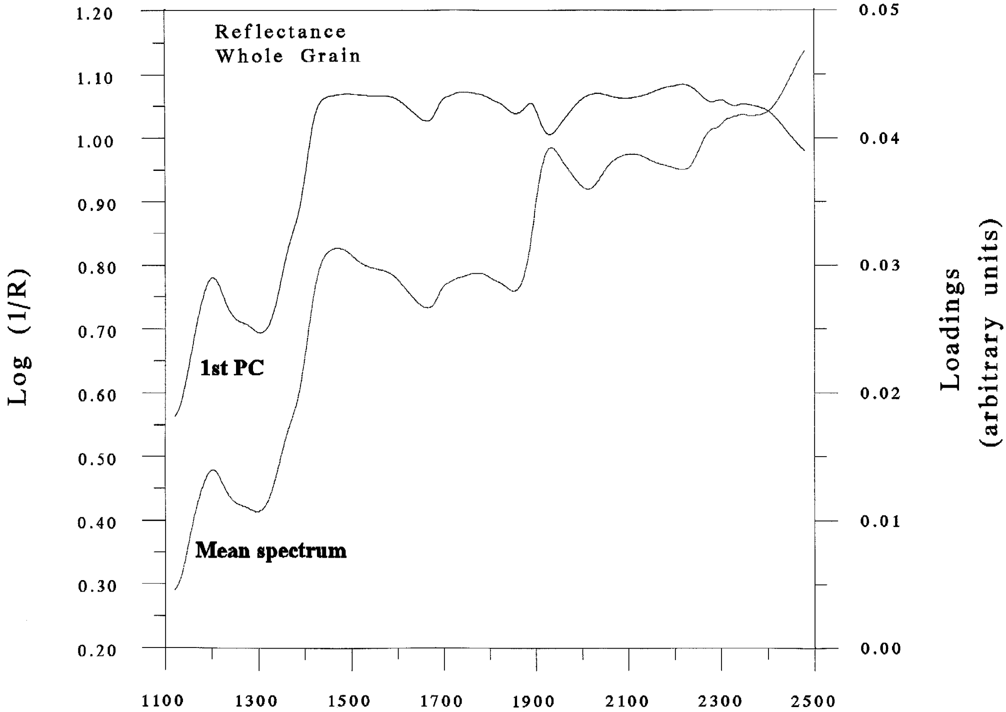

Figure 1. Mean spectrum and 1st PC.

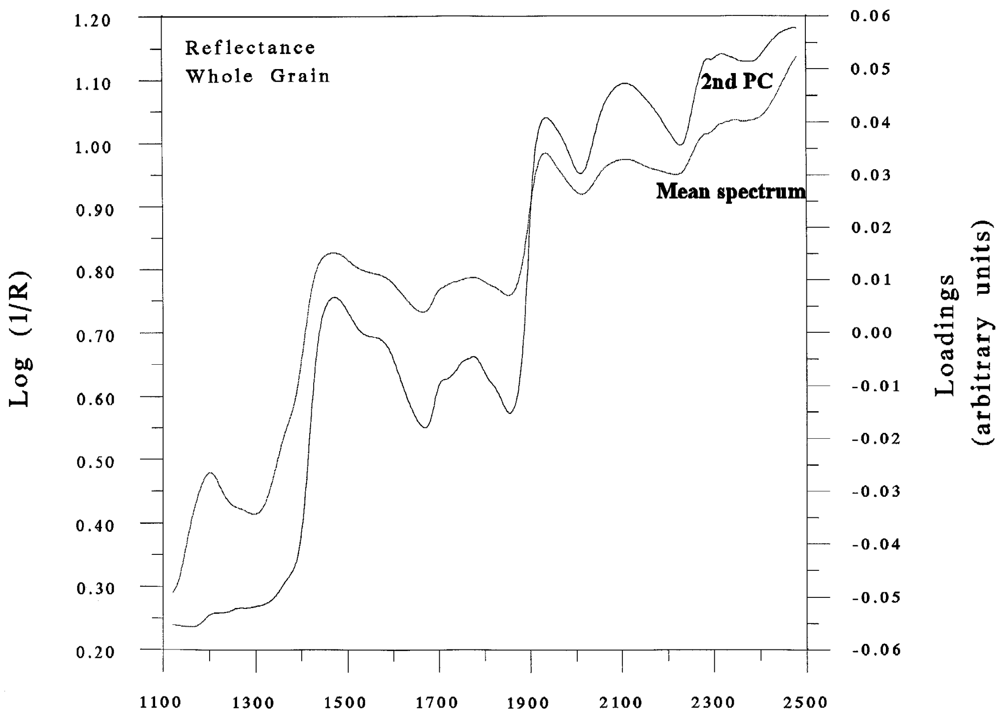

Figure 2. Mean spectrum and 2nd PC.

in the case of the whole wheat grain reflectance spectra, the spectral pattern of the 1st PC (Figure 1) did not completely follow the shape of the mean spectrum. It was interesting to observe the similarity of the 2nd PC to the mean spectrum (Figure 2), with only the 3rd PC indicating variation due to moisture. This supports the use of the first two principal components in the whole grain reflectance calibrations and only the 1st for ground wheat grain reflectance.

From these results it is clear that the 1st PC of ground wheat grain accounts for all the variation within the data set that describes wheat hardness, whereas for whole wheat grain the first two principal components account for the variation within the data set that describes wheat hardness.

No significant differences ($p < 0.05$) were found between wheat hardness calibrations using AJS and PSI as reference methods.

Conclusions

For whole wheat grain reflectance hardness calibrations, using the 1st and 2nd principal components proved to be the best. There is also the benefit of using fewer terms and no risk of overfitting as could be the case with empirical calibrations. These results show that pure scatter, as measured in the case of MSC and Area, is inadequate to describe hardness.

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