Differentiating Vitreous and Nonvitreous Durum Wheat Kernels by Using Near-Infrared Spectroscopy

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ABSTRACT

The vitreousness of durum wheat is used by the wheat industry as an indicator of milling and cooking quality. The current visual method of determining vitreousness is subjective, and classification results between inspectors and countries vary widely. Thus, the use of near-infrared (NIR) spectroscopy to objectively classify vitreous and nonvitreous single kernels was investigated. Results showed that classification of obviously vitreous or nonvitreous kernels by the NIR procedure agreed almost perfectly with inspector classifications. However, when difficult-to-classify vitreous and nonvitreous kernels were included in the analysis, the NIR procedure agreed with inspectors on only 75% of kernels. While the classification of difficult kernels by NIR spectroscopy did not match well with inspector classifications, this NIR procedure quantifies vitreousness and thus may provide an objective classification means that could reduce inspector-to-inspector variability. Classifications appear to be due, at least in part, to scattering effects and to starch and protein differences between vitreous and nonvitreous kernels.

The vitreousness of durum wheat (*Triticum durum*) has traditionally been regarded by the wheat industry throughout the world as an important quality factor. When compared to nonvitreous kernels, vitreous kernels generally have improved cooking quality, better pasta color, coarser granulation, higher protein content, are harder, and sell for a premium (Matsuo and Dexter 1980, Hoseney 1986, Blanco et al 1988). While most countries that market durum wheat include vitreousness as measure of quality, methods for estimating this quality factor vary widely. Menger (1973) reported that when inspectors from 17 countries were presented the same samples, the percentage of kernels they determined as vitreous had a range of 48–98%.

In the United States, the Grain Inspection, Packers and Stockyards Administration (GIPSA) uses specific criteria to visually determine whether durum wheat is vitreous (USDA 1997). Vitreous kernels appear glassy and translucent, whereas nonvitreous kernels appear starchy and opaque. Kernels that have mottled or chalky spots, regardless of size, are not vitreous. In addition, green immature kernels, kernels affected by scab, sprouted kernels, foreign material, and all other classes of wheat are not considered vitreous. However, bleached hard and vitreous kernels, or kernels that have cracks or checks that cause a cloudy or shadowy spot on the kernels are considered vitreous. Thus, incorrect visual classifications can occur if, for example, an inspector does not observe a small chalky spot, or if a shadowy spot causes a kernel to be incorrectly classified as nonvitreous.

Because vitreousness is an important quality factor worldwide, an objective and consistent means of measuring vitreousness is needed. Near-infrared (NIR) spectroscopy has been used to measure other wheat quality factors such as hardness (Delwiche 1993), color (Wang et al 1999), scab (Dowell et al 1999), internal defects (Dowell et al 1998), and protein content (Delwiche 1995). The previous success of NIR to measure other quality factors and the recent development of automated handling and NIR scanning of single kernels (Dowell et al 1998) indicates that this technology may provide the wheat industry with an objective tool to classify vitreous and nonvitreous wheat kernels. The objective of this research was to determine whether NIR spectroscopy could be used to classify vitreous and nonvitreous single kernels of durum wheat.

MATERIALS AND METHODS

GIPSA provided samples of crop year 1997 durum wheat that were separated into vitreous and nonvitreous components by the Board of Appeals and Review (BAR). These commercial samples originated from California, Minnesota, Illinois, South Dakota, and Arizona. The samples represented all grades for that year and were part of a larger ARS-GIPSA study to develop predictive tests for milling quality factors. Protein content, as measured by the BAR using standard NIR procedures, had a range of ≈9.8–15.0% and the percentage of vitreous kernels in each sample was ≈63–100%. All samples were equilibrated to the same ambient moisture content.

Spectra from 10 vitreous and 10 nonvitreous single wheat kernels from each of 62 samples (n = 1,240 kernels) were collected using a diode-array NIR spectrometer (DA 7000, Perten Instruments, Springfield, IL) integrated with a single kernel wheat characterization system (SKCS 4100, Perten Instruments, Springfield, IL). The DA 7000 measures absorbance at 400–1,700 nm by using arrays of silicon and indium-gallium-arsenide sensors and collects data at a rate of 30 spectra/sec. The SKCS 4100 can automatically deliver single kernels to a black container in the spectrometer viewing area at a rate of 2 kernels/sec. However, to minimize errors caused by kernel movement that can occur during automated spectra collection, the system was momentarily stopped for ≈15 sec after each kernel was automatically placed in the viewing area. Kernels were randomly orientated, and the entire kernel was in the sensor field-of-view. Six log (1/R) spectra were collected from each kernel, averaged to reduce noise and recorded in 5-nm increments. The black container served as the reflectance reference for calculating absorption spectra. These spectra were stored for subsequent analysis by using GRAMS/32 software (Galactic Industries Corp., Salem, NH).

Additional tests included 10 vitreous and 10 nonvitreous kernels from 20 of the above samples (n = 400 kernels). Only kernels that were easily distinguishable as completely vitreous or nonvitreous were included in these tests, whereas the previous test included kernels that may have been difficult to clearly visually classify as vitreous or nonvitreous. Each kernel was hand-placed in the viewing area.

Spectra were analyzed by using partial least squares (PLS) regression (Martens and Naes 1989). All spectra were mean centered before analysis. The data were analyzed by assigning values of 0.0 to vitreous kernels and 1.0 to nonvitreous kernels in the calibration set. The class of kernels predicted by the calibration was determined by how close the predicted value was to 0.0 or 1.0. If the calibration is perfect, all vitreous kernels will have a predicted value <0.5 and all nonvitreous kernel will have a pre-

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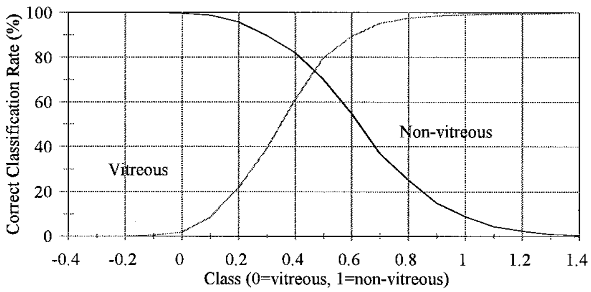

Fig. 1. Classification of representative vitreous and nonvitreous durum single wheat kernels using near-infrared (NIR) spectroscopy.

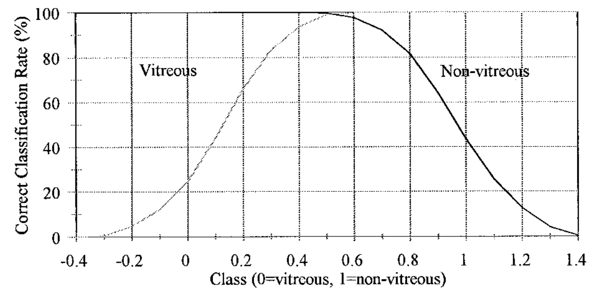

Fig. 2. Classification of obviously vitreous and nonvitreous durum single wheat kernels using near-infrared (NIR) spectroscopy.

TABLE I
Calibration and Classification Statistics Resulting from a Partial Least Squares (PLS) Cross-Validation When Classifying Vitreous and Nonvitreous Durum Wheat Kernels Using Near-Infrared (NIR) Spectra

Sample Set	n	f[a]	r^2	Correct Classification Rate
All kernels[b]	1,240	13	0.26	75%
Obvious vitreous or nonvitreous[c]	400	5	0.85	99.8%

[a] Number of PLS regression factors.
[b] Includes all types of vitreous and nonvitreous kernels present in the original samples.
[c] Only kernels clearly distinguishable as vitreous or nonvitreous included.

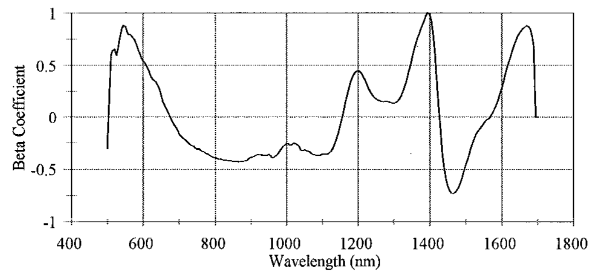

Fig. 3. Partial least squares analysis beta coefficients indicating wavelengths used in calibrations to classify vitreous and nonvitreous durum wheat.

dicted value >0.5. If kernels are not predicted with 100% accuracy when a cut-off value of 0.5 is selected, then that cut-off value can be increased or decreased to correctly classify more of one kernel class but usually at the expense of incorrectly classifying more of the other class. A perfect calibration was not expected because the reference determination of vitreousness determined by the BAR is a subjective measurement and therefore prone to some variability.

Cross-validation and prediction sets were used in the analyses. Cross-validation attempts to emulate predicting unknown samples by using the training data set itself. To do this, one sample was removed from the data set, a calibration was developed with the remaining samples, then the removed sample was predicted. This was repeated for all samples. A calibration was selected that resulted in the lowest residual sum of squares when using the least number of factors. Including more factors in calibrations can improve predictions, but including too many can over-fit the data.

To ensure the cross-validation calibration did not over-fit the data, a separate calibration was developed by randomly selecting 50 of the 62 samples (80%) for a calibration and predicting the vitreousness of the remaining kernels. The results from the prediction set were then compared to results obtained when those same kernels were included in the cross-validation set.

PLS reports the importance of wavelengths used in calibrations as beta coefficients. For any given wavelength, the absolute value of its beta coefficient multiplied by its spectral value indicates how important that wavelength was for classification. Thus, beta coefficient plots can be compared to NIR absorptions of specific wheat components such as protein or starch to indicate what causes unique NIR absorptions between vitreous and nonvitreous kernels.

RESULTS AND DISCUSSION

Figure 1 shows classification results achieved when all kernels were predicted using a calibration resulting from the cross-validation procedure (Table I). About 80% of vitreous and 70% of nonvitreous kernels were correctly classed when a value of 0.5 was used to discriminate between the two classes. Choosing a higher or lower value to discriminate between vitreous and nonvitreous kernels causes more kernels of one type to be correctly classed but usually at the expense of incorrectly classing more of the other type. For example, because vitreous and nonvitreous kernels were assigned a value of 0 and 1, respectively, in the calibration, selecting a discrimination value of 0.7 will result in ≈95% of vitreous and ≈35% of nonvitreous kernels being correctly classed.

The failure to completely discriminate between vitreous and nonvitreous kernels using NIR spectra may be due to the subjectivity involved in determining whether kernels are vitreous, or the inability of NIR to detect some defects such as small spots that cause kernels to be nonvitreous. To further test the ability of NIR to detect nonvitreous wheat, 10 kernels of distinctly vitreous and nonvitreous wheat kernels selected from each of the 20 samples were classed (Fig. 2). The PLS cross-validation results (Table I) showed almost perfect classification of vitreous and nonvitreous kernels when using a value of 0.5 to discriminate between the two classes.

Figure 3 shows beta coefficients resulting from the five factor PLS calibration of the distinctly vitreous and nonvitreous wheat kernels. Peaks occurred at ≈550, 1,200, 1,400, 1,465, and 1,670 nm. The peak at 550 nm indicates that some color differences exist between vitreous and nonvitreous samples, which was expected. The peaks at 1,670 and 1,200 nm occur around C-H 1st and 2nd overtones. The peak at 1,400 nm occurs around the C-H 1st combination overtone. The peak at 1,465 nm occurs around the N-H and O-H 1st overtones (Murray and Williams 1990). These same peaks, plus additional peaks, were observed in the beta coefficient plot resulting from the 13 factor calibration that included all kernels. The additional peaks in the 13 factor calibration are probably due to PLS attempts to explain the variation introduced by kernels that may not be distinctly vitreous or nonvitreous. Proper interpretation of the wavelength influence on classifications requires multiplication of beta coefficients by the absorbance spectra. However, beta coefficients were ≈10× larger than absorbance values and the product did not change interpretations. The difference spectrum calculated by subtracting an average nonvitreous kernel spectrum from an average vitreous kernel spectrum also shows peaks corresponding to the beta coefficient peaks (Fig. 4). Only obviously vitreous and nonvitreous kernels were used to calculate the difference spectrum. The positive value of the difference spectrum indicates that scattering probably influenced absorbance spectra and classifications.

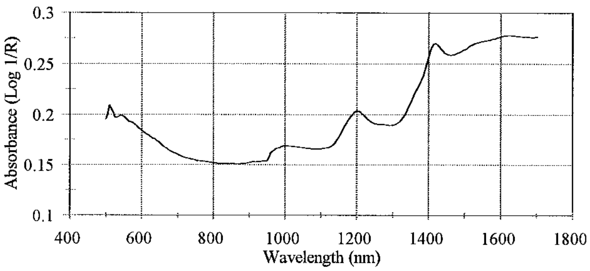

Fig. 4. Difference spectrum calculated by subtracting the average near-infrared (NIR) spectra of nonvitreous kernels from vitreous kernels.

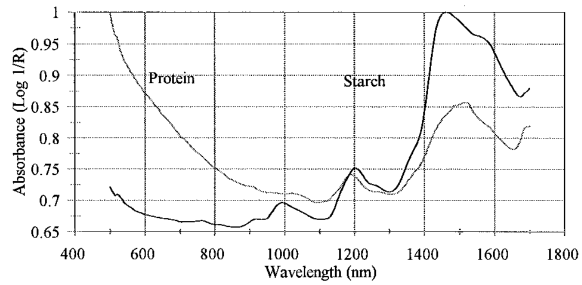

Fig. 5. Near-infrared (NIR) spectra of wheat protein and starch.

Protein and starch both absorb strongly at ≈1,200 nm and at ≈1,400–1,700 nm (Fig. 5). Thus, the differences in starch and protein in vitreous and nonvitreous kernels probably contribute to NIR classifications. Hoseney (1986) reported that the starch-protein bond in wheat is associated with vitreousness. Thus, it is reasonable that differences in the starch and protein NIR absorption regions are used by the PLS regression to classify vitreous and nonvitreous kernels. To further investigate whether protein, starch, and hardness significantly influence classifications, the 700–1,250 nm and 1,400–1,700 nm regions reported in literature (Tkachuk 1990, Shenk et al 1992, Delwiche 1993) for measuring these constituents were removed from the analyses. For the 400 obviously vitreous and nonvitreous samples, the classification accuracies when using the 500–700 nm, 1,250–1,400 nm, or both, regions were ≈96.8, 99.3, and 99%, respectively. Thus, information outside the ranges typically used for protein, starch, and hardness are useful for vitreous classifications and further indicates that scattering may contribute to classifications. The high classification accuracy resulting from the visible wavelength region (500–700 nm) agrees with results reported by Wishna (1998). A regression between vitreousness and protein content showed no significant relationship ($r^2 = 0.10$), further indicating information outside the protein absorption region is used for classifications.

The average spectra for obviously vitreous and nonvitreous kernels are shown in Fig. 6. Shape differences between the two curves are more readily seen in the difference spectra shown in Fig. 4. While the spectra appear similar in shape, the vitreous kernels have higher absorbance values. This probably occurs because nonvitreous kernels have air spaces that diffract and diffuse light, which causes kernels to appear opaque (Hoseney 1986). This results in more incident energy reflected to the NIR sensor and is recorded as lower absorbance values for nonvitreous kernels. Vitreous kernels are more tightly packed and have less air spaces to diffract light. Thus, light entering a vitreous kernel is more likely to pass through the kernel and results in a higher absorbance measured by the NIR sensor. This relationship between vitreousness and optical density agrees with that reported by Delwiche (1993). The spectra shown in Fig. 6 are averages of distinctly vitreous or nonvitreous kernels, and differences in most vitreous and nonvitreous kernels are not this pronounced.

When reanalyzing data by removing 20% of the kernels, developing new calibrations, and predicting kernels not in the calibration, results were similar to those achieved by the cross-validation calibration (Table II). That is, 100% of obviously vitreous and nonvitreous kernels in the prediction set were correctly classed. For the larger sample set, ≈72% of kernels in the prediction set were correctly classed, whereas 73% of these same kernels were correctly classed when included in the calibration set. Thus, the calibration developed from the cross-validation using all kernels did not over-fit the data.

TABLE II
Classification Accuracy of Kernels When Included (Calibration Set) or Left Out (Prediction Set) of Calibrations Developed Using a Partial Least Squares (PLS) Regression

		Correct Classification Rate	
Sample Set	*n*	Prediction Set	Calibration Set
All kernels[a]	240	72%	73%
Obvious vitreous or nonvitreous[b]	80	100%	100%

[a] Includes all types of vitreous and nonvitreous kernels present in the original samples.
[b] Only kernels clearly distinguishable as vitreous or nonvitreous included.

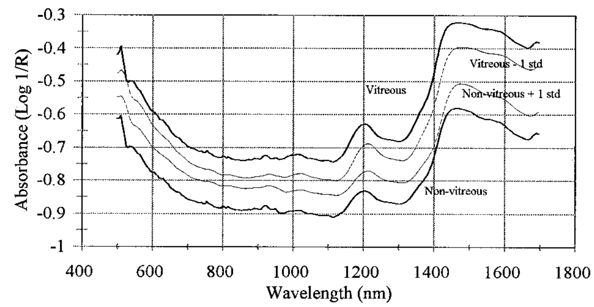

Fig. 6. Average and standard deviation of near-infrared (NIR) spectra of vitreous and nonvitreous durum wheat.

In summary, results showed that NIR spectroscopy can be used to quantify the vitreousness of durum wheat, possibly because of protein, starch, or scattering effects on NIR absorption. While this technology may not replace current visual classifications because it did not agree well with inspectors when kernels were not obviously vitreous or nonvitreous, it may provide a rapid, objective, and consistent means of measuring kernel vitreousness. In addition, this technology may be adaptable to rapid, on-line detection and sorting kernels based on levels of vitreousness. Additional research is needed to see whether these results are valid for other crop years and to compare the NIR procedure with some means of quantifying vitreousness.

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