Evaluation of a Near-Infrared Reflectance Spectrometer as a Granulation Sensor for First-Break Ground Wheat: Studies with Hard Red Winter Wheats[1]

M. C. Pasikatan,[2,3] J. L. Steele,[4] E. Haque,[5] C. K. Spillman,[6] and G. A. Milliken[7]

Cereal Chem. 79(1):92–97

ABSTRACT

A single wheat class or blended wheats from two wheat classes are usually milled in a flour mill. A near-infrared (NIR) reflectance spectrometer, previously evaluated as granulation sensor for first-break ground wheat from six wheat classes, was evaluated for a single wheat class, hard red winter (HRW) wheat, using offline methods. The HRW wheats represented seven cultivars ground by an experimental roller mill at five roll gap settings (0.38, 0.51, 0.63, 0.75, and 0.88 mm) which yielded 35 ground wheat samples each for the calibration and validation sets. Granulation models based on partial least squares regression were developed with cumulative mass of size fractions as a reference value. Combinations of four data pretreatments (log 1/R, baseline correction, unit area normalization, and derivatives) and subregions of the 400–1,700 nm wavelength range were evaluated. Models that used pathlength correction (unit area normalization) predicted well each of the four size fractions of first-break ground wheat. The best model, unit area normalization and first derivative, predicted all the validation spectra with standard errors of performance of 3.80, 1.29, 0.43, and 0.68 for the >1041, >375, >240, and >136 μm size fractions, respectively. Ground HRW wheats have narrower particle size distribution and better sieving properties than ground wheat from six wheat classes. Thus, HRW wheat granulation models performed better than the previously reported models for six wheat classes.

The granulation curve, which is a form of particle size distribution, is a tool of flour millers to monitor the wheat grinding process and to make necessary adjustments in the milling system. Granulation curve shows the cumulative percent mass of sieved wheat fractions greater than the sieve size (vertical axis) against the corresponding sieve sizes (horizontal axis). It can give information on roll corrugation condition, roll gap setting, and sifter efficiency (Posner and Hibbs 1997). The development of an online granulation sensor for ground wheat as the basis for roll gap automation of roller mills has been proposed by Pasikatan (2000). A system that would adjust roll gaps based on granulation information from sensors could help optimize flour milling and improve mill's profitability. The justifications for first-break as a logical starting point for granulation sensing studies were discussed in Pasikatan et al (2001).

Near-infrared (NIR) reflectance spectrometry has been identified as a potential technique for granulation sensing because of its dual sensitivity to absorbing compounds and to the particle size of a ground sample (Wendlandt and Hecht 1966; Kortüm 1969; Osborne et al 1981), its rapidity, and availability of fiber optic probes for remote measurement applications (Robertson et al 1989; Bickel 1989; O'Neil et al 1998). Theoretical equations relating absorption intensity or absorption coefficient to particle size have been derived by Duyckaerts (1955) and Felder (1964). Bull (1990) derived a simplified version of Kubelka-Munk equation for NIR reflectance that modeled the dependence of reflectance on particle size. Successful studies using NIR reflectance as particle size sensor for the range ≤400 μm have been cited in Pasikatan et al (2001). NIR reflectance was recently used to determine cumulative particle size distribution (11 size fractions, size range 5.8–564 μm) of microcrystalline cellulose (O'Neil et al 1999). Pasikatan et al (2001) evaluated NIR reflectance as a granulation sensor for first-break ground wheat using six wheat classes in an attempt to develop a universal calibration for sizes across wheat classes. Their results showed that the best granulation model for six wheat classes predicted 55 out of 60 validation spectra with standard errors of performance of 4.07, 1.75, 1.03, and 1.40 for the >1041, >375, >240, and >136 μm size fractions, respectively.

Flour mills are designed to mill either a single wheat class or blended wheats from two wheat classes, not different classes of wheat. Hence, it is necessary to evaluate the performance of NIR reflectance-granulation models using a single wheat class. Hard red winter (HRW) wheat was chosen for this study because it is the prevalent wheat class planted in Kansas. The objectives of this study were to 1) evaluate using offline methods an NIR reflectance spectrometer as a sensor for granulation of HRW first-break ground wheats, 2) develop and validate NIR reflectance granulation models, and 3) compare with the previously developed granulation models for six wheat classes.

MATERIALS AND METHODS

The seven cultivars of HRW wheats originated from 1996 and 1999 crop years (Table I). Two sets of representative samples (five samples per cultivar) were obtained for calibration and validation. The equipment and procedure used for cleaning, sampling, and measurement of physical properties were described in Pasikatan et al (2001). Wheats were ground using an experimental (first-break) roller mill described by Fang et al (1997). The roller mill settings used in this study were 52.3 rad/sec (500 rpm) fast roll speed, 20.9 rad/sec (200 rpm) slow roll speed, 2.5:1 roll speed differential, and 1.34 kg/m/sec feed rate. Roll gaps were set at 0.38, 0.51, 0.63, 0.75, and 0.88 mm using a feeler gage. These settings were selected so that the variations in granulation of the wheat samples could come only from wheat cultivars and roll gap and to enable comparison with the six wheat class models reported by Pasikatan et al (2001). The 440-g milling samples were tempered and ground using the procedure reported by Pasikatan et al (2001).

Ground wheats were collected and mixed manually 20× using the method described by Kaye and Naylor (1972) before sampling. Three representative parts from the 440-g sample ground wheats were obtained: one of 220 g (for NIR presentation) and two of 110 g (for sifting). The first 110-g ground wheats were sieved to determine the granulation, whereas the second 110-g ground wheats were set aside for a related study. Ground wheats were sieved for 2 min using a laboratory sifter (Great Western Co., Leavenworth, KS) with a stack of 20W (1,041 μm opening), 50GG (375 μm), 70GG

[1] Contribution no. 01-169-J from the Kansas Agricultural Experiment Station.

[2] Corresponding author. Email: choypc@gmprc.ksu.edu Phone: 785-776-2727. Fax: 785-776-2792.

[3] USDA-ARS, Grain Marketing and Production Research Center, Manhattan, KS 66502. Former graduate student, Department of Biological and Agricultural Engineering, Kansas State University, Manhattan, KS 66506. Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by the USDA implies no approval of the product to the exclusion of others that may also be suitable.

[4] Formerly USDA-ARS, Grain Marketing and Production Research Center, Manhattan, KS 66502.

[5] Department of Grain Science, Kansas State University, Manhattan, KS 66506.

[6] Department of Biological and Agricultural Engineering, Kansas State University, Manhattan, KS 66506.

[7] Department of Statistics, Kansas State University, Manhattan, KS 66506.

Publication no. C-2001-1204-06R.

(240 μm), and 10XX (136 μm) sieve and a pan. The mass of sieves and sieved fractions were measured by a digital balance (model DI-4KD, Denver Instruments, Arvada, CO) with 0.01-g resolution.

The NIR reflectance spectrometer evaluated for offline granulation sensing was a diode array type (Perten Instruments, Reno, NV). It was used to collect absorbance (log (1/R)) data from 400 to 1,700 nm at 5-nm increments and was set to collect 30 scans that were averaged. Four spectra were collected for each ground wheat sample and were averaged to reduce noise. The procedure and method of presenting ground wheat to the NIR spectrometer, and the formation of calibration file were described in Pasikatan et al (2001). Two replicate sets of HRW wheats were ground, sampled, and sifted independently, so that models would approximate actual milling conditions when ground wheats predicted would somewhat differ from those used in the calibration set. The size fractions corresponding to the sieve openings of the sifter (1041, 375, 240, 136, and <136 μm) were used as NIR reflectance-reference values. These are the sieve sizes used in flour mills for determining the granulation of first-break ground wheat. Cumulative %mass of >1041, >375, >240, and >136 μm is the customary form used by millers in granulation plots and, thus, was used as reference unit.

Models were developed based on partial least squares (PLS) regression (PLS-IQ, Galactic Industries Corp., Salem, NH). The spectral pretreatments evaluated were unit area normalization, baseline correction, first and second derivatives, and their combinations. Mean centering of spectra was common to all models and was done to scale the data to enhance the mathematics of spectral decomposition and correlation. Model performance was based on standard error of cross-validation (SECV), standard error of performance (SEP), coefficient of determination (R^2), number of PLS factors, and coefficient of variability (CV) defined as SEP × 100 divided by mean of reference values (Williams 1987).

RESULTS AND DISCUSSION

Effects of Wheat Cultivar and Roll Gap on Particle Size Distribution and Absorbance Spectra

The particle size distribution of ground HRW wheats was significantly influenced by cultivar and roll gap for all size ranges ($P < 0.05$) (Table II). Most of the wheat particles lay within the size ranges > 1,041 μm (74.0–77.36 %mass) and 375–1,041 μm (15.54–19.37%). Because wheats were from a single class, the hardness range is narrow (Pasikatan 2000). Because kernel hardness is related to wheat fracturing properties and thus particle size distribution (Watson et al 1977; Osborne and Fearn 1986), the particle size distribution of the HRW wheat set is narrower than that of the six wheat class set reported by Pasikatan et al (2001). It follows that the vertical shift or offset of the spectra from the coarser grind to the finer grind was narrower for the HRW wheats than those of the six wheat classes reported. For a specific roll gap but different HRW wheat cultivars, the spectra were nearly overlapping at the nonabsorbing or weakly absorbing bands (Fig. 1). The spectra for each HRW wheat cultivar became distinct from 1,450-1,700 nm because of the varying starch (1,450, 1,528, 1,540, 1,580 nm) and protein bands (1,510 nm). The water absorption bands of 760, 970, 1,190, and 1,450 nm (Curcio and Petty 1951) were the less likely cause of spectral variations because the HRW wheats were uniformly tempered to 16% (wet basis) before grinding. It is also clear that the absorptions at 760, 970, and 1,190 nm were not as distinct as those for the longer wavelengths (Fig. 1). Smaller roll gaps produced finer grinds than bigger roll gaps, and finer grinds had lower absorbance (higher reflectance) than coarser grinds (Fig. 2). The major effect of varying roll gaps for a given HRW wheat cultivar was vertical shifting or offset of the absorbance spectra because of particle size variations. This offset is not constant across

TABLE I
Physical Properties[a] of Hard Red Winter (HRW) Wheat Cultivars (untempered)

HRW Set	Cultivars	Location/Crop Year	BD	MC	TKW	MKS	TD
1	Abilene	KS, 1996	75.2	15.0	35.1	3.1	1.41
2	Arapahoe	NE, 1999	77.7	12.0	30.5	2.8	1.41
3	Rampart	MT, 1999	79.3	9.8	29.8	2.6	1.46
4	Neeley	MT, 1999	80.3	10.2	35.5	2.9	1.42
5	2137	NE, 1999	82.2	10.4	35.3	2.9	1.47
6	2137	OK, 1999	81.1	9.2	30.4	2.7	1.47
7	Tam 107	KS, 1999	81.5	9.7	37.1	3.0	1.48

[a] Bulk density (BD, kg/hL) or test weight; moisture content (MC, % wet basis) oven method; thousand kernel weight (TKW, g); mean kernel size (MKS, mm); true density (TD, g/cm^3).

TABLE II
Particle Size Distribution (% mass) of First-Break Ground Wheat from Grinding Seven HRW Wheat Cultivars[a]

	Roll Gaps				
HRW Set[b]	>1,041 μm	375–1,041 μm	240–375 μm	136–240 μm	<136 μm
Calibration 1	75.15 ± 12.84b	17.74 ± 9.43d	4.23 ± 2.31b	1.83 ± 0.99b	1.05 ± 0.69b
2	73.47 ± 12.39c	16.35 ± 7.48d	5.40 ± 2.85a	2.49 ± 1.42a	2.28 ± 0.72a
3	74.34 ± 14.23c	19.14 ± 10.73a	4.09 ± 2.35b	1.46 ± 0.61b	0.97 ± 0.67b–d
4	76.37 ± 13.81ab	17.74 ± 10.52c	3.68 ± 2.09d	1.74 ± 0.93b	0.46 ± 0.29d
5	74.27 ± 15.15c	18.71 ± 11.19ab	4.13 ± 2.58bc	1.99 ± 1.29b	0.90 ± 0.13b–d
6	73.88 ± 14.27c	18.76 ± 10.70b	4.37 ± 2.23bc	1.83 ± 0.85b	1.15 ± 0.56bc
7	76.13 ± 15.62a	17.77 ± 11.85bc	3.73 ± 2.47cd	1.73 ± 1.08b	0.64 ± 0.25cd
All	75.15 ± 12.84	17.74 ± 9.43	4.23 ± 2.31	1.83 ± 0.99	1.05 ± 0.69
Validation 1	77.36 ± 11.77b	15.54 ± 7.72d	4.08 ± 2.69b	1.26 ± 0.84b	1.76 ± 0.60b
2	74.00 ± 12.84c	15.93 ± 7.87d	5.10 ± 2.98a	2.08 ± 0.70a	2.89 ± 1.35a
3	74.02 ± 14.80c	19.37 ± 11.03a	4.00 ± 2.49b	1.36 ± 0.74b	1.25 ± 0.58b–d
4	76.73 ± 13.39ab	17.36 ± 10.44c	3.31 ± 1.92d	1.45 ± 0.61b	1.14 ± 0.52d
5	75.20 ± 16.18c	18.28 ± 12.03ab	3.71 ± 2.51bc	1.51 ± 1.11b	1.31 ± 0.57b–d
6	75.58 ± 13.24c	18.00 ± 10.11b	3.46 ± 1.95bc	1.44 ± 0.78b	1.53 ± 0.42bc
7	76.01 ± 14.90a	18.13 ± 11.29bc	3.45 ± 2.28cd	1.20 ± 0.73b	1.21 ± 0.63cd
All	75.56 ± 12.71	17.52 ± 9.33	3.87 ± 2.28	1.47 ± 0.78	1.59 ± 0.88

[a] Mean (% mass) ± standard deviation for each size range; $n = 5$ for each cultivar. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).
[b] Cultivars as in Table I.

the spectrum because the scattering property of small particles also varies as a function of wavelength (Kortüm 1969; Osborne and Fearn 1986; Burger et al 1997). Spectral offset has been attributed to pathlength variations due to varying particle sizes and sample porosity (Mark 1991; O'Neil et al 1998, 1999). The pathlength of light for diffuse reflectance spectroscopy is dependent on the scattering coefficient at a particular wavelength, absorption coefficient, particle density, particle size, and refractive index of the material (Birth and Hecht 1987; Switalski et al 1998).

NIR Reflectance-Granulation Models

The effect of wavelength range on the granulation model predictive ability was evaluated using the biggest size fraction, >1,041 μm (Table III). This size fraction is the most important because it is used to determine the break release (%mass of materials that pass through a 1,041 μm sieve) for first-break. The visible range (500–700 nm) and the very NIR region (700–1,100 nm) contains little particle size information as shown in higher SECV values and lesser R^2 values. The visible range contains color or pigmentation information, which hardly varies for HRW wheats. Much of the particle size information lay on the NIR region (1,100–1,700 nm) or the entire visible-NIR region (500–1,700 nm) (Table III). One possible explanation for this is particle size effects are expressed in the entire spectrum (Osborne and Fearn 1986; Devaux et al 1995), thus, models that use wider wavelength regions have better predictive ability. Another possible explanation is the breakage properties of wheats is influenced by endosperm starch-protein matrix (Kent and Evers 1969), and since the absorbing bands for starch and protein are in the NIR region, the granulation models perform better when these regions are included. Similar results were also reported by Pasikatan et al (2001) in six wheat class granulation models. Based on these results, three wavelength regions 800–1,600 nm, 700–1,500 nm, and 600–1,700 nm were identified as giving better results than other wavelength ranges, and thus were further evaluated. Table III also shows that unit area normalization, a pretreatment that corrects for pathlengths variations (Galactic) yielded better models than those that did not. Varying particle sizes, particularly coarse particles, and sample porosity varies the pathlength of light (Williams 1987; O'Neil et al 1998, 1999). The use of nonabsorbing or least absorbing wavelengths (those not affected by compositional changes) in the spectrum for particle size models has been proposed by Osborne et al (1981). However, for this data set, the region of least absorption (≈870–1100 nm) has less variation due to particle size effects compared to the 1200–1700 nm region (Figs. 1, 2 and Table III). Thus, PLS did not yield as good models.

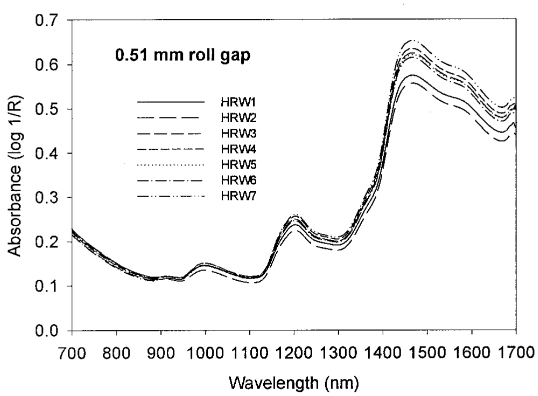

Fig. 1. Absorbance spectra of seven hard red winter (HRW) wheat cultivars ground at a roll gap of 0.51 mm.

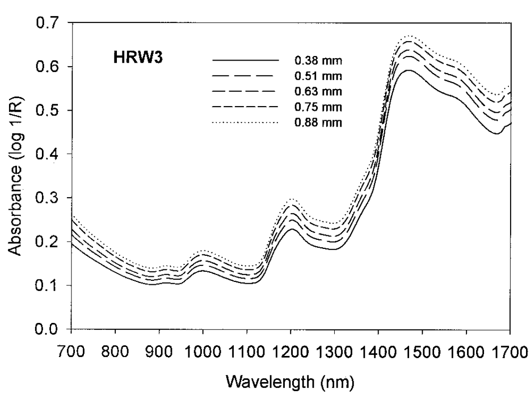

Fig. 2. Absorbance spectra of hard red winter (HRW3) wheat cultivar Rampart ground at various roll gaps (0.38–0.88 mm).

TABLE III
Comparison of Partial Least Squares Calibration Models for >1,041 μm Size Range

Models[a]/Wavelength Range (nm)	*F*	R^2	SECV
Log (1/R)			
500–700	1	0.89	4.18
700–1,100	1	0.89	4.26
1,100–1,700	2	0.87	4.52
500–1,700	2	0.90	3.96
Normalization 1st derivative			
500–700	5	0.75	6.35
700–1,100	12	0.73	6.93
1,100–1,700	2	0.93	3.23
500–1,700	2	0.92	3.64
Normalization 2nd derivative			
500–700	6	0.72	6.81
700–1,100	11	0.83	5.27
1,100–1,700	2	0.93	3.25
500–1,700	2	0.93	3.40

[a] Only representative models are shown. SECV = standard error of cross-validation.

TABLE IV
Partial Least Squares Statistics for Selected Calibration Models for NIR Reflectance-Granulation (700–1,500 nm)

Size Fraction /Pretreatment[a]	*F*[b]	R^2	SECV[c]
>1,041 μm			
Log(1/R)	2	0.89	4.27
Normalization	2	0.91	3.79
and baseline correction	2	0.91	3.77
and 1st derivative	2	0.93	3.35
and 2nd derivative	2	0.93	3.25
>375 μm			
Log(1/R)	2	0.83	1.50
Normalization	2	0.88	1.26
and baseline correction	2	0.85	1.43
and 1st derivative	2	0.88	1.29
and 2nd derivative	2	0.85	1.41
>240 μm			
Log(1/R)	8	0.78	0.72
Normalization	2	0.84	0.60
and baseline correction	2	0.83	0.62
and 1st derivative	2	0.84	0.59
and 2nd derivative	2	0.80	0.67
>136 μm			
Log(1/R)	2	0.61	0.43
Normalization	2	0.70	0.38
and baseline correction	2	0.70	0.37
and 1st derivative	2	0.73	0.36
and 2nd derivative	3	0.65	0.41

[a] Only representative models are shown.
[b] PLS factors.
[c] Standard error of cross-validation.

The better HRW wheat granulation models had fewer PLS factors (Tables III and IV) than the six wheat class models (2–3 vs. 3–9), which indicated robustness. This number of factors was addressed in a study by Devaux et al (1995) using various proportions of binary mixtures of ground wheat and rapeseed meal. They proposed that the first principal component (or factor) described the scattering effect, the second factor described the proportions of fractions at the sample surface, and the third factor provided certain information about the particles beyond the surface. Their results suggested that PLS factors beyond three are describing other effects, including noise in the system. Compared to the sieved fraction models involving six wheat classes (Pasikatan et al 2001), the HRW wheat models required fewer PLS factors for two reasons: 1) a narrower hardness range and 2) better sieving characteristics. The narrower hardness range meant lesser variability in the HRW wheat grinding or fracture response (Pasikatan 2000). Hard wheat flours also freely pass the sieve mesh, whereas soft wheat particles tend to adhere to the sieve mesh (Hareland 1994). The PLS factors beyond three in the six wheat class models, therefore, could have described the effects of wheat class or sieving behavior of soft wheats in the set.

Baseline correction and derivatives could correct for large baseline variations due to particle size effects (Norris and Williams 1984). It was expected, therefore, that only the log (1/R) models would predict particle size well. However, results showed that unit area normalization, alone or combined with either derivatives or baseline correction, improved the log (1/R) models (Table IV). Because unit area normalization corrects for pathlength variations (Galactic), it could be deduced from the results that pathlength variations caused by the coarseness of first-break ground wheat were the main source of spectral variations in this data set. Unit area normalization improved both SECV and R^2 of the log (1/R) models (Table IV). Relative to the log (1/R) models, normalization-based pretreatments reduced the SECV by ≈17% for the >1041 µm range.

Kubelka-Munk (KM) unit was also evaluated because Kubelka-Munk (1931) theory is the most widely accepted theory for light absorption and scattering in a layer of packed ground materials. KM models were expected to perform better than the log (1/R) models. However, contrary results were obtained (not shown). This could possibly be explained by 1) the coarseness of size range, where KM theory might not completely apply, or as Olinger and Griffiths

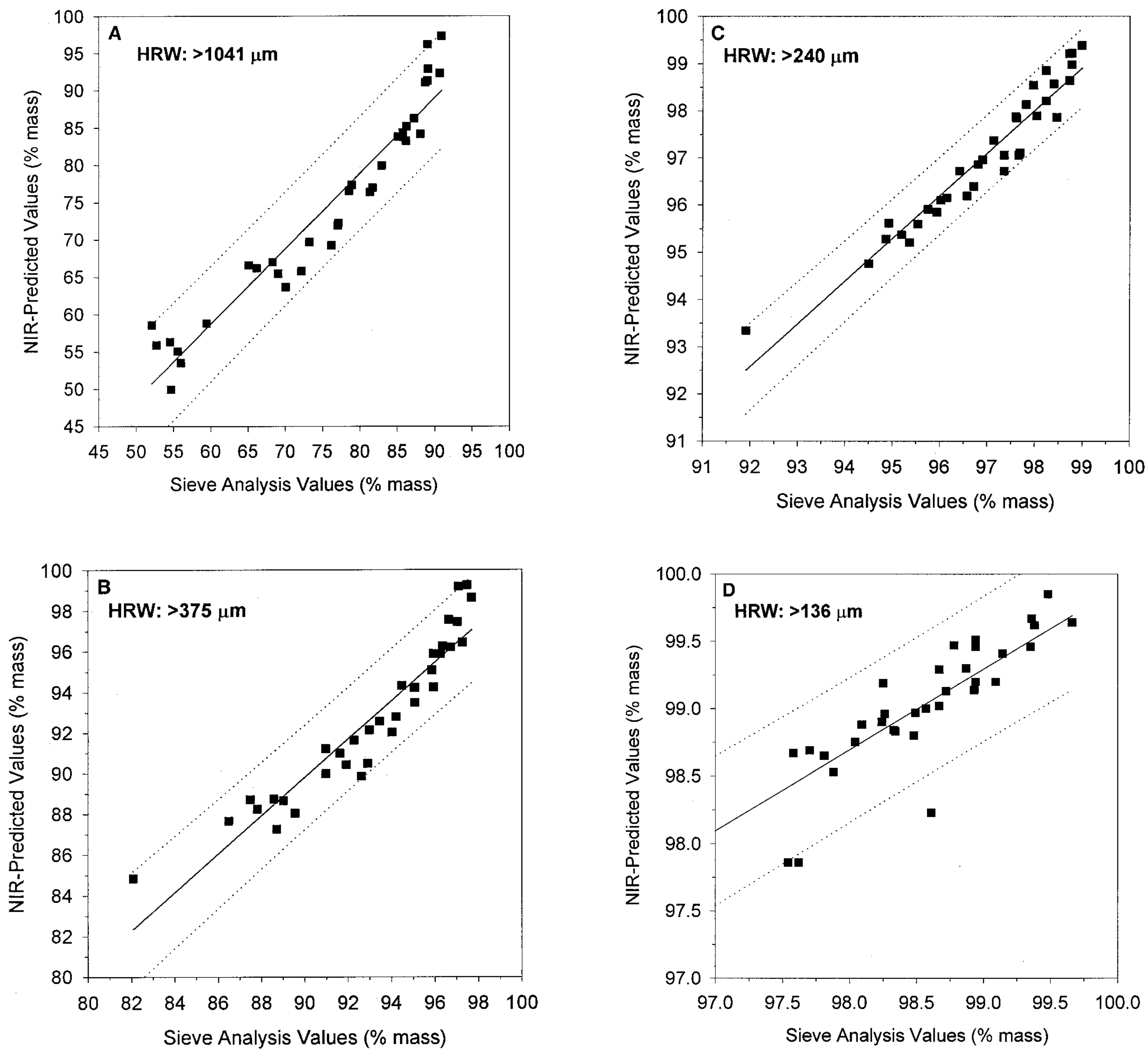

Fig. 3. Relationship between NIR-predicted and sieve analysis values of first-break ground HRW wheat for size fractions **A,** >1,041 µm; **B,** >375 µm; **C,** >240 µm; **D,** >136 µm. Outer lines are the 95% prediction intervals; middle line shows best fit.

(1988) proposed 2) when the matrix surrounding the analyte absorbs radiation at the same wavelength as the analytical band, deviations from the linearity of the KM function occurs, or 3) the wavelength range used did not include the 1,705–2,500 nm region, where distinct spectral variations due to particle size occur (Pasikatan 2000).

The >1041 µm range had the highest SECV because of larger standard deviation (compare Table IV to Table II). SECV decreased as the size fraction included more of the finer particles, which have lesser standard deviation. The R^2 decreased as size fraction included more of the finer particles. Being lesser in mass, the finer fractions were more prone to nonhomogenous dispersion when ground wheat was mixed and presented to the NIR spectrometer. Thus, the probability of scattered light encountering such fractions, followed by absorption and further scattering, were lower compared with the coarser and dominant size fractions. Also, decreasing R^2 with finer particles could have been caused by cumulative error in the reference values (sieving) as the mass of particles collected for each top sieve were summed for the next finer sieve.

Prediction performances of the better HRW granulation models (based on 700–1,500 and 800–1,600 nm) are shown in Table V. Models based on wavelength range 700–1,500 nm, which performed well in calibration (Table IV), did not perform as well in prediction. One spectrum was not predicted compared with the model based on 800–1,600 nm range. PLS reported that the unpredicted spectrum did not belong to the group based on Mahalanobis distance >3.0. Although wheats for the calibration and validation set were sampled from the same wheat cultivars, marked variations between the granulation of ground wheats used in the calibration model and those to be predicted could be expected because the two sets were ground, sampled, and sieved independently (Table II). Tempering, roll gap setting, and sampling of ground wheat were potential sources of variation. Furthermore, the two sets were presented to the NIR spectrometer independently. Although procedures were developed to minimize segregation, it could still occur because of the coarseness of grind and because the granulation of ground wheat sample was predicted based only on spectra from the NIR-penetrated layer, not the entire layer. The best model (normalization and first derivative, 800–1,600 nm) predicted all validation spectra with SEP values of 3.80, 1.29, 0.43, and 0.68 for the >1041, >375, >240, and >136 µm size fractions, respectively. The relationships of the predicted and measured values for the best HRW granulation model are shown in Fig. 3. Table VI shows the summary of the means and standard deviations for the reference method and NIR predicted values. For the same cumulative fraction, the best NIR reflectance-granulation model from HRW wheat spectra had generally better sensitivity (slope ≈1.0) and lower CV than the best six wheat class model reported by Pasikatan et al (2001).

Williams (1991) related wheat kernel texture (degree of hardness or softness) as expressed by particle size index (PSI) to NIR transmittance at 850–1,050 nm. PSI was defined as the %mass of ground wheat that passed through a 200-mesh (75 µm) sieve multiplied by 10. Williams (1991) predicted a single size fraction (<75 µm) from a much finer grind (wheat by Udy Cyclone grinder) than this study. SEP values of 3.37–3.47 and correlation coefficients of 0.90–0.92 were obtained. The average RPD, standard deviation of reference data divided by the SEP, was 3.43 for calibrations involving large samples, and 3.80 for the small sample cell (Williams 1991). Therefore, the RPD of 3.34 for the coarsest size fraction (>1,041 µm) and 3.63 for the >240 µm compared favorably with results of Williams (1991) (Table VI). The other size fractions (>375 and >136 µm) had RPD values lower than those reported by Williams (1991). Another method of evaluating calibrations proposed by Starr et al (1981) was RER (range of reference divided by the SEP). Williams (1991) large sample calibrations yielded an average RER of 11.7. Except for the >136 µm fraction, the RER values obtained for the other size fractions were comparable to values of Williams (1991) (Table VI).

The size fraction <136 µm was the least and the most poorly predicted. With the narrowest range, it was the most sensitive to the effect of a few poorly predicted points, and being the bottom fraction, its reference values were subject to the most cumulative sieving error. In flour milling, the <136 µm size range is called first-break flour. First-break is not intended to be a significant source of flour. Its purpose is to separate as much bran as possible from the endosperm. First-break flour does not go to another process like the other size fractions, hence its accurate prediction is not critical to the flour milling process. The prediction of the largest stream, >1,041 µm, is the most critical to the control of the milling process because break release is calculated from this and from break release the roll gap setting. The technique estimated this size fraction satisfactorily. The models also estimated well the >375 and >240 µm size range; these would be useful as is for both

TABLE V
Prediction Statistics for Selected Models for NIR Reflectance-Granulation

Size Fraction/Pretreatment	Region (nm)	R^2	SEP[a]	n[b]
>1041 µm				
Normalization	700–1,500	0.92	3.87	(35)1
and 1st derivative	800–1,600	0.92	3.80	(35)0
Normalization	700–1,500	0.92	3.89	(35)0
and 2nd derivative	800–1,600	0.92	3.92	(35)0
>375 µm				
Normalization	700–1,500	0.89	1.28	(35)1
and 1st derivative	800–1,600	0.89	1.29	(35)0
Normalization	700–1500	0.86	1.49	(35)0
and 2nd derivative	800–1600	0.85	1.54	(35)0
>240 µm				
Normalization	700–1,500	0.93	0.42	(35)1
and 1st derivative	800–1,600	0.93	0.43	(35)0
Normalization	700–1,500	0.92	0.45	(35)0
and 2nd derivative	800–1,600	0.91	0.47	(35)0
>136 µm				
Normalization	700–1,500	0.81	0.67	(35)1
and 1st derivative	800–1,600	0.80	0.68	(35)0
Normalization	700–1,500	0.80	0.69	(35)0
and 2nd derivative	800–1,600	0.73	0.74	(35)0

[a] Standard error of performance.
[b] Numbers in parentheses are total spectra of validation set; numbers outside parentheses are spectra not predicted.

TABLE VI
Validation Statistics for Best Models[a] for Predicting Size Fractions of First-Break Ground HRW Wheat

Size Fraction	Sieve Analysis (Reference Method)[b]	NIR Predicted Values[b]	Slope[c]	CV[d]	RPD[e]	RER[f]
>1,041 µm	75.56 ± 12.71	74.42 ± 13.36	1.01	5.03	3.34	10.22
>375 µm	93.07 ± 3.77	92.70 ± 3.76	0.94	1.39	2.92	12.10
>240 µm	96.94 ± 1.56	97.05 ± 1.46	0.90	0.44	3.63	16.47
>136 µm	98.41 ± 0.88	98.94 ± 0.59	0.60	0.69	1.29	6.58

[a] Normalization and first derivative model (800–1,600 nm).
[b] Mean %mass ± standard deviation for samples (n = 35).
[c] Based on linear regression.
[d] Coefficient of variability (standard error of performance [SEP] × 100/ mean of reference values).
[e] Standard deviation of reference values divided by SEP.
[f] Range of reference data divided by SEP.

monitoring and control of the processes after first-break grinding. However, after the separation of the >1041 μm size range, that is after first-break sieving, the NIR estimates of these finer size fractions (as well as the break products) would improve further because of narrower size range and more chemical homogeneity. The measurement could be done before these particles go to the size reduction roller mills. The NIR-size estimates could then be used to adjust roll gaps for the size reduction rolls.

In summary, offline calibration validated the diode array NIR reflectance spectrometer as a potential online granulation sensor for HRW first-break ground wheat. The NIR reflectance-granulation models for HRW wheats performed better than the previously reported six wheat class models. The reasons for this improved performance were near uniformity in absorption properties of HRW wheats, such that variations came mostly from particle size effects, and their better sieving properties. The best model, unit area normalization-first derivative, predicted all of the validation spectra with SEP of 3.80, 1.29, 0.43, and 0.68 for the >1041, >375, >240, and >136 μm size fractions, respectively. The performance of NIR granulation models for other wheat classes, or blended wheats should be further studied. The NIR reflectance spectrometer is ready for online evaluation as a granulation sensor for roll gap automation of roller mills.

LITERATURE CITED

Bickel, A. 1989. NIR analyzers earn their place in process control. Instrum. Control Syst. 62:45-48.

Birth, G. S., and Hecht, H. G. 1987. The physics of near-infrared reflectance. Pages 1-15 in: Near Infra Red Technology in the Agricultural and Food Industries. P. Williams and K. Norris, eds. Am. Assoc. Cereal Chem.: St. Paul, MN.

Bull, C. R. 1990. A model of reflectance of near-infrared radiation. J. Modern Optics 37:1955-1964.

Burger, T., Kuhn, J., Caps, R., and Fricke, J. 1997. Quantitative determination of the scattering and absorption coefficient from diffuse reflectance and transmittance measurements: Application to pharmaceutical powders. Appl. Spectrosc. 51:309-317.

Curcio, J. A., and Petty, C. C. 1951. The near-infrared absorption spectrum of liquid water. J. Optical Soc. Am. 41:302-304.

Devaux, M. F., Nathier-Dufour, N., Robert, P., and Bertrand D. 1995. Effects of particle size on the near-infrared reflectance spectra of wheat and rape-seed mixtures. Appl. Spectrosc. 49:84-91.

Duyckaerts, G. 1955. Contribution a l'analyse quantitative par les spectres d'absorption infrarogue des poudres. Spectrochim. Acta 7:25-31.

Fang, Q., Bolloni, I., Haque E., and Spillman, C. K. 1997. Comparison of energy efficiency between a roller mill and a hammer mill. Appl. Eng. Agric. 13:631-635.

Felder, B. 1964. Uber die teilchengrossenabhängigkeit der lichtabsorption in heterogenensystemen. I. Teoretishce betrachtungen. Helv. Chim. Acta 47:488-497.

Hareland, G. A. 1994. Evaluation of flour particle size distribution by laser diffraction, sieve analysis and near-infrared reflectance spectroscopy. J. Cereal Sci. 21:183-190.

Kaye, B. H., and Naylor, A. G. 1972. Powder sampling procedures. Pages 47-66 in: Proc. Seminar on Particle Technology. D. Venkateswarlu and A. Prabhakara Rao, eds. Indian Institute of Technology: Madras, India.

Kent, N. L., and Evers, A. D. 1969. Fine grinding and air classification of subaleurone endosperm of high protein content wheat. Cereal Sci. Today 14:142-149.

Kortüm, G. 1969. Reflectance Spectroscopy: Principles, Methods, Applications. Springer-Verlag: New York.

Kubelka, P., and Munk, F. 1931. Ein beitrag zur optik der farbanstriche. Z. Tech. Physik 12:593-601.

Mark, H. 1991. Principles and Practice of Spectroscopic Calibrations. Wiley: New York.

Norris, K. H. and Williams, P. C. 1984. Optimization of mathematical treatments of raw near infrared signal in the measurement of protein in hard red spring wheat. I. Influence of particle size. Cereal Chem. 61:158-165.

Olinger, J. M., and Griffiths, P. R. 1988. Quantitative effects of an absorbing matrix on near-infrared diffuse relectance spectra. Anal. Chem. 60:2427-2435.

O'Neil A. J., Jee, R. D., and Moffatt, A. C. 1998. The application of multiple linear regression to the measurement of the median particle size of drugs and pharmaceutical excipients by near-infrared spectroscopy. Analyst 123:2297-2302.

O'Neil A. J., Jee, R. D., and Moffatt, A. C. 1999. Measurement of cumulative particle size distribution of microcrystalline cellulose using near-infrared reflectance spectroscopy. Analyst 124:33-36.

Osborne, B. G., Douglas, S., and Fearn. T. 1981. Assessment of wheat grain texture by near-infrared reflectance measurements on Bühler-milled flour. J. Sci. Food Agric. 32:200-202.

Osborne, B. G., and Fearn, T. 1986. Near Infrared Spectroscopy for Food Analysis. Longman Scientific and Technical: Essex, UK.

Pasikatan, M. C. 2000. Development of first-break grinding models and a near-infrared reflectance technique for size estimation of first-break grinding fractions. PhD diss. Kansas State University: Manhattan, KS.

Pasikatan, M. C., Haque E., Steele, J. L., Spillman, C. K., and Milliken, G. A. 2001. Evaluation of a near-infrared reflectance spectrometer as a granulation sensor for first-break ground wheat: Studies with six wheat classes. Cereal Chem. 78:730-736.

Posner, E. S., and Hibbs, A. N. 1997. Wheat Flour Milling. Am. Assoc. Cereal Chem.: St. Paul, MN.

Robertson, A., Hall, M., and Scotter, C. 1989. Near infrared spectroscopy—Its use in rapid QC analysis and its potential in process control. Food Sci. Technol. Today 3:102-110.

Switalski, S. C., Colin, T., Redden, N., Stahlecker, E., and Parthasarathy, V. 1998. Near infrared diffuse reflectance fiber optic spectroscopy for process monitoring applications. Pages 158-166 in: Proc. SPIE Conference on Process Monitoring Applications of Fiber Optic Sensors. SPIE Vol. 3538. SPIE Publishing: Bellingham, WA.

Starr, C., Morgan, A. G., and Smith, D. B. 1981. An evaluation of near-infra-red reflectance analysis in some plant-breeding programmes. J. Agric. Sci. 97:107.

Watson, C. A., Shuey, W. C., Banasik, O. J., and Dick, J. W. 1977. Effect of wheat class on near infrared reflectance. Cereal Chem. 54:1264-1269.

Wendlandt, W. W., and Hecht, H. G. 1966. Reflectance Spectroscopy. Interscience Publishers: New York.

Williams, P. C. 1991. Prediction of wheat kernel texture in whole grains by near infrared transmittance. Cereal Chem. 68:112-114.

Williams, P. C. 1987. Variables affecting near infrared reflectance spectroscopic analysis. Pages 143-167 in: Near Infra Red Technology in the Agricultural and Food Industries. P. Williams and K. Norris, eds. Am. Assoc. Cereal Chem.: St. Paul, MN.

[Received November 30, 2000. Accepted September 13, 2001.]