Evaluation of a Near-Infrared Reflectance Spectrometer as a Granulation Sensor for First-Break Ground Wheat: Studies with Six Wheat Classes[1]

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ABSTRACT

In flour milling, a granulation sensor for ground wheat is needed for automatic control of a roller mill's roll gap. A near-infrared (NIR) reflectance spectrometer was evaluated as a potential granulation sensor of first-break ground wheat using offline methods. Sixty wheat samples, ground independently, representing six classes and five roller mill gaps, were each used for calibration and validation sets. Partial least squares regression was used to develop the models with cumulative mass of size fraction as the reference value. Combinations of four data pretreatments (log (1/R), baseline correction, unit area normalization, and derivatives) and three wavelength regions (700–1,500, 800–1,600, and 600–1,700 nm) were evaluated. Unit area normalization combined with baseline correction or second derivative yielded models that predicted well each size fraction of first-break ground wheat. Standard errors of performance of 4.07, 1.75, 1.03, and 1.40 and r^2 of 0.93, 0.90, 0.88, and 0.38 for the >1,041-, >375-, >240-, and >136-µm size ranges, respectively, were obtained for the best model. Results indicate that the granulation sensing technique based on NIR reflectance is ready for online evaluation.

The full automation of flour milling has long been delayed by the inability to translate human judgment in the milling system into corresponding analog or digital expressions (Posner and Hibbs 1997). The latest roller mill designs, for example, are equipped with programmable logic controllers for feed rate control and roll protection, but the essential feature for roll gap control and automation—a sensor for the ground wheat size distribution—is still missing. In flour milling, the term granulation is used to refer to size distribution of ground wheat products. Granulation is the plot of cumulative percent mass of sieved fractions greater than the sieve size against the corresponding sieve sizes. Granulation is the millers' basis for ensuring the optimum distribution of the ground products and the overall efficiency of the milling process. For first-break, the first roller mill operation in the milling process, granulation is obtained by sieving ground wheat on a sieve stack of sizes 20W (opening of 1,041 µm), 50GG (375 µm), 70GG (240 µm), 10XX (136 µm), and a pan (<136 µm). One of the points in the granulation curve, the % mass >1,041 µm, is used to compute the break release (% mass of ground wheat that passes through the 1,041-µm sieve). Break release is related exponentially to the roll gap, or clearance between rolls (Kuprits 1965) and, hence, millers use the target break release to set manually the approximate roll gap (Niernberger 1966). A system that would automatically compute break release is, until now, still being anticipated (McGee 1982; Sosland 1986). In some flour mills, ground wheat is sampled, then goes to a device that sifts and weighs the sieve fractions and sends a signal to a controller; this takes at least 20 sec per sample. Ideally, the roll gap should be set in real time by a signal from an online granulation sensor to optimize flour extraction from each break and reduction stage. Flour milling is a low-margin, high-volume process; even a 1% improvement in flour extraction or a 0.1% increase in flour protein in a typical mill would each result in an annual profit increase of ≈$100,000 (Clegg 1988; Smart 1990).

First-break is the logical starting point for this study because 1) the performance of the succeeding break and reduction stages and the flour mill in general are dependent on the performance of first-break roller mill, and 2) the study is intended to produce an outcome (the ground wheat properties) from the known or measurable input properties of wheat and roller mill parameters; thus, extending the granulation-sensing system to the next stage of milling is highly feasible. In contrast, the prevailing approach in flour milling automation is to model the milling characteristics from the input wheat properties based on typical performances of equipment in the milling system (Flores 1989; Kim 1996). Optimization on each stage of the milling process was assumed. Furthermore, automation in milling is heavily focused on the end product (flour) (Sosland 1987), a stage when control measures will have lesser benefit, compared with controlling intermediate products from preceding stages.

Near-infrared (NIR) reflectance spectroscopy is a potential technique for granulation sensing. The cross-sensitivity of NIR reflectance to the particle size of ground or granular materials has long been observed and reported (Wendlandt and Hecht 1966; Kortüm 1969; Wetzel 1983; Norris and Williams 1984). NIR reflectance spectrometry has been used successfully for determining particle size of various ground products, such as sorbitol, salt, and broken glass (Ilari et al 1988), pea flour (Chapelle et al 1989), wheat flour (Hareland 1994), and pharmaceutical powders (Ciurczak et al 1986; Blanco et al 1992; Frake et al 1998; O'Neil et al 1998; Rantanen and Yliruusi 1998). However, those studies used NIR reflectance for finely ground products with size of ≤400 µm. NIR reflectance still needs to be evaluated for a coarsely ground material such as first-break ground wheat (size range: <1 to 3,360 µm).

The objectives of this study were to 1) evaluate, using offline methods, an NIR reflectance spectrometer for granulation sensing of first-break ground wheat and 2) develop and validate NIR reflectance-granulation models. This study focused on six wheat classes to explore the possibility of obtaining a universal calibration for sizes across wheat classes (because size estimates should be independent of wheat class) and as a reference for subsequent studies using specific wheat classes.

MATERIALS AND METHODS

Wheat Samples and Physical Property Tests

Six wheat classes were used to develop NIR reflectance-granulation models for first-break: durum, hard red spring (HRS), hard red winter (HRW), hard white (HWH), soft red winter (SRW),

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and soft white (SWH). Two cultivars were obtained for each wheat class. Wheat was cleaned using three passes in a dockage tester (Carter Day Co., Minneapolis, MN). Clean wheat was mixed and then divided using a precision divider (Gamet, Minneapolis, MN) to obtain two sets of representative samples, one for each replicate. From each replicate set, wheat was sampled using the same divider to obtain smaller representative samples for milling and for measuring physical properties (Table I). Five milling samples (440-g) from each wheat cultivar or 60 milling samples per replicate set (six classes × two cultivars × five milling samples) were prepared.

The test weight of untempered, dockage-free samples was measured using a computer grain scale (model 8800A, Seedburo Co., Chicago, IL). The thousand-kernel weight was determined from clean, whole-kernel samples (40 g) using an electronic seed counter. Mean kernel size was obtained from untempered samples (100 g) using a rotap sifter (model RX-29, W. S. Tyler Corp., Mentor, OH) with a stack of U.S. Sieve Nos. 6, 7, 10, and 12 (openings of 3.36, 2.82, 2, and 1.68 mm, respectively). Sieving time was 10 min. The true density of wheat samples was measured using a multipycnometer (model MVP-1; Quanta Chrome Co., Syosset, NY). The oven moisture content (Approved Method 44-15A; AACC 2000) of wheat was used as the basis for tempering.

Grinding and Sifting

An experimental roller mill equipped with a computerized data acquisition system was used for the milling tests (Fang et al 1997). The settings of the roller mill 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-sec/m feed rate (2.04 kg/min for roll contact width of 25.4 mm). These settings were fixed so that the variations in particle-size distribution of the wheat samples could come only from wheat class and roll gap. Wheat samples were ground using five roll gaps (0.38, 0.51, 0.63, 0.75, and 0.88 mm) that were set with a feeler gage. The most likely roll gap range for first break of 0.51–0.75 mm plus two extreme points were used for three reasons: 1) to account for the case when the typical range is exceeded, 2) to obtain ground wheat with widely varying granulation for testing the limits of NIR reflectance technique, and 3) to enable comparison of these results with a separate study that modeled single-kernel properties of input wheat to size properties of ground wheat (reported in another article). The 440-g milling samples were placed in plastic tempering bottles (9 × 9 × 160 cm; Seedburo Co.) and tempered to 15.5 and 16% moisture content for 10 and 15 hr for soft and hard wheat, respectively. A tempering mixer (Chopin, Seedburo Co.) was used for the first 20 min of tempering time. Tempering was done in sequence, such that each bottle was opened and the tempered wheat was milled within ±0.5 hr of the scheduled time. Before milling, the roller mill was warmed up by allowing it to run for 1 hr and by grinding some excess wheat samples tempered at the same moisture content as the milling samples. The speed controller (model 1336, Allen-Bradley, Milwaukee, WI) could fix the roll speed only at no-load conditions; therefore, speeds were set to compensate for the grinding loads.

TABLE I
Physical Properties of Untempered Wheat Samples

Wheat Class[a]	Cultivars	Location/Crop Year	BD (kg/hL)[b]	MC (%)[c]	TKW (g)[d]	MKS (mm)[e]	TD (g/cm^3)[f]
Durum	Cando	North Dakota, 1996	78.5	14.9	43.9	3.0	1.42
	Monroe	North Dakota, 1996	80.0	14.9	41.3	3.1	1.43
HRS	Butte86	North Dakota, 1996	80.6	12.5	33.2	3.1	1.34
	Marshall	Minnesota, 1996	77.3	14.1	37.3	3.1	1.43
HRW	Abilene	Kansas, 1996	75.2	15.0	35.1	3.1	1.41
	Arapahoe	Nebraska, 1999	77.7	12.0	30.5	2.8	1.41
HWH	Arlin	Colorado, 1996	79.3	15.3	40.5	3.2	1.44
	Anza	California, 1996	76.9	9.4	41.9	3.3	1.46
SRW	Caldwell	Michigan, 1996	75.2	13.4	34.4	3.2	1.38
	Pocahontas	Virginia, 1996	77.5	12.7	40.5	3.4	1.36
SWH	Rod	Idaho, 1999	76.7	11.2	39.2	3.1	1.44
	Tres	Washington, 1993	80.1	11.6	37.1	3.1	1.41

[a] HRS = hard red spring, HRW = hard red winter, HWH = hard white, SRW = soft red winter, SWH = soft white.
[b] Bulk density or test weight.
[c] Moisture content (wet basis), oven method.
[d] Thousand kernel weight.
[e] Mean kernel size.
[f] True density.

TABLE II
Particle Size Distribution (% mass) of First-Break Ground Wheat After Grinding Six Wheat Classes at Five Different Roll Gaps[a]

	Particle Size (% mass) for Each Roll Gap				
Wheat Class[b]	>1,041 µm	375–1,041 µm	240–375 µm	136–240 µm	<136 µm
Calibration set					
Durum	82.01 ± 9.37a	14.49 ± 7.87a	2.21 ± 1.12a	0.96 ± 0.42a	0.33 ± 0.11a
HRS	73.83 ± 13.28b	18.48 ± 9.85b	3.87 ± 1.93b	1.17 ± 0.26b	2.66 ± 1.35b
HRW	75.68 ± 11.75c	15.74 ± 7.35c	4.59 ± 2.73c	1.67 ± 0.85c	2.33 ± 1.15c
HWH	70.63 ± 14.03d	20.93 ± 10.08d	4.34 ± 2.36c	1.55 ± 0.79c	2.56 ± 0.97b
SRW	65.76 ± 12.16e	20.03 ± 6.60e	9.02 ± 2.93d	2.82 ± 0.93d	2.37 ± 2.21c
SWH	64.36 ± 14.27f	23.77 ± 8.72f	7.38 ± 3.64e	2.35 ± 1.02e	2.13 ± 1.45d
Validation set					
Durum	80.13 ± 13.42a	15.80 ± 11.14a	2.55 ± 1.65a	1.01 ± 0.51a	0.50 ± 0.24a
HRS	72.69 ± 14.49b	19.28 ± 10.29b	4.51 ± 2.66b	2.13 ± 1.27b	1.39 ± 0.61b
HRW	75.51 ± 12.01c	16.04 ± 7.81c	4.81 ± 2.67c	2.01 ± 1.21c	1.63 ± 0.85c
HWH	68.74 ± 15.67d	22.43 ± 11.10d	5.13 ± 2.89c	2.21 ± 1.44c	1.49 ± 0.64b
SRW	65.68 ± 11.75e	20.26 ± 6.54e	9.34 ± 2.86d	3.95 ± 1.84d	0.77 ± 0.84c
SWH	64.08 ± 14.15f	24.22 ± 8.98f	8.39 ± 3.47e	2.82 ± 1.72e	0.49 ± 0.12d

[a] Mean ± standard deviation for each size range. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).
[b] HRS = hard red spring, HRW = hard red winter, HWH = hard white, SRW = soft red winter, and SWH = soft white; $n = 10$ for each wheat class.

Wheat was ground according to a randomization sequence. Ground wheat samples were collected and mixed manually 20× using the method described by Kaye and Naylor (1972). The mixing method combined inversion and rotation of a mixing cylinder (12.7 cm diameter × 17 cm length). The precision divider was used to obtain three parts from each 440 g of ground wheat: one of 220 g (for NIR presentation) and two of 110 g each (for sifting). The first 110-g ground wheat samples were sieved to determine the granulation while the second was set aside for a related study.

A laboratory sifter (Great Western Co., Leavenworth, KS) with a stack of 20W (opening of 1,041 μm), 50GG (375 μm), 70GG (240 μm), and 10XX (136 μm) sieves and a pan was used. Sieving time was 2 min. A digital balance (model DI-4KD, Denver Instruments, Arvada, CO) with 0.01-g resolution was used for weighing the sieves and the sieved fractions.

Sample Presentation Method

The method of dropping first-break ground wheat to the NIR spectrometer's sample cell was critical in this study because of the sample's wide size range (<1 to 3,360 μm), more than eight times the size range in the cited NIR reflectance-particle size studies. A study identified a sample presentation method that dropped a representative layer in a repeatable manner (Pasikatan 2000). The method used a Motomco drop cell (from a moisture meter of the same brand), which dropped a premixed, constant mass (≈200 g) from a height of 152.4 mm into a cylinder with three layers of randomizing rods. Details of the randomizing rods are described elsewhere (Pasikatan 2000).

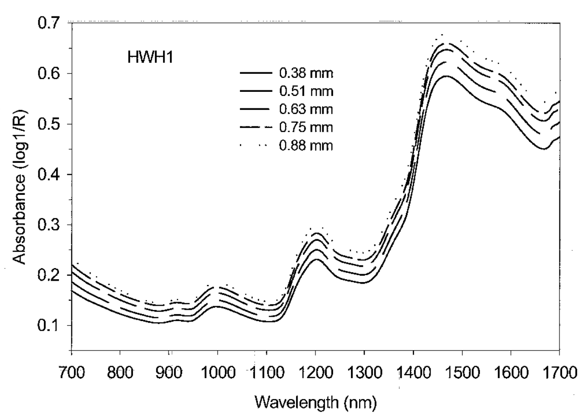

Fig. 1. Absorbance spectra of hard white wheat cultivar Arlin ground at roll gaps of 0.38–0.88 mm. Spectral shape is typical for different wheat classes.

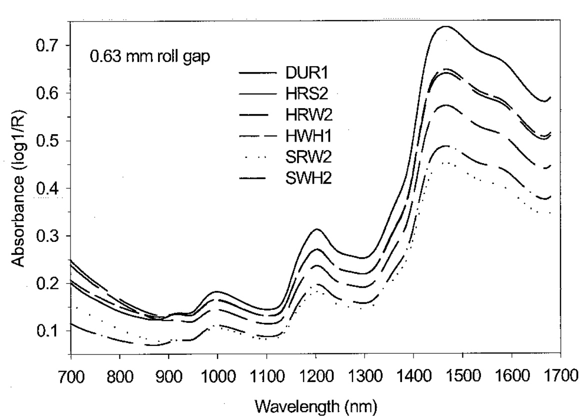

Fig. 2. Absorbance spectra of six wheat class samples ground at a roll gap of 0.63 mm. Absorbance values differ according to roll gaps but spectral profiles were similar.

Spectral Data Collection and Model Development

The NIR reflectance spectrometer evaluated for granulation sensing was a diode array type (Perten Instruments, Springfield, IL). It was used to collect absorbance (log 1/R) spectra of the 220-g ground wheat sample from 400 to 1,700 nm at 5-nm increments. Each ground wheat spectrum represented 30 spectral scans that were collected and averaged. Four spectra were collected for each ground wheat and were averaged to reduce noise. Each ground wheat was mixed in a mixing cylinder described previously and dropped by the sample presentation method into a sample ring (13.3 cm diameter, 2.54 cm thick) above the spectrometer's sample glass window. Presentations of the ground wheat were randomized by wheat class and then by roll gaps for each replicate. The cylinder with randomizing rods was rotated a quarter turn at a time from a reference point before dropping of ground wheat to remove orientation effects. Sieve analysis values were merged with the spectra to form the calibration file which was saved in a hard disk and exported to spectral analysis software (Grams/32, Galactic Industries Corp., Salem, NH) for model development. All spectra were mean-centered before analysis.

One grinding replicate of wheat (60 samples) was used for calibration development; the other replicate set (60 samples) was used for validation. Random assignment of ground wheat samples to calibration and validation set was not done because each represented a wheat class and roll gap combination and should be represented equally in both sets. Another approach, random assignment of replicate 1 and 2 ground wheat samples to calibration and validation sets, while ensuring equal representation of all combinations, was not used, either. This approach would distribute the variations due to grinding, sifting, and NIR presentations to the calibration and validation sets and thus yield less error. However, the predictions would not approximate actual milling conditions when ground wheat samples presented would somewhat vary (because of grinding, sampling, and NIR presentation) from those used in the calibration.

The size fractions corresponding to sieve openings of the laboratory sifter (1,041, 375, 240, 136, and <136 μm) were used as reference values for NIR reflectance. These are the sieve sizes used

TABLE III
Calibration Model Statistics of Selected Partial Least Squares (PLS) Models for Near-Infrared Reflectance-Granulation (800–1,600 nm)[a]

Size Fraction, Pretreatment[b]	F	r^2	SECV[c]
>1,041 μm			
Log (1/R)	11	0.91	3.95
Norm	7	0.94	3.15
Norm and BC	9	0.95	2.97
Norm and 1st deriv	7	0.94	3.16
Norm and 2nd deriv	8	0.95	3.05
>375 μm			
Log (1/R)	12	0.91	1.63
Norm	11	0.95	1.22
Norm and BC	10	0.95	1.18
Norm and 1st deriv	12	0.95	1.20
Norm and 2nd deriv	9	0.95	1.24
>240 μm			
Log (1/R)	10	0.80	1.00
Norm	10	0.87	0.80
Norm and BC	13	0.89	0.73
Norm and 1st deriv	11	0.88	0.76
Norm and 2nd deriv	9	0.86	0.81
>136 μm			
Log (1/R)	13	0.62	0.97
Norm	13	0.70	0.84
Norm and BC	13	0.76	0.75
Norm and 1st deriv	12	0.73	0.81
Norm and 2nd deriv	11	0.66	0.90

[a] To illustrate the effects of spectral pretreatment, only models that used the 800–1,600 nm region are shown.
[b] Normalization, baseline correction, first derivative, and second derivative.
[c] Standard error of cross-validation.

in flour mills for determining granulation of first-break ground wheat. The reference unit used in this study was a cumulative % mass >1,041, >375, >240, and >136 µm because this is the customary form used by millers in granulation plots. Laser diffraction as a reference method has advantages over sieving analysis (Hareland 1994) and has been used as a reference method in most NIR reflectance-particle size studies, but using percent volume measures is not suitable for milling applications where percent mass is used. Moreover, laser diffraction method cannot completely measure the wide size range of first-break ground wheat.

Models were developed based on partial least squares (PLS) regression (in Grams/32 spectral analysis software). This is a multivariate data analytical technique used to solve multivariate analysis problems by a sequence of least squares regression (Bjorsvik and Martens 1992). It extracts data, in this case all NIR information in relevant wavelengths, and compresses these data into a small number of new variables or factors, where each factor is a linear combination of the original variables (Gartwaithe 1994). The spectral pretreatments evaluated were unit area normalization, baseline correction, derivatives, and their combinations. 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).

RESULTS AND DISCUSSION

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

The dominant size range of first-break ground wheat was >1,041 µm (64.1–82.0% by mass) (Table II). Substantial amounts of particles also were in the ranges of 375–1,041 µm (14.5–24.2%) and 240–275 µm (2.2–9.3%). The finer size ranges of 136–240 and <136 µm had negligible amounts. Durum ground wheat contained the highest percent mass of particles in the >1,041-µm range and SWH contained the lowest. The combined effect of wheat class and roll gap significantly influenced the particle size distribution of ground wheat ($P < 0.05$). For a specific wheat class, narrow roll gaps produced finer grinds than wide roll gaps, and finer grinds had lower absorbance (higher reflectance) than coarser grinds (Fig. 1). Therefore, the major effect of roll gap was vertical shifting of the absorbance spectra. The lower absorbance for finer grinds was also observed in previous studies and can be explained by light scattering effect (Wetzel 1983; Norris and Williams 1984; Williams 1987). For a specific roll gap but different wheat classes, both baseline and vertical shifting occurred because of variations in both absorption and scattering properties (Fig. 2). This is an indication that granulation models based on a single wheat class might perform better than a six-wheat class model.

NIR Reflectance-Granulation Models

The wavelength region of 900–1,300 nm was identified by PLS as having the least standard deviation and highest r^2. However, models that included regions wider than 900–1,300 nm performed better in terms of SECV. The wavelength ranges of 800–1,600, 700–1,500, and 600–1,700 nm yielded better models than other wavelength ranges and thus were used for further model development. For ground materials, particle size affects absorbance values of the entire spectrum (Osborne and Fearn 1986; Devaux et al 1995), which explains why wider wavelength regions have better predictive ability for particle size than narrow wavelength regions. Also, particle size effects are more pronounced in the longer wavelength regions of the NIR (Norris and Williams 1984; Chapelle et al 1989). Hence, models using more of these regions performed better than those which used less.

Spectral pretreatments also influenced the performance of the models (Table III). Baseline correction could correct for baseline shifts due to particle size effects but, in this spectral data set, it did not improve the log (1/R) models (data not shown). A probable reason for this was the relatively large standard deviation for baseline-corrected spectra. First and second derivatives also could correct for large baseline variations (Norris and Williams 1984), but these did not improve the log (1/R) models (not shown), an indication that baseline shifting was not a major cause of spectral variation for this spectra set. Unit area normalization improved the log (1/R) models (Table III). SECV was reduced by ≈15–25%, depending on size range, and PLS factors were fewer. Coarse or uneven particles could change path lengths to varying degrees (Williams 1987) and unit area normalization could correct for path length effects and some simple nonlinearities (Grams/32 spectral analysis software); therefore, it could be deduced that the main effect of the dominant coarse size fraction (>1,041 µm) was path length, not baseline variation. Unlike the constant path length of light for absorption spectroscopy, the path length of light for diffuse reflectance spectroscopy is a function of the microscopic structure of the material containing the absorber (Birth and Hecht 1987). Therefore, correcting for path length effects by unit area normalization improved the performance of log (1/R) models more than did baseline correction. However, using baseline correction after path length correction had a better effect than path length correction alone (Table III). Even derivatives produced better models with normalized log (1/R) spectra than with log (1/R) spectra only. Normalization and derivatives produced two wavelength regions: a high-noise, high-r^2 region (500–800 nm) and a low-noise, high-r^2 region (1,200–1,600 nm) (not shown). Using more of the latter region yielded better models.

The coarsest size fraction (>1041 µm) had the highest SECV (Table III). This size fraction consists of bran flakes and fragments whose shapes are irregular and vary widely (Table II). Sieve analysis assumed these sizes to be spherical or near-spherical. First-break ground wheat has a distinctive property (i.e. finer size fractions have lesser amounts and varied less than the coarser fractions) (Table II). This is not so with wheat flour or other ground materials cited in the NIR reflectance-particle size studies, in which the finer size fractions are dominant. Therefore, size estimation of first-break ground wheat would tend to yield larger SECV than reported in those studies. SECV decreased as more of the finer particles are included in the size range (Table III). This is because the finer size fractions have lesser standard error (Table II). In such a case, as the mean particle size of the ground wheat decreased (more fine particles), diffuse reflectance increased and,

TABLE IV
Prediction Statistics for Selected Models of Near-Infrared Reflectance-Granulation

Pretreatment[a]	Region (nm)	r^2	SEP[b]	n[c]
>1,041 µm				
Norm and BC	700–1,500	0.91	4.41	(60) 5
	800–1,600	0.90	4.54	(60) 5
Norm and 2nd deriv	800–1,600	0.91	4.28	(60) 8
	600–1,700	0.93	4.07	(60) 5
>375 µm				
Norm and BC	700–1,500	0.88	2.02	(60) 5
	800–1,600	0.89	1.90	(60) 5
Norm and 2nd deriv	800–1,600	0.91	1.65	(60) 8
	600–1,700	0.90	1.75	(60) 5
>240 µm				
Norm and BC	700–1,500	0.84	1.33	(60) 5
	800–1,600	0.72	1.74	(60) 5
Norm and 2nd deriv	800–1,600	0.90	0.97	(60) 8
	600–1,700	0.88	1.03	(60) 5
>136 µm				
Norm and BC	700–1,500	0.49	2.24	(60) 5
	800–1,600	0.44	2.24	(60) 5
Norm and 2nd deriv	800–1,600	0.41	1.72	(60) 8
	600–1,700	0.38	1.40	(60) 5

[a] Normalization, baseline correction and second derivative.
[b] Standard error of performance.
[c] Numbers in parentheses are the total spectra of validation set; numbers outside parentheses are spectra not predicted.

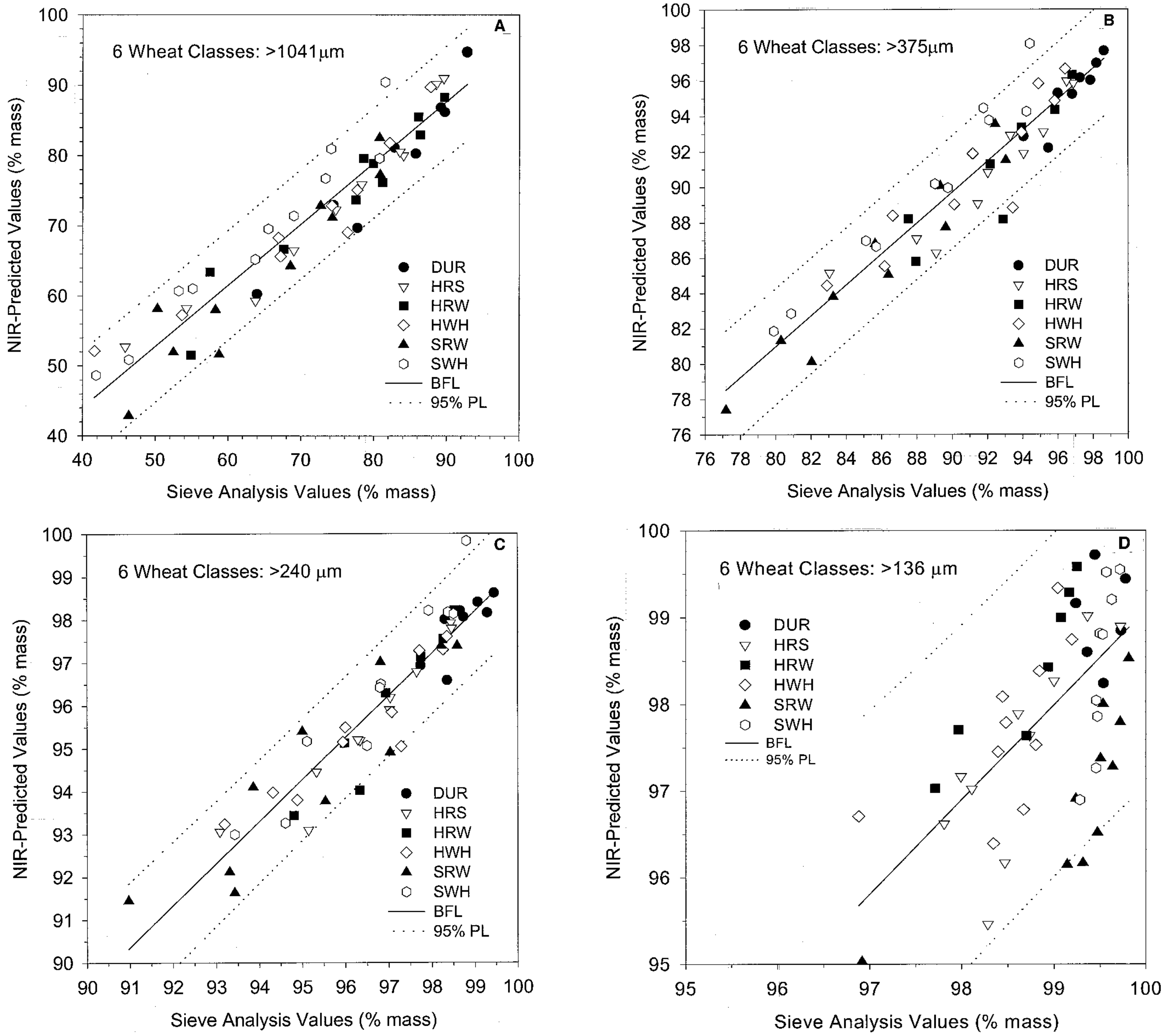

Fig. 3. A, Relationship between near-infrared (NIR) reflectance-predicted and sieve analysis values of first-break ground wheat for >1,041 μm fraction (**A**); >375 μm fraction (**B**); >240-μm fraction (**C**); >136-μm fraction (**D**). Dotted lines = 95% prediction; BFL = best-fit line.

thus, the NIR reflectance-granulation method became more accurate in predicting particle size. However, in the case of first-break ground wheat where the amounts of finer size fractions were much less than the amount of the coarsest fraction (Table II), and where the contribution of each size fraction to the spectrum was weighted by its amount (Pasikatan 2000), reduced size meant less spectral information related to particle size. This resulted in the decrease in r^2 with decreased particle size (Table III). Therefore, for size prediction using NIR reflectance spectroscopy, a substantial amount of a size fraction was a critical requirement. For first-break ground wheat, a substantial amount could be defined as ≥5% by mass (Pasikatan 2000). In contrast, for chemical applications of NIR reflectance spectroscopy, concentrations of ≥1% can be detected (Wetzel 1983). The use of cumulative mass (%) as a reference unit for NIR reflectance provided a larger mean value for a size fraction that included fine size ranges, such that deviations relative to its mean particle size were smaller. It enabled size fractions that included fine particles to satisfy the substantial mass requirement.

Prediction statistics of the better performing models (normalization-baseline correction and normalization-second derivative) are shown in Table IV. For the >1041-μm size fraction, the models predicted 52–55 out of 60 validation spectra with SEP and r^2 of 4.07–4.54 and 0.90–0.93, respectively. PLS reported that the unpredicted spectra did not belong to the group based on a Mahalanobis distance >3.0. The unpredicted spectra could be attributed to variations in particle size distribution between the calibration and validation set due to grinding, sampling, and sample presentation. In milling applications, this would not necessarily be a disadvantage because 1) the unpredicted spectra were usually borderline cases that, when predicted, could increase the SEP; and 2) subsequent measurements could compensate for the unpredicted spectra. Moreover, variations between the particle size distribution of ground wheat used in the calibration model and those to be predicted could be expected in practice. Thus, the errors obtained in this study approximate what could be obtained in milling.

The means and standard deviations for the reference method and NIR reflectance-predicted values based on the normalization-second derivative model are summarized in Table V. Most of the variations in the first-break ground wheat (which caused much of the path length variations) came from the >1041-μm size. The predicted and measured values for the four size fractions are shown in Fig. 3A–D. The close agreement of predicted and measured values shows that the NIR reflectance technique is feasible for estimation of granulation and is ready for online evaluation.

TABLE V
Validation Statistics for Best Model for Predicting Size Fractions of First-Break Ground Wheat

Size Fraction[a]	% Mass[b] Sieve Analysis (Reference Method)	Near-Infrared Reflectance Predicted Values	y-Intercept[c]	Slope[c]	CV[d]
>1,041 μm	71.52 ± 14.27	72.38 ± 12.79	10.74	0.86	5.72
>375 μm	90.83 ± 5.38	90.41 ± 4.95	11.16	0.87	1.93
>240 μm	96.65 ± 1.94	95.89 ± 2.02	1.11	0.98	1.06
>136 μm	99.02 ± 0.70	98.01 ± 1.24	–10.37	1.09	1.41

[a] Normalization-second derivative model (600–1,700 nm).
[b] Mean percent mass ± standard deviation; $n = 55$. Validation set had 60 spectra but only 55 spectra were predicted by partial least squares model.
[c] Based on linear regression equation.
[d] Coefficient of variability ([standard error of performance × 100]/mean of reference values) (Williams 1987).

First-break is the logical starting point for milling automation studies. The NIR reflectance-granulation system could be extended to other breaks and size reduction systems because, if NIR reflectance could estimate granulation of first-break ground wheat, a coarsely ground product, it should be able to estimate more accurately the relatively finer ground products like second- or third-break, sizings, and middlings. As the intermediate milling products become finer they become more chemically homogenous, which would make granulation estimation by NIR reflectance easier and more accurate. Furthermore, the typical size range of first-break ground wheat should be narrower than what was used in this study; hence, the estimates of granulation should improve. The advantage of an NIR reflectance-granulation system is that only one NIR reflectance spectrometer would be used for each roller mill or for the entire flour mill because several detectors could be connected to a single spectrometer by fiber-optic probes. NIR technology has been proven in other applications for multipoint, multiconstituent monitoring (Bickel 1989; Robertson et al 1989). NIR reflectance-based systems for online analysis of flour protein, ash, moisture, and adjustments of additives such as gluten or starch have been reported (Williams et al 1981; Osborne and Fearn 1986; Posner and Wetzel 1986; Sosland 1987; Davies 1988; Anonymous 1994; Posner and Hibbs 1997). Some flour mills are already using an NIR spectrometer for flour constituent monitoring. Therefore, a redesign of the control system would not need a separate sensor for granulation. NIR reflectance technology could pave the way for the total control of flour milling based on simultaneous measurement of physical (granulation) and chemical properties of input wheat, intermediate products, and flour.

The method described here used offline methods as a first step to evaluate the feasibility of using an NIR reflectance spectrometer for online granulation sensor. The next step is to evaluate this technique as a part of an online system. In a flour mill, for a particular roll stand, the ground wheat could be sampled to get the desired amount, then mixed and dropped to the NIR reflectance sample cells using the described sample presentation method. The NIR reflectance system could, therefore, include a sampler, mixer, and sample presentation device for each roller mill. A fiber-optic cable could link the NIR beam source and detector for each roller mill to a single spectrometer. A relationship between granulation and roll gap would be established as a basis of the algorithm for online roll-gap control.

SUMMARY AND CONCLUSIONS

This study validated the diode array NIR reflectance spectrometer as an offline granulation sensor for first-break ground wheat. PLS regression models using unit area normalization combined with baseline correction or second derivative had excellent predictive ability for each of the four size fractions (>1,041, >375, >240, and >136 μm. Unit area normalization corrected for the path length effects of first-break ground wheat coarse size fraction (>1,041 μm). Cumulative mass data, expressed in terms of larger than a size, helped finer low-mass size fractions improve prediction because of higher sample mass. The best model had standard errors of prediction of 4.07, 1.75, 1.03, and 1.40 and r^2 of 0.93, 0.90, 0.88, and 0.38 for particle-size ranges of >1,041, >375, >240, and >136 μm, respectively. It is projected that, as particle size becomes finer for succeeding break and reduction stages, granulation sensing using an NIR reflectance spectrometer would become more accurate. Follow-up studies are needed to evaluate the NIR reflectance spectrometer with ground wheat from specific wheat classes and for online granulation sensing.

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