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Granulation sensing of first-break ground wheat using a near-infrared reflectance spectrometer: studies with soft red winter wheats†

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Abstract: A near-infrared reflectance spectrometer, previously evaluated as a granulation sensor for first-break ground wheat from six wheat classes and hard red winter (HRW) wheats, was further evaluated for soft red winter (SRW) wheats. Two sets of 35 wheat samples, representing seven cultivars of SRW wheat ground by an experimental roller mill at five roll gap settings (0.38, 0.51, 0.63, 0.75 and 0.88 mm), were used for calibration and validation. Partial least squares regression was applied to develop the granulation models using combinations of four data pretreatments (log(1/*R*), baseline correction, unit area normalisation and derivatives) and subregions of the 400–1700 nm wavelength range. Cumulative mass of size fraction was used as reference value. Models that corrected for path length effects (those that used unit area normalisation) predicted the bigger size fractions well. The model based on unit area normalisation/first derivative predicted 34 out of 35 validation spectra with standard errors of prediction of 3.53, 1.83, 1.43 and 1.30 for the >1041, >375, >240 and >136 µm size fractions respectively. Because of less variation in mass of each size fraction, SRW wheat granulation models performed better than the previously reported models for six wheat classes. However, because of SRW wheat flour's tendency to stick to the underside of sieves, the finest size fraction of these models did not perform as well as the HRW wheat models.

Keywords: near-infrared; near-infrared reflectance; granulation; particle size; flour milling; soft wheat

INTRODUCTION

Granulation is a form of particle size distribution (in cumulative per cent mass) which flour millers use as a basis for adjustments in the milling system. Granulation can give information on roll corrugation condition, roll gap setting and sifter efficiency.[1] Because it is a criterion for ensuring the optimum distribution of ground wheat products, Pasikatan[2] proposed the development of an on-line granulation sensor as a basis for roller mill automation. Real-time adjustment of roll gap based on granulation information from a sensor could help optimise flour yield. The near-infrared (NIR) reflectance spectrometer has been identified as a potential granulation sensor because of its sensitivity to the particle size of a ground sample in addition to the absorbing compounds,[3–5] its rapidity and the availability of fibre optic probes for remote measurement applications.[6–8] O'Neil *et al*[9] recently used NIR reflectance to determine the cumulative particle size distribution (11 size fractions, size range 5.8–564 µm) of microcrystalline cellulose. Theoretical equations and empirical studies showing the feasibility of this technique for particle size sensing have been reviewed by Pasikatan *et al.*[10]

Using an off-line method, a diode array NIR reflectance spectrometer was previously evaluated as a potential on-line granulation sensor for first-break ground wheat from six wheat classes[11] with the intent of developing a wheat class-independent calibration. However, non-linearity could be reduced by subdivid-

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ing samples and developing separate calibrations for subgroups.[12] Thus grouping calibrations according to specific wheat classes has the potential to improve calibrations. This was also consistent with the practice of milling specific wheat classes in a particular mill. These findings were confirmed by results of granulation models for hard red winter (HRW) wheats.[13] Improved predictions were obtained for HRW wheat models, compared with six-wheat-class models, because of their narrower particle size distribution and better sieving properties.[13] Spectral variations were caused mostly by particle size effects. This study was an extension of the two earlier studies. Its objectives were (1) to evaluate further the NIR reflectance spectrometer as a granulation sensor using first-break ground wheat from soft red winter (SRW) wheat cultivars, (2) to develop and validate NIR reflectance–granulation models and (3) to compare these with the previously developed granulation models for six wheat classes and HRW wheats.

MATERIALS AND METHODS

Seven cultivars of SRW wheat from the 1996 and 1999 crop years were selected (Table 1). From each cultivar, 10 representative wheat samples (560 g each) were obtained, five samples each for the calibration and validation sets. The equipment and procedures used for cleaning and for sampling to obtain subsamples for milling and for measurement of physical properties were described by Pasikatan *et al.*[11]

Milling subsamples (440 g each) were ground using an experimental (first-break) roller mill described by Fang *et al.*[14] Roller mill settings used in this study were $52.3\,\text{rad}\,\text{s}^{-1}$ (500 rpm) fast roll speed, $20.9\,\text{rad}\,\text{s}^{-1}$ (200 rpm) slow roll speed, 2.5:1 roll speed differential and $1.34\,\text{kg}\,\text{m}^{-1}\,\text{s}^{-1}$ feed rate. Roll gaps were set at 0.38, 0.51, 0.63, 0.75 and 0.88 mm. These settings were chosen so that variations in particle size distribution of the wheat samples could come only from SRW wheat cultivars and roll gap, thus enabling comparison with the granulation models from six wheat classes and HRW wheats. The procedures for first-break grinding and sifting were reported by Pasikatan *et al.*[11] The milling subsamples for the validation set were ground, sampled and sifted independently of the subsamples for the calibration set, so that models would approximate actual milling conditions where ground wheats predicted would differ somewhat from those used in the calibration. Ground wheats were sieved using a Lab Sifter (Great Western Co, Leavenworth, KS, USA) with a stack of 20W (1041 μm opening), 50GG (375 μm), 70GG (240 μm) and 10XX (136 μm) sieves and a pan. These are the sieve sizes used in flour mills for determining the granulation of first-break ground wheat. Cumulative per cent mass of >1041, >375, >240 and >136 μm is the customary form used by millers in granulation plots and thus was used here as reference unit.

A diode array NIR reflectance spectrometer (Perten Instruments, Springfield, IL, USA) was used to collect spectral data as absorbance ($\log(1/R)$) from 400 to 1700 nm at 5 nm increments. Each sample's spectrum represented 30 spectral scans collected and averaged. Four spectra were collected from each ground wheat sample and averaged to reduce noise. Ground wheat samples were dropped using a Motomco (Dickey-John Co, Auburn, IL, USA) drop cell above a raised cylinder with three layers of randomising rods onto the sample window of the spectrometer. This was done to ensure repeatability of the representative layer presented to the NIR spectrometer.[11]

Cumulative per cent mass models were developed based on partial least squares (PLS) regression[15] (hereafter, model refers to cumulative per cent mass model). All spectra were mean centred before analysis. The spectral pretreatments evaluated were unit area normalisation, baseline correction, first and second derivatives and their combinations. A third-order polynomial with 11 and 25 data points was used to calculate the first and second derivatives respectively by the Savitzky–Golay method.[16] Since granulation of first-break ground wheat refers to the four cumulative size fractions, each model considered was for all size fractions for faster implementation of size estimation in future automated systems.

Table 1. Physical properties of soft red winter (SRW) wheat cultivars (untempered) used in experiments

Label	Cultivar	Origin (state, crop year)	BD[a] (kg hl^{-1})	MC[b] (%)	TKW[c] (g)	MKS[d] (mm)	TD[e] (g cm^{-3})
SRW1	Caldwell	MI, 1996	75.2	13.4	34.4	3.2	1.38
SRW2	Pocahontas	VA, 1996	77.5	12.7	40.5	3.4	1.36
SRW3	Coker 9543	AR, 1999	78.2	12.0	33.6	3.0	1.39
SRW4	Hopewell	GA, 1996	74.3	12.3	36.4	3.3	1.40
SRW5	Jaypee	AR, 1996	75.6	12.2	31.1	2.9	1.35
SRW6	Pioneer	NC, 1999	77.7	11.8	31.4	2.9	1.44
SRW7	Roane	VA, 1999	80.3	13.0	32.9	3.1	1.43

[a] Bulk density or test weight.
[b] Moisture content (wet basis), oven method.
[c] Thousand-kernel weight.
[d] Mean kernel size.
[e] True density.

Table 2. Particle size distribution of first-break ground wheat obtained from grinding seven soft red winter (SRW) wheat cultivars at different roll gaps. Mean[a] (% mass) ± standard deviation for each size range

Cultivar[b]	>1041 μm	375–1041 μm	240–375 μm	136–240 μm	<136 μm
Calibration set					
SRW1	66.98 ± 11.44a	19.20 ± 6.40c	9.33 ± 2.93c	3.98 ± 1.96cd	0.52 ± 0.23ab
SRW2	64.38 ± 13.26cd	21.33 ± 7.23ab	9.34 ± 3.13c	3.93 ± 1.95d	1.02 ± 1.17a
SRW3	62.29 ± 12.74e	21.24 ± 6.14b	11.20 ± 3.48a	4.75 ± 2.85a	0.53 ± 0.37bc
SRW4	65.32 ± 12.15b	19.20 ± 5.64c	10.60 ± 3.51b	4.44 ± 2.56b	0.44 ± 0.48c
SRW5	64.00 ± 12.63c	18.84 ± 5.66c	10.51 ± 3.46b	5.63 ± 2.64a	1.02 ± 1.02bc
SRW6	67.00 ± 13.47a	22.14 ± 8.62a	8.05 ± 3.53d	2.51 ± 1.16e	0.30 ± 0.19c
SRW7	63.01 ± 13.17de	22.10 ± 6.76a	10.37 ± 3.69b	4.11 ± 2.40bc	0.41 ± 0.44c
All	64.71 ± 11.66	20.58 ± 6.24	9.92 ± 3.24	4.19 ± 2.25	0.61 ± 0.66
Validation set					
SRW1	67.28 ± 12.30a	18.43 ± 6.24c	8.87 ± 3.07c	3.10 ± 1.02cd	2.32 ± 2.33ab
SRW2	64.25 ± 13.24cd	21.62 ± 7.26ab	9.17 ± 3.14c	2.53 ± 0.85d	2.43 ± 2.37a
SRW3	62.70 ± 12.92e	20.59 ± 6.03b	10.78 ± 3.33a	5.19 ± 2.85a	0.73 ± 0.77bc
SRW4	66.42 ± 12.05b	19.01 ± 5.59c	10.23 ± 3.88b	3.97 ± 2.38b	0.38 ± 0.26c
SRW5	64.97 ± 12.28c	18.35 ± 5.29c	10.59 ± 3.18b	5.29 ± 3.15a	0.79 ± 0.66bc
SRW6	67.75 ± 14.13a	21.76 ± 9.14a	8.50 ± 3.81d	1.75 ± 1.11e	0.23 ± 0.10c
SRW7	63.63 ± 12.14de	21.70 ± 6.28a	10.28 ± 3.41b	3.99 ± 2.31bc	0.40 ± 0.17c
All	65.29 ± 11.70	20.21 ± 6.22	9.78 ± 3.21	3.69 ± 2.30	1.04 ± 1.48

$n = 5$ for each cultivar.
[a] Means in each column followed by the same letter are not significantly different ($p < 0.05$).
[b] SRW1 = Caldwell, MI, 1996; SRW2 = Pocahontas, VA, 1996; SRW3 = Coker 9543, AR, 1999; SRW4 = Hopewell, GA, 1996; SRW5 = Jaypee, AR 1996; SRW6 = Pioneer, NC, 1999; SRW7 = Roane, VA, 1999.

RESULTS AND DISCUSSION

Wheat cultivar and roll gap effects on particle size distribution and absorbance spectrum

Both roll gap and SRW wheat cultivar significantly influenced the mean per cent mass of first-break ground wheat for each size fraction ($p < 0.05$) (Table 2). The per cent masses of size fractions >1041 and <136 μm of ground SRW wheats were less than those previously reported for six wheat classes[11] and HRW wheats.[13] Thus the amounts of size fractions 375–1041, 240–375 and 136–240 μm of the SRW wheats were greater than those of the corresponding size fractions of the six wheat classes and HRW wheats. The standard deviations for the size fractions >1041, 375–1041 and <136 μm were smaller for SRW wheats than for the six wheat classes and HRW wheats. Typical of first-break ground wheat, most of the wheat particles lay within the size ranges >1041 μm (62.29–67.75% by mass) and 375–1041 μm (18.35–22.14%). For a particular SRW wheat cultivar the effect of roll gap on the ground wheat could be distinguished by NIR reflectance, ie finer grinds due to smaller roll gaps had lower absorbances than coarser grinds (Fig 1). For a particular roll gap the effect of SRW wheat cultivar on the ground product could also be distinguished by NIR reflectance, ie grinds for some SRW wheat cultivars were finer and thus had lower absorbances (Fig 2). The differences were more distinct at the longer NIR wavelengths of about 1450–1700 nm. In this region lie the absorption wavelengths for starch and protein, ie 1450 and 1580 nm (starch) and 1510 nm (protein).[17] Because the protein matrix and starch–protein adhesion influence wheat hardness,[18] and hardness influences

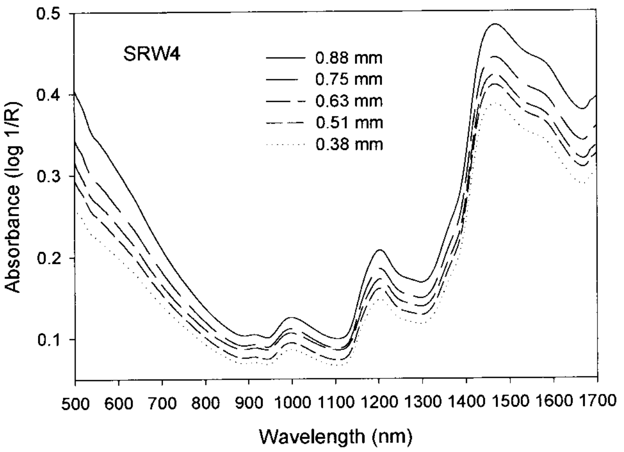

Figure 1. Absorbance spectra of Hopewell soft red winter (SRW4) wheat ground at various roll gaps (0.38–0.88 mm).

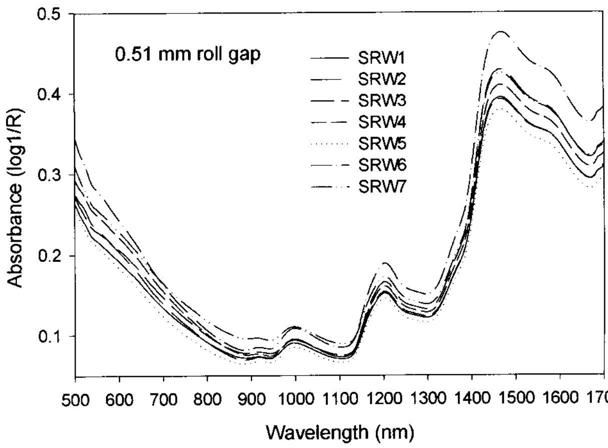

Figure 2. Absorbance spectra of seven soft red winter (SRW) wheat cultivars ground at a roll gap of 0.51 mm.

Table 3. Comparison of partial least squares calibration models for >1041 μm size range

Model[a]/Wavelength range (nm)	F[b]	R^2	*SECV[c]*
Log(1/*R*)			
550–700	4	0.69	6.42
700–1100	11	0.88	4.09
1100–1700	4	0.90	3.57
550–1700	11	0.93	3.18
Normalisation and 1st derivative[d]			
550–700	3	0.67	6.58
700–1100	11	0.89	3.91
1100–1700	8	0.92	3.26
550–1700	4	0.95	2.48
Normalisation and 2nd derivative[e]			
550–700	3	0.64	6.87
700–1100	10	0.88	4.09
1100–1700	8	0.88	3.97
550–1700	4	0.95	2.65

[a] Only representative models are shown.
[b] PLS factors.
[c] Standard error of cross-validation.
[d] Third-degree polynomial with 11 data points by Savitzky–Golay method.
[e] Third-degree polynomial with 25 data points by Savitzky–Golay method.

grinding response as expressed in particle size, shape and manner of fracture,[19] Fig 2 indicates that cultivar differences in absorbance were due to hardness. For example in Fig 2, Jaypee (SRW5, the one with the least absorbance) is the softest among the seven cultivars, with a mean hardness of 19.7 (as measured by a single-kernel characterisation system, where 0 is softest and 100 is hardest), whereas Roane (SRW7, the one with the highest absorbance) is the hardest, with a mean hardness of 35.8.

NIR reflectance–granulation models

The wavelength range that yielded granulation models with good predictive ability was evaluated using the biggest size fraction, >1041 μm (Table 3). For first break the prediction of this size fraction is the most important, because it is used to determine the break release, and break release is used to approximate the roll gap. The visible range (550–700 nm), which contains colour information, has very little particle size information, as shown in higher standard errors of cross-validation (SECVs) and lower R^2 values. Some particle size information lies in the short-wave NIR region (700–1100 nm), but much more in the NIR region (1100–1700 nm) (Table 3). However, more particle size information is obtained when the entire visible–NIR region (550–1700 nm) is used (Table 3). Osborne and Fearn[17] and Devaux *et al*[20] reported that particle size effects are expressed in the entire spectrum, so models that use wider wavelength regions have better predictive ability. Also, the breakage properties of wheats are influenced by the endosperm's starch–protein matrix,[21] and since the absorbing bands for starch and protein are in the NIR region, the granulation models perform better when these wavelength regions are included (Fig 2). Similar results were reported for granulation models using six wheat classes[11] and HRW wheats.[13] Table 3 also shows that unit area normalisation, a pretreatment that corrects for path length variations,[15] yielded better models than those pretreatments that do not. In diffuse reflectance, varying particle size (particularly coarse particles) and sample porosity changes the path length of light.[8,9,22]

The better SRW wheat granulation models have fewer (one to four) PLS factors (Tables 3 and 4), nearly as few as those of HRW wheat granulation models (two or three).[13] As suggested by the results of Devaux *et al*,[20] factors beyond three could be describing effects other than particle size and surface effects. The most likely effect is the sieving behaviour of ground soft wheat. Soft wheat particles tend to adhere to the mesh of fine sieves, whereas hard wheat flours freely pass the sieve mesh.[23] This has been attributed to the rough surface or texture of soft wheat flour particles, which are therefore more cohesive or less free flowing than hard wheat flours.[24,25]

For the >1041 μm size fraction, SRW wheat models had lower SECVs than these previously reported for six-wheat-class[11] and HRW wheat[13] models; R^2 values were about the same. This could be explained by the lower standard deviations for particles of this size fraction for SRW wheat models compared with those for six-wheat-class and HRW wheat models. For size fractions >375 and >240 μm the SECVs for SRW

Table 4. Partial least squares statistics of selected calibration models for NIR reflectance–granulation (550–1700 nm)

Size fraction/Pretreatment	F[a]	R^2	*SECV[b]*
>1041 μm			
Log(1/*R*)	11	0.93	3.18
Normalisation	7	0.94	2.78
Normalisation and baseline correction	6	0.95	2.51
Normalisation and 1st derivative[c]	4	0.95	2.48
Normalisation and 2nd derivative[d]	4	0.95	2.65
>375 μm			
Log(1/*R*)	13	0.95	1.30
Normalisation	8	0.92	1.68
Normalisation and baseline correction	6	0.93	1.58
Normalisation and 1st derivative	4	0.93	1.53
Normalisation and 2nd derivative	4	0.94	1.48
>240 μm			
Log(1/*R*)	11	0.90	0.86
Normalisation	3	0.79	1.24
Normalisation and baseline correction	2	0.90	1.22
Normalisation and 1st derivative	2	0.80	1.22
Normalisation and 2nd derivative	2	0.81	1.19
>136 μm			
Log(1/*R*)	1	0.41	0.50
Normalisation	1	0.52	0.45
Normalisation and baseline correction	1	0.56	0.43
Normalisation and 1st derivative	1	0.58	0.42
Normalisation and 2nd derivative	1	0.58	0.42

[a] PLS factors.
[b] Standard error of cross-validation.
[c] Third-degree polynomial with 11 data points by Savitzky–Golay method.
[d] Third-degree polynomial with 25 data points by Savitzky–Golay method.

wheat models were higher than those for six-wheat-class and HRW wheat models. More endosperm adheres to the bran in soft wheats, and more firmly, than in hard wheats.[26,27] Also, soft wheat endosperm is amorphous and crumbles into smaller particles than hard wheat endosperm.[1] Thus ground SRW wheats have more particles of size 240–1041 µm to fill the spaces between coarse particles (>1041 µm) in the layer than the HRW wheats and six wheat classes (Table 2). Because SRW wheat particles (240–1041 µm) are more cohesive than those of ground HRW wheats, the layer presented to the NIR beam was likely more compacted than that obtained for ground HRW wheats. The NIR beam could then not penetrate as deep as in ground HRW wheats and thus did not encounter enough of the varying particle sizes in the layer. Therefore the diffused reflectance carried less particle size information from the layer, resulting in poorer calibration for SRW than HRW wheat models.

It was expected that only log(1/*R*) models would predict particle size well, because baseline correction and derivatives were designed to correct for large baseline variations caused by particle size effects.[28] However, models that used unit area normalisation have substantially improved SECV and R^2 compared with the mean-centred log(1/*R*) models (Table 4). The SECV reduction was 16–23% for SRW wheat models for the >1041 µm size range. This was consistent with the results from six-wheat-class and HRW wheat granulation models. The coarseness of first-break ground wheat could cause path length variations,[11,13] and unit area normalisation could correct for path length variations;[15] thus the main source of variations in these spectral data was path length effects.

As the particle size of the size fractions decreased, the SECV decreased (Table 4). The decrease in SECV corresponded with the reduction in standard deviation as the size fractions became finer and lesser in amount (Table 2). However, first-break ground wheat has more coarse particles (375 to >1041 µm), about 81–89% by mass, so light will likely encounter these particles more than the finer particles. The total absorption spectral characteristics would then be related more to the coarser than to the finer size fractions. Therefore, as the amount of the size fraction decreased, less of the spectral information would be related to particle size, and the prediction becomes difficult. It is critical then for size prediction using NIR reflectance that the size fraction must represent a substantial amount.[2]

The better SRW wheat granulation models were used to predict cumulative per cent mass from the validation set spectra. The best model, based on the prediction of larger size fractions, was unit area normalisation/first derivative. It predicted 34 out of 35 validation spectra with standard errors of prediction of 3.53, 1.83, 1.43 and 1.30 for the >1041, >375, >240 and >136 µm size fractions respectively (Table 5). Corresponding R^2 values were 0.94, 0.93, 0.81 and 0.32 respectively. The relationships of the predicted and measured values for this granulation model are shown in Fig 3.

Table 6 shows the validation statistics for the reference method and NIR-predicted values for the best SRW model. Except for the prediction for the size fraction >136 µm, all size fractions were predicted well. In a related study, where NIR reflectance spectroscopy was used to predict the cumulative particle size distribution of microcrystalline cellulose, O'Neil *et al*[9] reported that the decreased precision of calibration at the extreme quantiles or size fractions could be explained by their more skewed distribution curve. In flour milling, the size fraction <136 µm, which influences the prediction of the >136 µm fraction, is called first-break flour and is present in

Table 5. Prediction statistics for selected NIR reflectance–granulation models[a]

Size fraction/Pretreatment	*Region (nm)*	R^2	*SEP*[b]
>1041 µm			
Normalisation and baseline correction	700–1500	0.94	4.22
Normalisation and 1st derivative[c]	550–1700	0.94	3.53
Normalisation and 1st derivative	700–1500	0.95	3.69
>375 µm			
Normalisation and baseline correction	700–1500	0.92	2.03
Normalisation and 1st derivative	550–1700	0.93	1.83
Normalisation and 1st derivative	700–1500	0.93	1.93
>240 µm			
Normalisation and baseline correction	700–1500	0.81	1.39
Normalisation and 1st derivative	550–1700	0.81	1.43
Normalisation and 1st derivative	700–1500	0.81	1.38
>136 µm			
Normalisation and baseline correction	700–1500	0.32	1.30
Normalisation and 1st derivative	550–1700	0.32	1.30
Normalisation and 1st derivative	700–1500	0.30	1.30

[a] All models predicted 34 out of a total of 35 spectra of the validation set.
[b] Standard error of performance.
[c] Third-degree polynomial with 11 data points by Savitzky–Golay method.

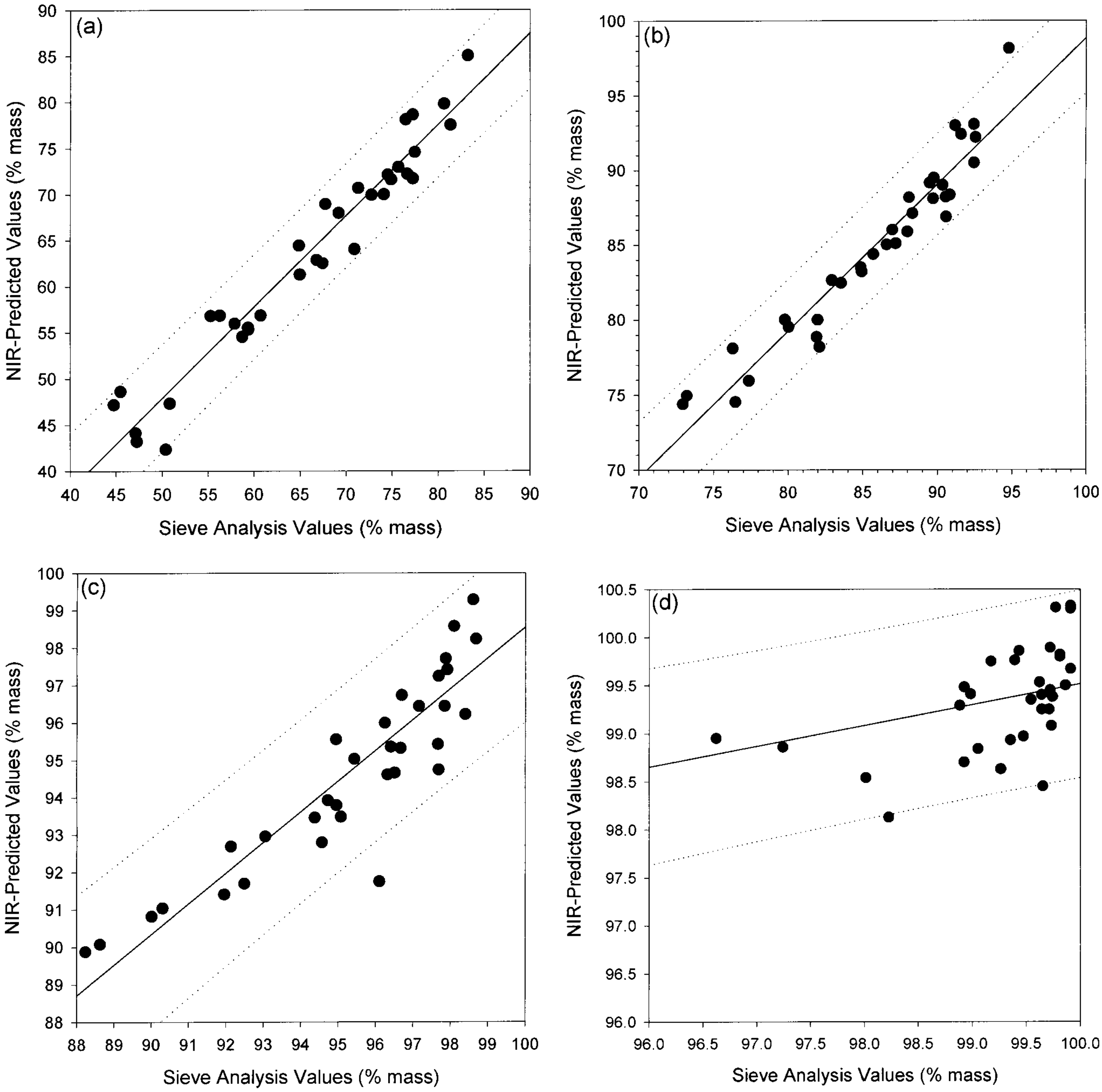

Figure 3. Relationship between NIR reflectance-predicted and sieve analysis values for (a) >1041, (b) >375, (c) >240 and (d) >375 μm size fractions of first-break ground SRW wheat. Dotted lines are the 95% prediction intervals; full line shows the best fit.

Table 6. Validation statistics for best model[a] for predicting granulation of first-break ground SRW wheat

Size fraction (μm)	*Sieve analysis (reference method)*[b] *(% mass)*	*NIR-predicted value*[b] *(% mass)*	*Slope*[c]	*CV*[d]	*RPD*[e]	*RER*[f]
>1041	65.85 ± 11.38	63.57 ± 11.60	0.99	5.36	3.22	10.90
>375	85.76 ± 5.87	84.90 ± 5.97	0.98	2.13	3.21	11.96
>240	95.37 ± 2.96	94.75 ± 2.70	0.82	1.50	2.07	7.66
>136	98.95 ± 1.05	99.29 ± 0.57	0.22	1.31	1.15	4.88

[a] Normalisation/first derivative (550–1700 nm) predicted 34 out of 35 validation spectra.

[b] Mean per cent mass ± standard deviation for *n* samples.

[c] Based on linear regression equation.

[d] Coefficient of variability (SEP × 100/ mean of reference values).[22]

[e] Standard deviation of reference values divided by the SEP.

[f] Range of reference data divided by the SEP.

negligible amount. First-break flour does not go on to a further process like the larger size fractions of first-break ground wheat, so its accurate prediction is not as critical to the control of flour milling as the larger fractions. As this NIR reflectance-based system is applied to the succeeding break and reduction stages in milling, the predictions would further improve, because the particles would be finer and the products would be more chemically homogeneous. When chemical-based information, such as protein content and starch damage, is included in a control algorithm in addition to granulation, an NIR reflectance-based control system would be more suitable than other proposed systems, such as that based on image analysis.[29]

CONCLUSIONS

Off-line calibration validated the diode array NIR reflectance spectrometer as a potential on-line granulation sensor for SRW first-break ground wheat. NIR reflectance–granulation models from ground SRW wheat spectra performed better than the previously reported six-wheat-class models owing to reduced variation in the mass of size fractions. However, the finest size fraction ($>136\,\mu m$) in SRW wheat models did not perform as well as in HRW wheat models, because of the tendency of soft wheat flours to adhere to the underside of sieves. The best model, unit area normalisation/first derivative, predicted 34 out of 35 validation spectra with standard errors of prediction of 3.53, 1.83, 1.43 and 1.30 for the >1041, >375, >240 and $>136\,\mu m$ size fractions respectively. These results further validated the NIR spectrometer as a potential on-line granulation sensor for automating roller mills. As this technique performed better for a specific wheat class than an all-wheat-class model, the performance of NIR reflectance–granulation models for other wheat classes or blended wheats should be further studied.

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