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Evaluation of Flour Particle Size Distribution by Laser Diffraction, Sieve Analysis and Near-infrared Reflectance Spectroscopy

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ABSTRACT

Laser diffraction and sieve analysis were used to measure flour particle size distributions as per cent volume and per cent weight, respectively, among different wheat types and milling methods. Near-infrared (NIR) reflectance spectroscopy was used to predict the per cent volume of flour particles within selected size ranges based on laser diffraction reference values. According to laser diffraction analysis, 89–98% of the flour particles were distributed within the size ranges 10–41 μm and 41–300 μm, and 2–11% of the particles were distributed within the size range <10 μm. Flour particle size distributions were different ($P<0{\cdot}05$) among the wheat types tested, except that hard red winter and hard white wheats were not different in flour particle size <10 μm, and hard red spring and hard white wheats were not different in flour particle size within 10–41 μm and 41–300 μm. The milling method affected particle size distributions of hard wheat flours but not those of soft wheat flours. A high correlation ($r=0{\cdot}95$) occurred between per cent volume and per cent weight of hard wheat flour particles <45 μm, but the correlation decreased ($r=0{\cdot}77$) when soft wheat flours were included in the comparison. Near-infrared calibration equations were developed by partial least-squares regression for predicting the per cent volume of flour particles. The per cent volume of flour particles within the size ranges <10 μm, 10–41 μm and 41–300 μm were predicted by NIR within ±2 standard errors for ⩾96% of the flours tested.

INTRODUCTION

Flour comprises a range of particle sizes. According to the type of wheat milled, flour is often evaluated subjectively by the 'feel' and described by its sharpness, smoothness, silkiness, granularity and fluffiness[1]. Objective measurements have been made of particle size distributions of flours and of ground wheats as a means of evaluating flour quality. These measurements have included sieve analysis[2–10], microscopy[11,12], sedimentation[5,13], Coulter Counter[14,15] and laser diffraction[9,16,17]. The particle size of wheat starch has been evaluated by image analysis[18]. The granulation properties of hard and soft wheat endosperms were highly correlated with microscopic measurements, kernel hardness and particle size index values[12]. Particle size was affected by wheat hardness and wheat class[19], type of grinder[9,20], and grinding time[21]. NIR reflectance spectra were shown to be affected by variability in the particle size characteristics of ground wheats due to variability in grinding[22,23].

This investigation reports similarities and differences in the particle size distributions of flours derived from various wheat types and from different experimental milling methods. The objectives of the study were to compare the laser diffraction and sieve analysis methods for measuring flour particle size distributions, and to de-

ABBREVIATIONS USED: HRS = hard red spring; HRW = hard red winter; HW = hard white; SRW = soft red winter; SW = soft white; NIR = near infrared; PLS = partial least squares; SEC = standard error of calibration; SECV = standard error of cross validation; SEP = standard error of performance.

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velop a method for predicting flour particle size distributions accurately by NIR reflectance spectroscopy.

EXPERIMENTAL

Selection of wheat types and milling methods

Flours were obtained from hard wheats, which included durum, hard red spring (HRS), hard red winter (HRW) and hard white (HW) wheats, and from soft wheats, which included soft red winter (SRW), soft white (SW) and club wheats. Wheats were cleaned and tempered to 15·0–15·5% moisture basis[17]. Three milling methods were employed: Miag pilot mill[24], Buhler experimental mill[17] and micro mill using Brabender Quadrumat Senior break and reduction heads and sieving in a Strand sifter with Tyler test sieves no. 35 (420 μm) and no. 80 (178 μm). The mirco-mill method was modified from the procedure reported by Finney and Bolte[25].

Flour particle size analysis by laser diffraction

A Coulter LS 130 optical bench (Coulter Scientific Instruments, Hialeah, Florida) was used to measure the per cent volume of flour particles distributed within selected size ranges. Flour (*ca.* 0·25 g) was suspended in methanol and circulated within the closed system of the optical bench and attached hazardous fluids module. Subsequently, flour particle size distributions were determined in triplicate (90 s/analysis) by laser diffraction light scattering according to the Fraunhofer diffraction theory as described by Hoff and Bott[26].

Flour particle size analysis by sieving

A GilSonic AutoSiever (Gilson Company, Worthington, Ohio) was used to separate flours into fractions according to sieve mesh size. Flour (3·0 g) was applied to a sieve stack, which included U.S. Standard sieves no. 40 (425 μm), no. 50 (300 μm) and no. 325 (45 μm). The total sieve time was set at 5·4 min, and included both vertical and horizontal tapping and sonic pulsing. Sonic pulsing consisted of 3600 pulses/min (50/60 Hz), and the amplitude of the pulse was adjusted to allow the flour to flow freely on the sieves. The per cent weight of flour particles that passed through the no. 325 sieve was determined and compared with the per cent volume of particles <45 μm previously determined by laser diffraction. The results from this comparison were used to establish which values, either per cent weight or per cent volume, could be used best to develop calibrations for predicting flour particle size distributions by NIR.

NIR predictions of flour particle size distributions

NIR and visible spectra were obtained from 296 samples of flour as $\log(1/R)$, where R = reflectance from 400 to 2500 nm, with an NIRSystems model 6500 spectrophotometer (Silver Springs, Maryland). The spectra of the sample population were defined according to algorithms described by Shenk and Westerhaus[27,28]. Based on principal component analysis[29], spectra were arranged according to standardized '*H*' (Mahalanobis) distances of each sample spectrum from the average spectrum. Spectra with '*H*' values >3·0 were eliminated as outliers as a method to establish population boundaries. Following the elimination of spectral outliers, the sample population was divided equally: one half was used to develop calibration constants and the other half was used as a validation set to test the calibration equations. Calibration equations were developed using partial least-squares (PLS) regression[27,28,29] for predicting flour particle size by NIR.

Statistics

The results were analyzed by SAS[30] procedures using analysis of variance and pairwise *t*-tests.

RESULTS AND DISCUSSION

Laser diffraction analysis of flour particle size distributions

Flour particles were distributed primarily within the size ranges 10–41 μm and 41–300 μm (Fig. 1). The two size ranges accounted for approximately 89–98% of the total particles, and the remaining 2–11% of the particles were distributed within the size range <10 μm. The particle size distributions of flours obtained from the various wheat types, including flours produced by all milling methods, are indicated in Table I. Durum wheat flour contained the highest and soft wheat flour the lowest per cent volume of particles distributed within the range 41–300 μm. Conversely, soft wheat flour contained the highest per cent volume of particles

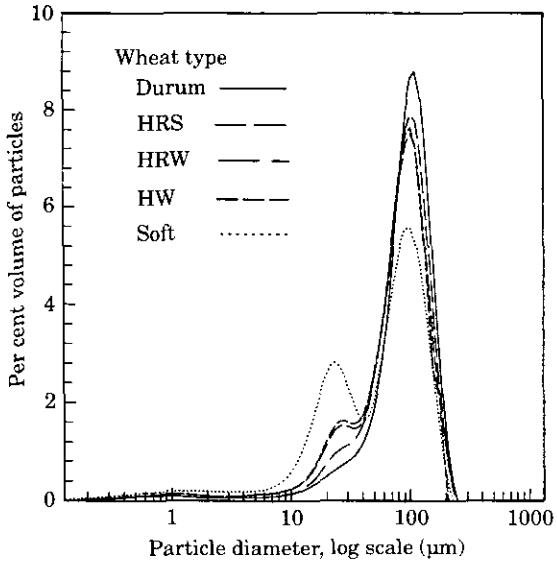

Figure 1 Particle size distributions of representative flours from durum, HRS = hard red spring, HRW = hard red winter. HW = hard white and soft wheats. Log scale of particle diameter (μm) versus per cent volume of flour particles.

II). For each particle size range, the comparison between duplicate sets of Buhler-milled HRS wheats reflected differences ($P<0.05$) in environmental conditions of the same wheat cultivars grown in two separate years. The comparison between the Buhler and micro-milling methods reflected differences ($P<0.05$) in the same HRS wheats milled by two different methods, but the same soft wheats milled by the two methods were not different at the same probability level. The comparison between HRS and HRW wheats milled in the Miag pilot mill reflected differences ($P<0.05$) between two types of hard wheat.

Hard wheat flours produced using the micro mill contained the highest proportion of large particles compared with flours obtained using the Buhler and Miag mills. Factors that could affect this difference are the number of break and reduction roll sections or pairs in each mill and variations in roll gap settings, roll speed differentials and roll configurations. The micro mill included two break and two reduction roll sections, the Buhler mill included three pairs of break and reduction rolls, and the Miag pilot mill included five pairs of break and six pairs of reduction rolls.

Table I Particle size distributions (per cent volume)[a] of flours obtained from various wheat types and measured by laser diffraction

	Particle size range		
Wheat type[b]	<10 μm	10–41 μm	41–300 μm
Durum ($n=8$)	$2.4\pm0.9a$	$5.5\pm2.6a$	$92.1\pm3.5a$
HRS ($n=145$)	$3.8\pm0.7b$	$12.0\pm3.2b$	$84.2\pm3.8b$
HRW ($n=57$)	$4.5\pm0.6c$	$17.1\pm4.0c$	$78.5\pm4.5c$
HW ($n=20$)	$4.4\pm0.7c$	$13.7\pm2.2b$	$82.2\pm2.7b$
SRW, SW, Club ($n=26$)	$8.6\pm1.0d$	$30.0\pm2.5d$	$61.5\pm3.3d$

[a] Mean per cent volume of flour particles for (n) samples ± standard deviation; means within each column with the same italic letter are not different ($P<0.05$).
[b] Wheat type: durum; HRS = hard red spring; HRW = hard red winter; HW = hard white; SRW = soft red winter; SW = soft white; club.

distributed within the ranges 10–41 μm and <10 μm. Flour particle size distributions were different ($P<0.05$) for the different wheat types tested, except that HRW and HW were not different in flour particle size <10 μm, and HRS and HW were not different in flour particle size within the ranges 10–41 μm and 41–300 μm.

Flour particle size distributions were affected by the milling method and the wheat type (Table

Other variations in flour particle size distributions may be attributable to differences in the starch–protein matrix of hard and soft wheat endosperms. Glenn and Saunders[19] reported that hard and soft wheats varied in the continuity of the protein matrix, starch–protein adhesion and intracellular spaces within the endosperm. Hard wheats were pliable and cohesive when sectioned, but soft wheats tended to crumble. Kent and Evers[6]

Table II Variations on flour particle size distribution (per cent volume)[a] as affected by milling method and wheat type

Wheat type[b]	Milling method	Particle size range		
		<10 μm	10–41 μm	41–300 μm
1-HRS (n=32)	Buhler	4·6±0·5	14·8±2·5	80·6±3·0
2-HRS (n=32)	Buhler	4·0±0·3	10·9±1·4	85·2±1·5
3-HRS (n=32)	Micro	2·8±0·4	7·7±1·6	89·3±2·0
4-HRS (n=49)	Miag	3·7±0·3	13·5±1·9	82·7±2·2
5-HRW (n=37)	Miag	4·6±0·6	18·9±3·6	76·4±4·2
6-Soft (n=13)	Micro	8·3±0·9	29·5±2·7	62·2±3·5
7-Soft (n=13)	Buhler	8·8±1·0	30·5±2·2	60·7±3·0

[a] Mean volume per cent of particles for (n) samples ± standard deviation.

[b] 1 and 2 include four cultivars of hard red spring wheat grown at four locations in 1991 and 1992, respectively; 2 and 3 include the same hard red spring wheats milled by two different methods; 4 and 5 include hard red spring and hard red winter wheats obtained from various growing locations in the U.S. in 1989; and 6 and 7 include the same soft wheats milled by two different methods.

indicated that the fragmentation properties of endosperm cells during milling were dependent largely upon protein content.

The distribution of flour particles within the size ranges <10 μm, 10–41 μm and 41–300 μm may be attributable to differences in the quantities of A-type and B-type starch granules dissociated from the protein mattix during milling. A-type starch granules were reported to range in size from 10 μm up to 36–50 μm in diameter; however, the upper size range was dependent upon cultivar and growing season[11,13]. B-type starch granules were reported to range in size from 1–10 μm. Pratt[32] indicated that flour particles falling within the size range 0 to 20 μm Stokes equivalent diameter (SED) were free protein, small starch granules, cell-wall material and damaged starch granules; within 20 to 35 μm SED were free starch granules; and above 35 μm SED were endosperm chunks with adhering protein. Because of these previous studies on flour particle size, the <10 μm, 10–41 μm and 41–300 μm size ranges were selected for this study.

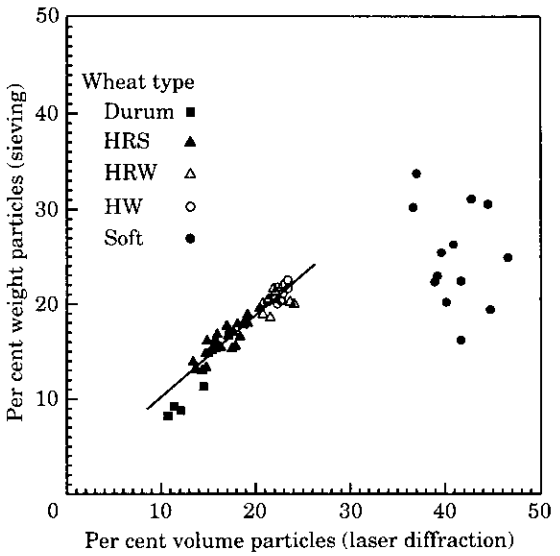

Figure 2 Relationship between the per cent volume and per cent weight of flour particles <45 μm as determined by laser diffraction and sieve analysis, respectively. Flours from Buhler-milled wheats included durum, HRS=hard red spring, HRW=hard red winter, HW=hard white and soft wheats. r=0·95.

Comparison between per cent volume and per cent weight of flour particles

A comparison was made between the per cent volume and per cent weight of flour particles measured by laser diffraction and sieve analysis, respectively (Fig. 2). Among the hard wheat flours, a high correlation coefficient (r=0·95) occurred between per cent volume and per cent weight of particles. When soft wheat flours were included in the comparison, the correlation decreased (r=

Table III Partial least-squares statistics for predicting flour particle size distribution (per cent volume) within three size ranges

Particle size range	n	Laboratory reference method[a]	NIR analysis method[a]	SEC[b]	r^2[c]	SECV[d]
10 μm	130	4·4 ± 1·4	4·3 ± 1·4	0·16	0·99	0·26
10–41 μm	132	14·5 ± 5·9	14·7 ± 5·8	0·59	0·99	0·87
41–300 μm	136	81·1 ± 7·2	80·3 ± 7·2	0·75	0·99	1·11

[a] Mean per cent volume of particles distributed within each size range for (n) samples in calibration set ± standard deviation.
[b] SEC = standard error of calibration.
[c] r^2 = coefficient of determination.
[d] SECV = standard error of cross validation.

0·77). For hard wheat flours, the mean per cent volume of particles <45 μm was slightly higher than, but not significantly different from, the corresponding mean per cent weight of particles passing through 45-μm sieve openings (18·4% ± 3·5 and 17·2% ± 3·5, respectively, $n = 51$, $P < 0·05$).

For soft wheat flours, however, the mean per cent volume and mean per cent weight of particles <45 μm were different (41·1% ± 3·0 and 25·1% ± 5·2, respectively, $n = 13$, $P < 0·05$), and the correlation coefficient between per cent volume and per cent weight was poor ($r = -0·24$). During the sieving operation, soft wheat flour particles did not pass freely through the sieve openings and adhered to the sieve mesh, but hard wheat flours flowed freely without adhering to the sieve mesh. The results of the comparison of the laser diffraction and sieve analysis methods suggested that, for all wheat types, the per cent volume of particles represented the best laboratory reference values for NIR calibration development and prediction of flour particle size distribution.

Predicting flour particle size distribution by NIR

NIR calibrations were developed for predicting flour particle size based on the per cent volume of particles distributed within the size ranges <10 μm, 10–41 μm and 41–300 μm. PLS regression[27,28,29] provided the best calibration equations when using the (2, 10, 10) mathematical transformation treatment: the second derivative of log ($1/R$), a segment length of 10 data points, over which the derivative was taken, and the segment length of 10, over which the function was smoothed.

The results of the PLS calibration statistics are shown in Table III. For each particle size range, PLS analysis resulted in low standard errors of cross validation (SECV) and high coefficients of determination (r^2). The SECV, or estimate of prediction error, was derived by splitting the calibration samples into groups, in which one group was reserved for validation and the other groups were used for calibration. Four cross-validations were performed, which resulted in the elimination of samples with high 't' residuals ($t > \pm 2·5$), or differences between laboratory reference values and predicted values. The number (n) of samples in the calibration set for each size range was determined after the elimination of spectral outliers and samples with high 't' residuals. The standard error of calibration (SEC) measured the best fit of the calibration samples, in which the lower the SEC, the better the fit. High coefficients of determination (r^2) were indicative of the performances of the regression equations[29,33].

Validating NIR calibration equations

The validation set of samples included flours that were not part of the calibration set of samples. Durum flours were eliminated as spectral outliers, and were not included in either the calibration or validation sets of samples. Three flour samples were eliminated from the validation set because of high 't' residual differences ($t > \pm 2·5$), thus resulting in the prediction of 139 samples per size range (Table IV). The validation statistics indicate the performance of the calibration equations, which include similarities between the laboratory reference and NIR predicted means and standard deviations, low standard errors of performance (SEP) associated with uncertainty of prediction, and high coefficients of determination (r^2). Since

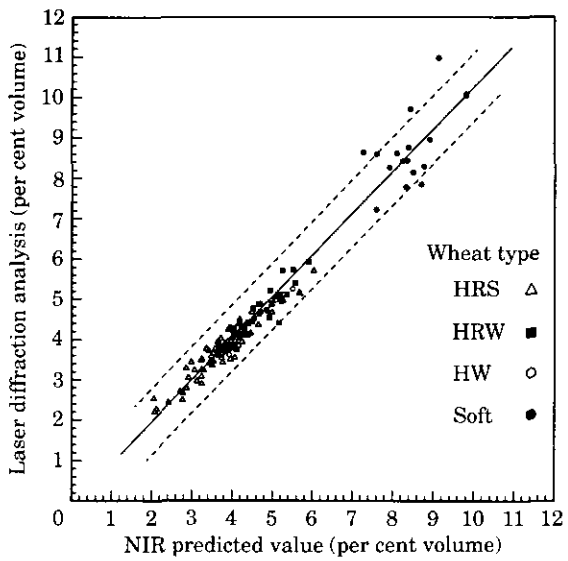

Figure 3 Relationship between the laser diffraction analysis method and NIR analysis method for predicting the per cent volume of flour particles for the size range <10. Dotted lines represent ±2 SEP limits. Flours include HRS = hard red spring and HRW = hard red winter wheats from the micro, Buhler and Miag milling methods and HW = hard white and soft wheats from the micro and Buhler milling methods. $y = 1{\cdot}03x - 0{\cdot}16$; $r^2 = 0{\cdot}96$; $n = 139$.

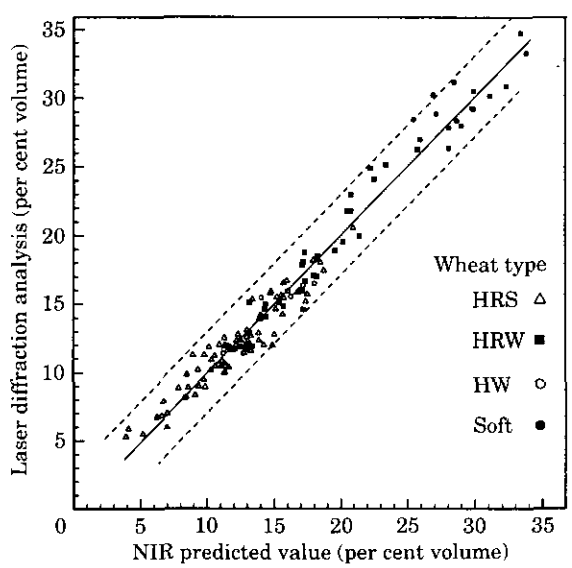

Figure 4 Relationship between the laser diffraction analysis method and NIR analysis method for predicting the per cent volume of flour particles for the size range 10–41 μm. Dotted lines represent ±2 SEP limits. Flours include HRS = hard red spring and HRW = hard red winter wheats from the micro, Buhler and Miag milling methods and HW = hard white and soft wheats from the micro and Buhler methods. $y = 1{\cdot}01x - 0{\cdot}21$; $r^2 = 0{\cdot}97$; $n = 139$.

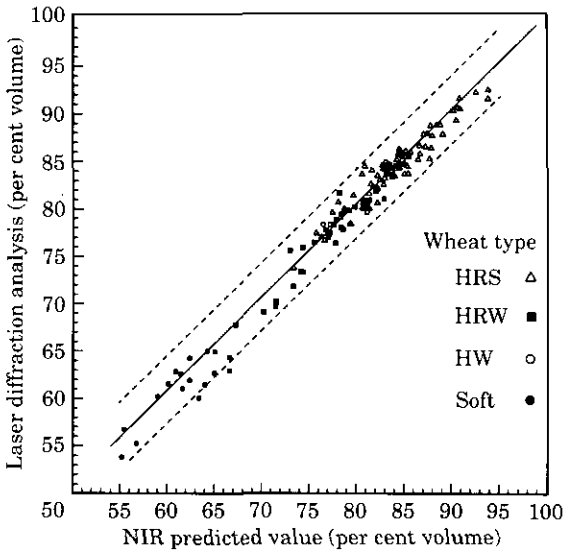

Figure 5 Relationship between the laser diffraction analysis method and NIR analysis method for predicting the per cent volume of flour particles for the size range 41 – 300 μm. Dotted lines represent ±2 SEP limits. Flours ionclude HRS = hard red spring and HRW = hard red winter wheats from the micro, Buhler and Miag milling methods and HW = hard white and soft wheats from the micro and Buhler milling methods. $y = 1{\cdot}00x - 0{>}15$; $r^2 = 0{\cdot}98$; $n = 139$.

the validation samples were independent of the calibration samples, the SEP values were slightly higher, and the r^2 values slightly lower, than the corresponding SECV and r^2 values for the calibration samples shown in Table III.

The relationships between the laser diffraction analysis method and NIR analysis method for predicting flour particle size for the size ranges <10 μm, 10–41 μm and 41–300 μm are illustrated in Figs 3, 4 and 5, respectively. NIR accurately predicted the per cent volume of flour particles within ±2 SEP for ⩾96% of the flour samples tested.

CONCLUSIONS

The particle size distributions of flours from all wheat types tested could be measured more precisely by laser diffraction than by sieve analysis. Soft wheat flours did not sieve as efficiently as hard wheat flours. Flour particle size was affected by milling method, wheat type and environmental growing conditions of the wheat. Flour particle size distributions within three size ranges could be predicted by NIR with high accuracy based on reference values obtained by laser diffraction analysis.

Table IV Validation statistics for predicting flour particle size distribution (per cent volume) within three size ranges

Particle size range	*n*	Laboratory reference method[a]	NIR analysis method[a]	SEP[b]	r^2[c]
<10 μm	139	4·6 ± 1·7	4·6 ± 1·6	0·35	0·96
10–41 μm	139	15·6 ± 6·7	15·6 ± 6·5	1·17	0·97
41–300 μm	139	79·8 ± 8·4	79·6 ± 8·2	1·31	0·98

[a] Mean per cent volume of particles distributed within each size range for (*n*) samples ± standard deviation.
[b] SEP = standard error of performance.
[c] r^2 = coefficient of determination.

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