Prediction of Damaged Starch in Straight-Grade Flour by Near-Infrared Reflectance Analysis of Whole Ground Wheat

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ABSTRACT

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The amount of damaged starch generated by grinding 25 g of whole wheat samples in a Udy cyclone mill was measured by enzymatic and near-infrared reflectance (NIR) methods. High correlations were achieved between the methods. In addition, it was possible to predict the amount of damaged starch induced by controlled Allis-Chalmers and Quadrumat Junior millings by the NIR analysis of the Udy-ground samples. Hard and soft classes of wheat could usually be separated by NIR analysis of damaged starch.

Much attention has been paid to the mechanical damage incurred by starch during milling. Starch granules are damaged by pressure or shear during milling, and many properties of doughs and baked goods are affected by the quantity of damaged starch present in the flour. For example, water absorption, gassing power, handling properties of doughs, and product texture are all affected by the quantity of damaged starch in the flour.

Early workers such as Pulkki (1938) and Jones (1940) measured damaged starch content in flour by utilizing staining techniques and microscopic examination. Later methods for damaged starch determination measured the amount of maltose produced when starch was partially digested by α-amylase. Because the damaged starch granules were more easily digested than sound starch granules, by measuring the maltose produced under controlled conditions, it was possible to quantify damaged starch. Such enzymatic methods were developed by Sandstedt and Mattern (1960) and by Donelson and Yamazaki (1962); the latter became an approved method of the AACC (1983). Farrand (1964) also measured maltose, but the units obtained by that method are normally about three times greater than the AACC approved methods (Williams and Le Seelieur 1970).

Near-infrared reflectance (NIR) spectroscopy analysis of grains and grain products was introduced in 1971 for measurement of grain moisture utilizing the principal developed by Norris (1964). That method provides very rapid nondestructive measurements, which in some instances are nearly as precise as standard laboratory tests. Williams (1979, 1986) applied NIR technology to measuring hardness (particle size index) and reported NIR hardness correlated highly with damaged starch. NIR was used to measure and predict the degree of damaged starch in purified starch and wheat flour by Osborne and Douglas (1981).

Watson et al (1977) found that differences among log values are sometimes related to the general hardness classification of the wheats, and that separate calibrations were necessary for different classes of wheat. Williams (1979) found that protein determinations could be improved by separate calibrations for hard and soft wheats.

The objectives of this study were to develop a method for estimating the amount of damaged starch in whole ground wheats by NIR analysis and to predict the amount of damaged starch that would be produced when subsamples of the same wheats were milled into straight-grade flours.

MATERIALS AND METHODS

The Calibration Set

Seventy-two wheats (comprising 36 cultivars and 36 commercial samples) that were selected to give a range in hardness across most wheat classes, included 15 hard red winter (HRW), 10 hard red spring (HRS), 11 durum, 10 club, nine soft red winter (SRW), nine

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eastern soft white winter (SWW), and eight western SWW wheat lots. Samples were subdivided to allow for a 1,500-g Allis-Chalmers (AC) milling, a 250-g Quadrumat Junior (QJ) milling, and five 25-g Udy cyclone grindings.

The Validation Set

A group of 50 samples from the 1986 Uniform Eastern Red Nursery (UERN) and Uniform White Nursery (UWN) were AC and QJ milled and cyclone ground. The 50 samples were cyclone ground twice (A and B), and each grind was analyzed twice by NIR. In addition, the cyclone samples from grind B were analyzed for damaged starch in duplicate by the lab method (AACC 1983). Twelve samples from grind A were analyzed (six red, six white) and used to adjust the NIR bias and skew. QJ and AC flours were also analyzed in duplicate for damaged starch by the lab method.

Sample Preparation

The 1,500-g AC samples were milled using the six-break variable reduction-pass system described by Yamazaki and Andrews (1982). A second sample of 250-g was milled with the QJ system described by Finney and Andrews (1986). Whole wheat samples were prepared using a Udy cyclone mill equipped with a 1.0-mm screen and a sample mill feed controller. Five 25-g subsamples were ground from each of the wheat lots. Untempered samples were ground at ambient moisture content; however, moisture differences between samples were small.

Analyses

QJ millings were analyzed for softness equivalence as described by Finney and Andrews (1986). Cyclone-ground wheats, QJ flours, and AC flours were analyzed for damaged starch content using the rapid enzymatic method described by Donelson and Yamazaki (1962).

TABLE I
Filters and Regression Coefficients for Hard and Soft Wheat Damaged Starch Calibrations Using Near-Infrared Reflectance

Wheat	Filter (nm)	Regression Coefficient
Hard[a]		
	2,336	1,556.0
	2,310	−1,272.0
	2,208	−339.2
	1,818	−852.0
	1,940	−16.6
	1,680	991.6
Bias		−7.3
Soft[b]		
	2,348	1,058.1
	2,310	−1,400.1
	2,139	591.8
	1,759	525.7
	1,940	−107.2
	1,722	−732.8
Bias		−3.5

[a] From 25 hard wheat samples (durum excluded).
[b] From 36 soft wheat samples.

TABLE II
Correlation Between Enzymatic and Near-Infrared Analysis Methods for Damaged Starch Determination on Cyclone-Ground Wheats

Wheat[a]	No. of Entries	Correlation Coefficient (r)	Coefficient of Determination (r^2)	Standard Error of Estimate
HRW	15	0.83	0.69	0.21
HRS	10	0.88	0.77	0.70
Hards	25	0.84	0.71	0.49
Club	10	0.72	0.52	0.25
SRW	9	0.63	0.40	0.19
ESWW	9	0.82	0.68	0.16
WSWW	8	0.84	0.70	0.18
Softs	36	0.83	0.69	0.22
All	61	0.97	0.93	0.35

[a] HRW = Hard red winter, HRS = hard red spring, SRW = soft red winter, ESWW = eastern soft white winter, and WSWW = western soft white winter.

NIR spectra were obtained on all 72 cyclone-ground samples using a Technicon InfraAlyzer 400. Log (1/reflectance) values were obtained for the 19 standard filters at wavelengths ranging from 1,445 to 2,348 nm. In preliminary NIR studies the results for hard and soft wheat classes separated into two distinct populations, and separate damaged starch calibrations were derived for them. It was found that hard and soft wheats could readily be identified by examining the log (1/R) values at 1,759 nm. If the log (1/R) values were larger than 0.28, the samples were considered hard, and the hard wheat calibration was used. The remaining samples were analyzed by using the soft wheat calibration values for damaged starch determination. Multiple linear regression analyses were performed to establish the relationship between damaged starch content and log (1/R) values. The regression coefficients then became the values that were programmed into the spectrophotometer and used for the analysis. Table I presents the filters used and the accompanying regression coefficients necessary for the determination of damaged starch in either hard or soft wheats. AACC approved method 39-70, which calls for 1,680 and 2,230 nm wavelengths, was used to determine wheat hardness for enzymatically determined damaged starch and various milling parameters.

Each of the five cyclone grindings was analyzed once by NIR so that variation due to grinding could be determined. In addition, one grinding was analyzed twice so that the variation due to the NIR method could be separated from the variation due to grinding.

The durum wheats were analyzed for damaged starch for comparison with hardness, but they were found to be so different from the other classes that they were not included in the comparisons between NIR and laboratory determinations of damaged starch.

A second group of samples was analyzed to confirm the validity of the calibrations.

RESULTS AND DISCUSSION

NIR Versus Enzymatic Determinations of Damaged Starch

The cultivars evaluated represented six classes or subclasses of wheat, with durum wheats being omitted as previously stated, and included a wide range (5.6 percentage points) in damaged starch (DS) content. Table II presents the correlation coefficients (r), coefficients of determination (r^2), and the accompanying standard errors of prediction determined by correlating NIR DS values (derived using calibrations in Table I) and laboratory values. When the entire set of samples was analyzed, a correlation coefficient of 0.97 was calculated between the laboratory method for damaged starch determination and the NIR method. Hard and soft wheats as separate groups correlated with r values of 0.84 and 0.83, respectively.

Table III presents damaged starch values. The enzymatic method generally separated the hard and soft wheats into two distinct populations and ranged from 2.60 to 4.30% and from 4.02 to 8.20% for soft or hard wheats, respectively. The overlap was due to a low value (4.02) for Oslo, a HRS wheat, and a high value (4.30) for Hill 81, a western SWW wheat. The narrow range of percent damaged starch for the nine SRW wheats (2.84–3.65% by the laboratory method) lowered the r^2 value (0.40) compared to other subclasses of wheat. However, it is noteworthy that the SRW samples had a low standard error of estimate of 0.19% (Table II), indicating that the NIR predictive values were meaningful.

Effect of Milling Method

Using the enzymatic method, the DS was determined on cyclone-ground wheat and on flour milled by two milling methods. Data were adjusted to 14% moisture content for comparison. There are high correlations between the amounts of damaged starch generated by all three milling methods; however, QJ and AC millings both exhibited a much narrower range of damaged starch

TABLE III
Comparison of Enzymatic and Near-Infrared (NIR) Methods for Damaged Starch Determination on Cyclone-Ground Samples

Wheat Class[a]/ Method	Range	Mean	SD[b]	SD[c]
HRW				
Enzyme	4.44–6.29	5.70	0.12	···
NIR	4.74–6.14	5.63	0.27	0.17
HRS				
Enzyme	4.01–8.20	6.08	0.16	···
NIR	3.38–8.71	5.78	0.15	0.22
Club				
Enzyme	2.60–3.96	3.40	0.08	···
NIR	2.69–3.96	3.47	0.20	0.24
SRW				
Enzyme	2.87–3.65	3.20	0.13	···
NIR	2.85–3.56	3.12	0.08	0.19
ESWW				
Enzyme	2.74–3.61	3.20	0.14	···
NIR	2.79–3.53	3.11	0.10	0.24
WSWW				
Enzyme	3.00–4.30	3.81	0.07	···
NIR	3.32–4.14	3.75	0.13	0.22
Hards				
Enzyme	4.01–8.20	5.86	0.14	···
NIR	3.38–8.71	5.69	0.23	0.19
Softs				
Enzyme	2.60–4.30	3.39	0.11	···
NIR	2.69–4.14	3.56	0.13	0.20
All				
Enzyme	2.60–8.20	4.40	0.12	···
NIR	2.69–8.71	4.31	0.18	0.21

[a] HRW = Hard red winter, HRS = hard red spring, SRW = soft red winter, ESWW = eastern soft white winter, and WSWW = western soft white winter.

[b] Two determinations, one milling.

[c] Five millings, one determination for each.

TABLE IV
Comparison of Correlation Coefficients Relating Four Methods of Determining Damaged Starch

Methods[a] Compared	Correlation Coefficient (*r*)
Cyclone-Enzymatic vs. Cyclone-NIR	0.96
QJ-Enzymatic vs. Cyclone-NIR	0.91
AC-Enzymatic vs. Cyclone-NIR	0.94
Cyclone-Enzymatic vs. QJ-Enzymatic	0.94
Cyclone-Enzymatic vs. AC-Enzymatic	0.96
QJ-Enzymatic vs. AC-Enzymatic	0.95

[a] Cyclone = Udy cyclone grinder, QJ = Quadromat Junior mill, AC = Allis-Chalmers mill, NIR = near-infrared.

TABLE V
Correlation Coefficients Relating Wheat Hardness with Damaged Starch, Break Flour Yield, and Softness Equivalence[a]

		Correlation Coefficients for NIR Hardness vs.		
Sample	Entries	Damaged Starch (*r*)	Break Flour (*r*)	Softness Equivalent (*r*)
All wheats	72	0.92	−0.97	0.98
Hard	36	0.89	−0.96	0.96
Hard	25	0.58	−0.79	0.80
Soft	36	0.45	−0.71	0.66

[a] Hardness was determined by the near-infrared (NIR) method of the AACC (1983). Damaged starch was determined by the enzymatic method (Donelson and Yamazaki 1962) on Allis-Chalmers milled flours. Break flour yield was determined for the Allis-Chalmers mill. Softness equivalent was determined for Quadrumat Junior millings.

values than did cyclone-ground samples. The range of damaged starch for QJ flours was 1.53–4.51% for all classes of wheat, and the range for AC flours was 2.00–5.45%. The range of damaged starch for cyclone-ground samples was 2.60–8.20%.

There were high correlations between NIR and enzymatic damaged starch values for all milling methods (Table IV). The regression equations for such estimates are:

$$\text{QJ DS } (\%) = (0.583859 \times \text{NIR DS}) + 0.4398$$

$$\text{AC DS } (\%) = (0.731011 \times \text{NIR DS}) + 0.246234$$

The standard error of prediction for these equations was 0.369 and 0.246% DS for QJ and AC, respectively.

Test of Soft Wheat Calibration

Because of this laboratory's focus on soft wheat, a validation of the soft wheat calibration was performed. During the validation process it was found that the samples from the two nurseries (UERN and UWN) were best analyzed separately, either because they were two different populations (nurseries) or because one was red and the other white wheat. The bias and skew of the calibration was adjusted for the UERN by using the six-grind A UERN samples, and then adjusted for the UWN by using the UWN six-grind A samples. Percentage QJ- and AC-damaged starch were predicted from the adjusted calibration equations. Comparing the enzymatic and NIR determinations of damaged starch, the correlation coefficients for cyclone-ground enzymatic versus NIR, QJ enzymatic versus NIR, and AC enzymatic versus NIR were $r = 0.928$, $r = 0.945$, and $r = 0.931$, respectively, while the standard deviations for mean starch damage were 0.176, 0.206, and 0.233%, respectively. Thus, it is possible to predict the amount of damaged starch generated from a QJ or AC milling by using a 25-g cyclone-ground sample and the NIR method.

Effect of Hardness

On the calibration set of 72 wheats, there was a good correlation between AC break flour yield and the AACC NIR hardness method (Table V). NIR hardness also correlated highly with the QJ softness equivalence. When hardness was compared with DS, there were some noteworthy patterns. When all wheats were considered, there was a high correlation between hardness and DS. However, when the wheats were separated into soft or hard and the durums were excluded, the correlations materially dropped, indicating that although hardness affects the amount of DS generated, hardness and DS are different measurements (Table V).

CONCLUSION

NIR analysis of cyclone-ground wheat meals offers an alternative to the enzymatic methods currently in use for estimating DS. The correlation between the two methods is high on the sample set and the associated error limits are similar. The NIR method is more than five times faster than the enzymatic method. Two hundred samples can easily be analyzed by NIR in an 8-hr day after the bias and skew are appropriately adjusted. The enzymatic method requires substantial time to prepare reagents and about 36 samples can be analyzed in a day. In addition, when analyzing by NIR, information such as moisture, protein, and hardness can be obtained at the same time. Furthermore, it is possible to reasonably accurately predict the DS that would likely be generated by the AC and QJ mills under controlled conditions by analyzing a 25-g cyclone-ground sample with NIR.

Although the hardness of the wheat has an effect upon the quantity of DS generated, the two measurements are not the same when measured by NIR. Classification by NIR DS content shows promise as a method for separating hard and soft wheats. When QJ or AC flour was used, a better separation between hard and soft wheats was achieved, even though the range of DS was narrower for the more refined flours than for the cyclone-ground meals.

LITERATURE CITED

AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1983. Approved Methods of the AACC. Method 39-70, approved October 1986; Method 76-30A, revised October 1984. The Association: St Paul, MN.

DONELSON, J. R., and YAMAZAKI, W. T. 1962. Note on a rapid method for the estimation of damaged starch in soft wheat flours. Cereal Chem. 39:460-462.

FARRAND, E. A. 1964. Flour properties in relation to the modern bread processes in the United Kingdom, with special reference to alpha-amylase and starch damage. Cereal Chem. 41:98-111.

FINNEY, P. L., and ANDREWS, L. C. 1986. Revised microtesting for soft wheat quality evaluation. Cereal Chem. 63:177-182.

JONES, C. R. 1940. The production of mechanically damaged starch in milling as a governing factor in the diastatic activity of flour. Cereal Chem. 17:133-169.

NORRIS, K. H. 1964. Reports on design and development of a new moisture meter. Agric. Eng. 45:370-372.

OSBORNE, B. G., and DOUGLAS, S. 1981. Measurement of the degree of starch damage in flour by near infrared reflectance analysis J. Sci. Food Agric. 32:328-332.

PULKKI, L. H. 1938. Particle size in relation to flour characteristics and starch cells of wheat. Cereal Chem. 15:749-765.

SANDSTEDT, R. M., and MATTERN, P. J. 1960. Damaged starch quantitative determination in flour. Cereal Chem 37:379-390.

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.

WILLIAMS, P. C. 1979. Screening wheat for protein and hardness by near-infrared reflectance spectroscopy. Cereal Chem. 56:169-172.

WILLIAMS, P. C. 1986. Attempts at standardization of hardness testing of wheat. II. The near-infrared reflectance method. Cereal Foods World 31:417-420.

WILLIAMS, P. C., and LESEELIEUR, G. C. 1970. Determination of damaged starch in flour: A comparative study of present-day procedures. Cereal Sci. Today 15:4-19.

YAMAZAKI, W. T., and ANDREWS, L. C. 1982. Experimental milling of soft wheat cultivars and breeding lines. Cereal Chem. 59:41-45.

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