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Abstract

A corn density calibration was developed for a near-infrared transmittance instrument (Infratec 1225). The calibration sample set included 96 corn samples grown in various locations of the United States. Samples were selected by principal component analysis (PCA) from a larger set of 410 samples, representing the 1986 through 1992 crop years. Samples for instrument and temperature stabilization were included in the calibration. The partial least squares calibration was then validated with 35 randomly selected samples not in the calibration sample set. With 14 PCA factors, the standard error of calibration was 0.0173 g/cm3, and the standard error of prediction was 0.0164 g/cm3. Fourteen factors were required because the first 12 reflected the physical correlations of density to protein and density to starch.

Keywords

Near-infrared transmittance, Corn, Density

Disciplines

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Comments

CORN DENSITY MEASUREMENT BY NEAR-INFRARED TRANSMITTANCE

J. Siska, C. R. Hurburgh Jr.

ABSTRACT. *A corn density calibration was developed for a near-infrared transmittance instrument (Infratec 1225). The calibration sample set included 96 corn samples grown in various locations of the United States. Samples were selected by principal component analysis (PCA) from a larger set of 410 samples, representing the 1986 through 1992 crop years. Samples for instrument and temperature stabilization were included in the calibration. The partial least squares calibration was then validated with 35 randomly selected samples not in the calibration sample set. With 14 PCA factors, the standard error of calibration was 0.0173 g/cm³, and the standard error of prediction was 0.0164 g/cm³. Fourteen factors were required because the first 12 reflected the physical correlations of density to protein and density to starch.*

*This calibration gives the Infratec analyzer capability to measure moisture, protein, oil, starch, and density simultaneously on an unground sample. **Keywords.** Near-infrared transmittance, Corn, Density.*

Corn hardness is an important corn characteristic, often defined as the ratio between the mass of hard endosperm and the total endosperm mass (Watson, 1987). Hardness is most important to dry-milling industries that produce large flaking grits to make breakfast cereals, snack foods, etc. The harder the corn, the greater the yield of flaking grits. A hard corn produces 40 to 50¢/bushel more end product values in these uses (Hurburgh, 1989).

Several indirect methods have been designed to test corn hardness. Usually, direct measurements are slow and expensive compared with indirect methods. Direct measurements are dissection, magnetic resonance imaging, and milling test. Among the most widely used indirect methods are measurement of floaters, kernel density, and near-infrared reflectance.

The corn-float test (Wichser, 1961) compares the specific gravity of corn kernels by placing them in a solution of known specific gravity. Accuracy of this measurement depends upon the specific gravity of the solution. The long time required to perform the test (more than 20 min.) does not satisfy the requirements of current end-users for rapidity, and the corn is destroyed.

Pycnometer density provides a precise and nondestructive measurement of corn hardness. Density is obtained by measuring the air volume displaced by a measured weight of corn. Thompson and Isaacs (1967)

have related pycnometer density to product yield in dry milling, as have recent works at the USDA-Northern Regional Laboratory (Wu and Berquist, 1991).

Near-infrared spectroscopy enables quick, inexpensive, and precise measurement of corn-grain constituents. Near-infrared reflectance (NIR) is commonly used for analyzing ground-grain samples (Hurburgh, 1988). Whole seed near-infrared transmittance (NIT) has the advantage, compared to NIR, of being nondestructive. Avoiding sample grinding increases measurement repeatability and sample preservation.

Several attempts have been made to measure wheat and corn hardness by NIR, based on particle-size differences between various classes of ground wheat (Yamazaki, 1972). Williams and Sobering (1986b) established a tentative near-infrared index for identifying wheat on the basis of hardness. A particle size index (PSI) (Williams and Sobering, 1986a) was then developed to measure hardness. Delwiche and Norris (1993) used principal component analysis (PCA) to develop models for distinguishing two classes of wheat, hard red winter and hard red spring, with only moderate success. Williams (unpublished data) developed a calibration for PSI on whole-grain hard red spring wheat on the Infratec model 1225, an NIT analyzer. All these methods used particle size distribution as a reference method for calibration of near-infrared instruments.

Pomeranz et al. (1984) reported correlations of corn density (r = 0.75 for commercially dried hybrids) with NIR reflectance. In the Pomeranz study, there was high correlation between average particle size (APS) ground material and corn density, as determined by an air-comparison pycnometer.

Although extensive research has determined wheat and corn hardness by near-infrared reflectance technology, there are no data on corn hardness determination by whole-grain NIT spectroscopy. A hardness measurement would be a valuable addition to the established capabilities of NIT to measure composition.

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OBJECTIVE

The objective of this study was to predict corn air-comparison pycnometer density with a NIT instrument.

MATERIALS AND METHODS

SAMPLES

The corn samples for this study were selected from the calibration set used by the Grain Quality Laboratory at Iowa State University, Ames, for moisture content and chemical composition calibration. Stored samples (refrigerated at 2° C) represented 1986 through 1992 crop years. Sample composition properties as determined by wet chemistry methods (Woodson-Tenent Labs, Inc., Des Moines, Iowa), oven moistures, and density values determined by air-comparison pycnometer at Iowa State are shown in table 1.

KERNEL DENSITY

Corn density (g/cm^3) was determined by dividing kernel mass (g) by the kernel volume (cm^3) determined by a Beckman model 930 air-comparison pycnometer (Beckman Instruments, Inc., Fullerton, Calif.). Procedures

Table 1. Composition and density parameters for the total sample set

Year	Moisture (%)	Protein* (%)	Oil* (%)	Starch* (%)	Density* (g/cm^3)
1986					
n†	2	2	2	2	2
Mean	12.9	8.1	3.6	60.1	1.275
Range	12.8-13.1	8.1-8.2	3.4-3.7	59.0-61.3	1.253-1.296
1987					
n	1	1	1	1	–
Mean	13.0	7.9	3.6	60.3	–
Range	–	–	–	–	–
1988					
n	7	7	7	7	3
Mean	12.6	9.8	3.5	58.6	1.241
Range	8.2-14.0	8.0-11.8	3.3-3.8	56.2-60.2	1.230-1.256
1989					
n	27	27	27	27	27
Mean	12.6	7.9	3.6	61.1	1.282
Range	10.0-15.4	6.9-9.9	2.9-4.3	58.8-62.7	1.242-1.320
1990					
n	46	46	46	46	46
Mean	10.6	7.9	3.5	61.1	1.267
Range	3.5-14.2	6.7-9.9	3.0-4.1	56.7-62.8	1.214-1.301
1991					
n	85	85	85	85	85
Mean	10.3	8.4	3.6	59.7	1.282
Range	5.0-13.1	5.3-11.9	3.0-5.9	54.7-63.7	1.209-1.323
1992					
n	242	101	101	101	242
Mean	20.4	8.4	3.5	60.4	1.286
Range	6.5-45.4	5.5-13.0	2.8-5.4	55.0-65.3	1.200-1.369
1986-1992					
n	410	269	269	269	405
Mean	16.5	8.3	3.5	60.3	1.282
Range	3.5-45.4	5.3-13.0	2.8-5.9	54.7-65.3	1.200-1.369

* Values converted to 15% moisture content.
† n = number of samples.

for using the air-comparison pycnometer were described by Thompson and Isaacs (1967).

Each corn sample was tested twice. If the difference between two replicates was more than two times the previously established standard deviation of measurement (0.003 g/cm^3), a third replication was made. Air-oven moisture was determined for each sample (AACC, 1991). Density was converted to 15% moisture content by the following equation (Dorsey-Redding et al., 1990):

$$D_f = D_i - 0.00289(M_f - M_i) \qquad (1)$$

where

D_f = final density (g/cm^3)
D_i = initial density (g/cm^3)
M_f = final moisture content (%)
M_i = initial moisture content (%)

The instrument used for near-infrared absorbance collection was an Infratec 1225 Grain Analyzer (Tecator A/B, Höganäs, Sweden, 1987), which has a scanning monochromator generating a wavelength range of 850 to 1050 nm. This is the same instrument approved by Federal Grain Inspection Service for soybeans (FGIS, 1989) and wheat (FGIS, 1993). Absorbance data was stored on an internal disk drive. Samples were presented in the factory sample transport cuvette, which held about 150 g of corn, with a pathlength of 30 mm. Ten subsamples scans were averaged per 150-g sample.

STATISTICAL ANALYSIS

From the total set, 35 samples were randomly selected over all year data for the validation set. These samples were not included in the calibration. Principal component analysis (PCA) (Mulaik, 1972) was used to identify major variations of the sample absorbance spectra. The calibration sample set was then selected from the total set by examining the influence of each sample on the model (leverage) and by studying how well the samples fit the model (variance). Individual samples with high influence and high subsample spectral variation were eliminated from the calibration as being atypical of the population. This is the procedure recommended by the manufacturer and published by the author of the calibration software (Martens and Naes, 1987, 1989). The PCA procedure characterizes the data with a set of factors (linear multiplicative combinations of independent variables) that are not correlated with each other. Each sample has a score in each factor, created by solving the PCA equations. Samples with large scores in a particular factor have large, disproportionate influence on the population variance. Samples with similar factor scores are spectrally similar. Therefore, calibration sample selection involves spreading samples across the range of factor scores without choosing samples that have either high influence or that have unusual chemical properties for their factor scores.

The total sample set (410 samples) was also used for the selection of samples for protein, oil, and starch calibrations (Brumm, 1993). The moisture percentage, constituent properties, and density values are listed in table 2.

The Partial Least Squares (PLS) method with orthogonal (not intercorrelated) factors (Martens and Naes, 1987) was used to establish the calibration model. In PLS, factors are linear combinations of the wavelength

Table 2. Composition and density parameters for the calibration and validation sets

Set	Moisture (%)	Protein* (%)	Oil* (%)	Starch* (%)	Density* (g/cm³)
Calibration 1987					
n†	96	68	68	68	96
Mean	16.1	8.4	3.6	60.2	1.283
Range	3.5-45.4	5.3-13.0	2.9-5.4	55.0-64.5	1.214-1.369
Validation 1989					
n	35	31	31	31	35
Mean	13.2	8.5	3.6	59.9	1.275
Range	5.1-29.9	6.7-11.2	0.9-4.2	56.0-62.5	1.217-1.319

* Values converted to 15% moisture content.
† n = number of samples.

Table 3. Evaluation of Infratec corn calibrations

Calibration Property*	No. of Factors	Calibration Set SEC† (%)	Validation Set SEP‡ (%)	Validation Set r	Validation Set Bias (%)
Density (g/cm³)	14	0.0173	0.0164	0.76	0.002
Moisture§ (%)	10	0.74	0.48	1.00	0.03
Protein§ (%)	11	0.44	0.36	0.98	−0.10
Oil§ (%)	12	0.24	0.21	0.91	0.05
Starch§ (%)	12	1.36	1.01	0.95	−0.16

* Converted to 15% moisture content.
† Standard error of calibration.
‡ Standard error of prediction.
§ Developed on this sample set as part of the other research. Included for comparison purposes.

absorbances and reference data. This method can be divided into two steps: (1) calculation of the calibration factors and (2) prediction. The program "Unscrambler" (CAMO A/S, 1987) was used.

Evaluation. The following statistical parameters were used to analyze the success of the calibration:

Bias = average deviation from the reference value
SEP = standard error of prediction, adjusted for bias
r = Pearson correlation coefficient

TEMPERATURE AND INSTRUMENT STABILIZATION

Two types of stability were included in the calibration to compensate for temperature and instrument spectral differences.

The calibration contained scans taken at different temperatures. Three groups of eight samples were run at three different sample temperatures: cold (3° C), warm (43° C), and room (21° C) temperature. This procedure resulted in these eight samples being included three times each.

Each instrument has two spectral-shaped parameters (called "O" and "P" by the company) that theoretically provide for uniform spectral performance. The O and P values are the full-spectrum bias and slope constants applied to adjust spectral data to equivalent data on a single master unit, through the procedure recommended by Tecator for use with the Unscrambler software (Camo A/S, 1987). The O and P values are unique to each instrument and are keyboard entered. To simulate instrument spectral variation (such as a new light source or a new detector) in the calibration, 2 groups of 10 samples each were scanned at slightly different values of O and P constants. These samples were mandatorily included in the calibration set, in addition to scans of the same samples taken at the original values of the instrument constants. Parameters for the temperature and instrument stabilization sets are listed in table 3. The instrument stabilization procedure has the practical effect of absorbing some of the unavoidable instrument-to-instrument spectral variations in the base calibration, thus reducing pressure on calibration transfer.

RESULTS AND DISCUSSION

Calibration and validation statistics are summarized in table 3. The statistics for composition were derived as part of other research and are included only for comparison

purposes. However, a critical element of NIRS calibration is robustness in all properties that affect spectra, not just the one of immediate interest. Including stabilization samples into the calibration set resulted in the higher value of standard error of calibration (SEC) (0.0173 g/cm³) for the calibration set than standard error of prediction (SEP) (0.0164 g/cm³) for the validation set. These represented the best models with the optimum number of factors.

Explained statistical variance for density, as a function of number of factors, is shown in figure 1. The contribution of each additional factor's explanation of both calibration variance, c_T, and validation variance, v_T, decreases as the number of factors increases. Validation variance, v_T, was obtained by the leverage correction method (Martens and Naes, 1987) on the calibration sample set. The decision concerning the number of factors used in PLS was based on figure 2, which shows the SEP for the validation set as a function of the number of factors.

The SEP decreased sharply with the first six factors, reaching a minimum after 14 factors. Factors over 14 were attempting to fit unique characteristics of the sample set, at the expense of overall accuracy. The values of predicted versus measured density for the validation sample set are plotted in figure 3.

There was statistically significant correlation among protein, starch, and density values for total, calibration, and validation sample sets, even though these sets were chosen to have as much uniformity as possible across the range of all constituents. Density-composition correlations were reflected in the b-vectors (regression coefficients) for the density protein and starch calibrations. The protein and
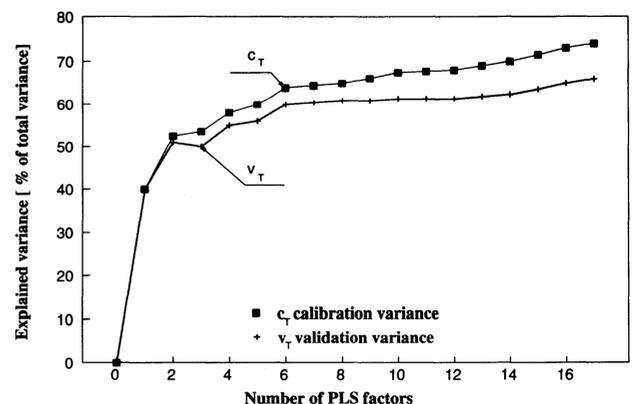

Figure 1–Explained corn density variance for the calibration sample set.
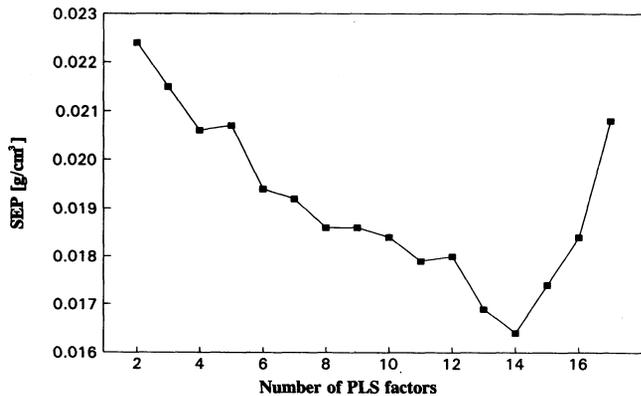
Figure 2–Standard error of corn density prediction (SEP) for the validation sample set, as a function of number of factors.

starch calibrations were developed by Brumm (1993) using 11 and 12 factors, respectively. The density calibrations required 14 factors to first remove the correlations with composition, then to identify the unique contribution of material density to absorbance.

The density calibration represents a major addition to near-infrared capabilities. At least five constituents important to feed, wet milling, and dry milling can now be measured in a single operation.

CONCLUSIONS

A calibration of the Infratec 1225 NIT grain analyzer for corn density measurement was developed. The PCA was used to select a set of 96 calibration samples from 410 samples (from 1986 to 1992 crops) with spread spectra. The PLS method produced a 14 factor calibration with calibration and prediction standard errors of 0.0173 g/cm^3 and 0.0164 g/cm^3, respectively, and with calibration r^2 of 76%.

Corn protein and starch contents had important impacts on the calibration for corn density measurement because they were positively and negatively correlated with density, respectively. The calibration required 14 factors to identify the unique density information beyond the cross-correlation.

The ability to measure density is an important addition to near-infrared capabilities because density is indicative of
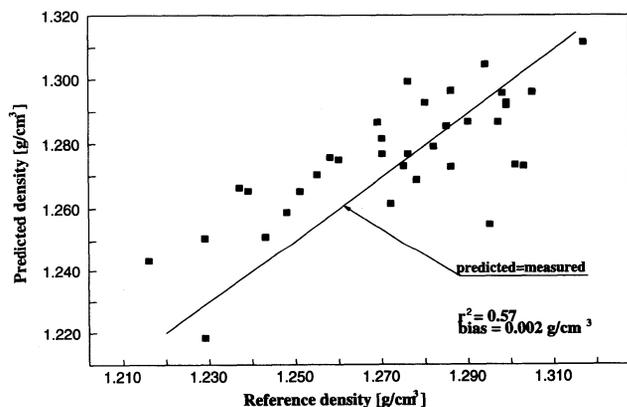

Figure 3–Predicted vs. measured corn density for the validation sample set (n = 35).

product yields in dry corn milling. This capability adds to its proven ability to measure compositional factors.

REFERENCES

AACC. 1991. Moisture-air oven methods. AACC Method 44-15A. In *Approved Methods of the American Assoc. of Cereal Chemists,* Vol. II. Minneapolis, Minn.: AACC.

Brumm, T. J. 1993. Private communication on composition calibration. Ames, Iowa: MBS.

FGIS. 1989. Certificate of Conformance. Certificate No. 89-101. Kansas City, Mo.: Quality Assurance and Research Div., Testing and Instrument Service Branch, Federal Grain Inspection Service, USDA.

————. 1993. Near-infrared transmittance (NIT) protein testing services. In *Program Bulletin.* Washington, D.C.: Field Management Div., Federal Grain Inspection Service, USDA.

CAMO A/S. 1987. *Unscrambler Program Package. Infratec Calibration Maker,* Ver. 2.3. Camo, Norway: AB.

Delwiche, S. R. and K. H. Norris. 1993. Classification of hard red wheat by near-infrared diffuse reflectance spectroscopy. *Cereal Chemistry* 70(1):29-35.

Dorsey-Redding, C., C. R. Hurburgh Jr., L. A. Johnson and S. R. Fox. 1990. Adjustment of maize quality data for moisture content. *Cereal Chemistry* 67(3):292-295.

Dutta, P. K. 1986. Effects of moisture, drying methods, and variety on breakage susceptibility of shelled corn as measured by the Wisconsin Breakage Tester. Ph.D. diss., Iowa State Univ., Ames.

Hurburgh Jr., C. R. 1988. Moisture and composition analysis in the corn and soybean market. *Cereal Foods World* 33(6):503-505.

————. 1989. The value of quality to new and existing corn uses. ASAE Paper No. 89-6016. St. Joseph, Mich.: ASAE.

Martens, H. and T. Naes. 1987. Multivariate calibration by data compression. In Near-infrared Technology in the Agricultural and Food Industries, 57-87, eds. P. Williams and K. Norris. St. Paul, Minn.: AACC.

————. 1989. *Multivariate Calibration.* London, U.K.: John Wiley & Sons Ltd.

Mulaik, S. A. 1972. Principal component analysis. In *The Foundations of Factor Analysis,* 177-179. New York.: McGraw-Hill Book Co.

Pomeranz, Y., C. R. Martin, D. D. Taylor and F. S. Lai. 1984. Corn hardness determination. *Cereal Chemistry* 61(1):147-150.

Thompson, R. A. and G. W. Isaacs. 1967. Porosity determinations of grains and seeds with an air-comparison pycnometer. *Transactions of the ASAE* 10(7):693-696.

Watson, S. A. 1987. Measurement and maintenance of quality. In *Corn Chemistry and Technology.* St. Paul, Minn.: AACC Press.

Wichser, W. R. 1961. The world of corn processing. *Am. Miller and Processor* 89(1):29.

Williams, P. C. and D. C. Sobering. 1986a. Attempts at standardization of hardness testing of wheat. I. The grinding/sieving (particle size index) method. *Cereal Foods World* 31(5):359-364.

————. 1986b. Attempts at standardization of hardness testing of wheat. II. The near-infrared reflectance method. *Cereal Foods World* 31(6):417-420.

Williams, P. C. 1987. Commercial near-infrared reflectance analyzers. In *Near-infrared Technology in the Agricultural and Food Industries,* 107-142, eds. P. Williams and K. Norris. St. Paul, Minn.: AACC.

Williams, P. C. and K. H. Norris. 1987. Qualitative applications of near-infrared reflectance spectroscopy. In *Near-infrared Technology in the Agricultural and Food Industries,* 241-246, eds. P. Williams and K. Norris. St. Paul, Minn.: AACC.

Wu, Y. V. and R. R. Berquist. 1991. Relation of corn density to yields of dry milling products. *Cereal Chemistry* 68(5):542.

Yamazaki, W. T. 1972. A modified particle-size index test for kernel texture in soft wheat. *Crop Sci.* 12(1):116.