# Prediction of Corn Dry-Milling Quality by Near-Infrared Spectroscopy[1]

R. L. WEHLING,[2,3] D. S. JACKSON,[2] and B. R. HAMAKER[4]

**ABSTRACT**

Cereal Chem. 73(5):543–546

The potential for near-infrared (NIR) reflectance spectroscopy to predict corn dry-milling quality has been investigated. NIR spectra were obtained from samples of whole-kernel yellow dent corn. Dry-milling quality of the samples was evaluated with a Tangential Abrasive Dehulling Device (TADD), and by a short-flow laboratory milling procedure that allowed calculation of a milling evaluation factor (MEF). Calibrations relating TADD index and MEF to NIR measurements were developed and tested with validation sample sets. The results indicate that NIR spectroscopy can predict dry-milling characteristics with a reliability suitable for at least rough screening. For MEF, the best calibration yielded a correlation coefficient of 0.90 and a standard error of prediction (SEP) of 1.55%. SEP values for TADD index were somewhat higher. Derivatization of the NIR spectra lowered the SEP values compared to the use of underivatized log 1/*R* measurements.

The availability of a rapid test for predicting the dry-milling characteristics of corn would be highly useful in a breeding program for selecting cultivars with good milling qualities, and for identifying corn shipments in commerce that have good industrial processing characteristics. Several researchers have investigated the relationships of various chemical and physical tests to dry-milling yields. In general, to produce a high yield of the desired No. 4 flaking grits, corn with a high ratio of vitreous to floury endosperm is needed. Paulsen and Hill (1985) found that corn with a high test weight and low breakage susceptibility provided the best yield of flaking grits from a commercial dry mill. In addition to test weight, other measures of density including 100 kernel weight and % floaters, as determined in a solution with specific gravity of 1.275, were also highly correlated with the yield of grits. Kirleis and Stroshine (1990) reported that milling evaluation factor (MEF) values, determined with a short flow laboratory milling procedure, were highly correlated ($r = 0.92$) with flaking grits yield obtained on a commercial corn dry mill. Kirleis and Stroshine (1990) then determined the milling quality of three dent hybrids exposed to different drying conditions using MEF values, and they also measured various physical properties of the samples. For the samples evaluated, a two-variable regression model combining test weight and kernel density was highly correlated with MEF values ($R^2 = 0.907$). Similarly, for 15 different samples Wu and Bergquist (1991) found a significant correlation ($r = 0.894$) between kernel density measured with an air comparison pycnometer and total grits obtained by a laboratory milling procedure. In another study, Peplinski et al (1992) reported that first break grits yield was higher for corn with high test weight, low % floaters and a low percentage of broken kernels as measured by the Stein breakage test. Hill et al (1991) reported a study with a larger number of samples (98) in which several of the more complex physical and chemical tests had correlations >0.75 with the MEF. The best predictors of MEF were Stenvert time to grind, % oil, % floaters, and % flint genetic background in the cultivar. In general, higher correlations between the physical tests and MEF were obtained for flint samples than for dent samples.

Near-infrared (NIR) spectroscopy has become widely used in the grain trade and processing industries for determining the composition of corn and other cereals. The ability of NIR spectroscopy to measure other quality characteristics of corn simultaneously with chemical composition has been investigated and can provide a rapid and simple means for quality evaluation. Pomeranz et al (1984, 1986) used reflectance from ground samples at 1,680 nm to measure kernel hardness. Siska and Hurburgh (1994) included NIR measurements of protein, oil, and starch contents in a multiple linear regression equation to predict Wisconsin Tester breakage susceptibility. Wehling et al (1993) used NIR reflectance measurements from whole kernel corn to directly estimate starch yields obtainable by wet milling.

The objective of the current research was to investigate the feasibility of using NIR spectroscopy to directly predict the dry-milling characteristics of yellow dent corn. Development of a successful NIR protocol could provide a rapid and relatively simple means of estimating corn dry-milling quality. Correlations between NIR reflectance measurements and Tangential Abrasive Dehulling Device (TADD) data, as well as between NIR and the MEF obtained from a short flow laboratory milling procedure, were evaluated.

## MATERIALS AND METHODS

### Samples

Eight different genetic lines of yellow dent corn, representing both commercial cultivars and experimental crosses, were grown at five different geographic locations in Nebraska during the 1990 crop year. The lines were representative of the range of kernel hardness found in hybrids commonly grown in the Midwestern United States. Three of the lines were also grown at three locations during the 1991 crop year. All experimental plots were irrigated.

At the end of the growing season, the corn was allowed to air dry in the field before harvest. After mechanical harvesting and field shelling, large pieces of cob or other debris in the bulk samples were removed by hand. Samples were stored at ambient temperature (15–25°C) until needed for analysis.

### Tangential Abrasive Dehulling

Corn (40 g) was placed into each of eight sample cups of a Tangential Abrasive Dehulling Device (model 4E-220, Venables Machine Works, Saskatoon, SK). The corn was abraded for 10 min while the material removed from the corn was simultaneously suctioned off. The remaining material from each sample was weighed and a TADD index calculated as: TADD index (%) = ([initial wt — wt of material remaining]/initial wt) × 100.

[1]Published as paper 11275, Journal Series, Agricultural Research Division, University of Nebraska, Lincoln, NE 68583-0704.

[2]Department of Food Science and Technology, 143 Filley Hall, University of Nebraska, Lincoln, NE 68583-0919.
[3]Corresponding author. E-mail: rwehling@foodsci.unl.edu
[4]Food Science Department, 1160 Smith Hall, Purdue University, West Lafayette, IN 47907-1160.

Publication no. C-1996-0801-06R.

Results reported are the average of triplicate determinations for samples from both the 1990 and 1991 crop years.

**Milling Evaluation Factor**

A short-flow corn dry-milling procedure, previously described by Kirleis and Stroshine (1990) and Hill et al (1991), was used to determine the MEF. MEF is a numerical index reflecting grit and total endosperm yields. Before milling, ≈1,000 g of corn were conditioned to 18% moisture content for 1 hr, and then to 20% moisture content for 15 min. Tempered samples were passed through a horizontal drum-type degermer operated at 2,150 rpm at a feed rate of about 450 g/min. Stock from the degermer was screened for 30 sec over a 3-1/2 W sieve on a Smico laboratory test sifter. Stock remaining over the sieve was given a second pass through the degermer at a speed of 2,250 rpm and combined with the stock that passed through the sieve. Combined stocks were dried for 1 hr at 45°C to a moisture content of 17 ± 1%. Dried stock was separated by screening over 3-1/2, 5, 7, 10, and 16 W sieves on a Smico laboratory test sifter for 1 min. Fractions remaining over each sieve were aspirated on a Bates laboratory aspirator to remove hull material. After aspiration, the 5, 7, and 10 W overs were floated in sodium nitrate solution (1.275 specific gravity) to separate germ and endosperm pieces. All fractions were dried for 16–18 hr at 45°C and weighed. MEF was calculated as:

$$MEF = (EN_{3\text{-}1/2W} + EN_{5W} + EN_{7W})(TEP/100)$$

where: EN = weight percentage of endosperm remaining on the screen identified by the subscript and TEP = weight percentage of total sample recovered in all endosperm products. MEF was determined for 1990 samples only. Results are the average of duplicate determinations.

**Near-Infrared Spectroscopy**

A scanning spectrometer (model 6500, NIRSystems Division of Perstorp Analytical, Silver Spring, MD) was used to obtain spectra of the corn samples. For instrument control and calibration development, the spectrometer was interfaced to an MS-DOS personal computer running the Near Infrared Spectral Analysis Software (NSAS) package (version 3.16) provided by NIRSystems.

Diffuse reflectance spectra in a log $1/R$ format were obtained from whole-kernel samples over a spectral range of 400–2,500 nm. All spectra were collected at 2-nm intervals. Spectra were obtained using an NIRSystems coarse sample cell and sample transport module. Each sample's spectrum represented 32 individual spectral scans collected and averaged over the length of the filled sample cell as it was moved through the infrared beam by the transport mechanism. The total sampling area was ≈80 cm$^2$.

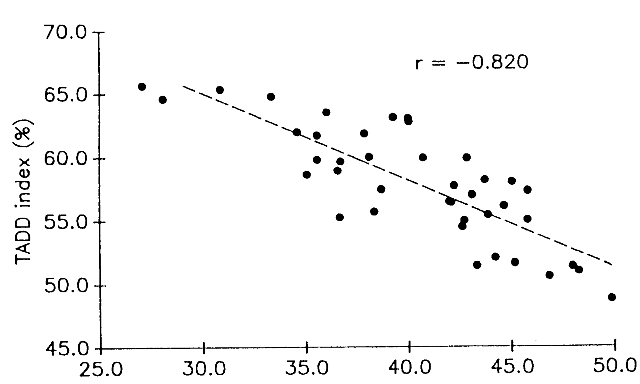


**Fig. 1.** Relationship of milling evaluation factor (MEF) and Tangential Abrasive Dehulling Device (TADD) index (%) for the 40 samples grown and harvested in the 1990 crop year.

Thirty samples from the 1990 crop year were selected for calibration development. The 10 remaining 1990 crop year samples comprised the validation set for MEF calibrations. These ten 1990 samples, plus the nine 1991 crop year samples, were included in the validation set for TADD index.

Multiterm linear regression (MLR) equations relating log $1/R$ values to TADD index and MEF were developed using forward stepwise and best possible regressions algorithms. The best possible regressions algorithm allowed a maximum of 70 wavelengths, evenly spaced across the near-infrared region, to be considered for regression. Multiterm equations using first and second derivative transformations of the spectral data were also developed. The optimum number of wavelengths for inclusion in the calibration equations was determined by comparing regression results for multiple correlation coefficients ($R$), standard error of calibration (SEC), partial $F$-values for each term in the equation, and the overall $F$ of regression (Workman and Mark 1992). Additionally, these statistical parameters were used to select the optimum segment and gap values for calculating the derivatives (Williams 1987).

A partial least squares (PLS) algorithm was also used to develop calibration equations from log $1/R$ and derivatized data to determine whether this technique could provide results that were superior to those obtained using calibrations developed by multiple linear regression. The use of PLS algorithms allows information from all available wavelengths to be included in the calibration rather than information from only a few wavelengths. In some cases, PLS calibrations have been found to provide prediction results superior to those obtained from MLR equations. Underivatized spectra were scaled to a mean of zero and variance of one before PLS regression. The optimum number of terms for inclusion in a PLS calibration equation was selected based on the standard errors of cross validation, which should be minimized (Workman and Mark 1992), along with the $R$ and SEC values obtained from the regressions.

The standard error of prediction (SEP) (Workman and Mark 1992) of each calibration equation was determined by predicting the TADD index or MEF for each sample in the validation set.

## RESULTS AND DISCUSSION

The MEF was selected as a measure of dry-milling quality because of its reported high correlation ($r$ = 0.92) with flaking grits yield obtained on a commercial corn dry mill (Kirleis and Stroshine 1990). The standard error of the laboratory dry-milling procedure was calculated from duplicate millings of the 40 samples collected during the 1990 crop year, using the equation described by Workman and Mark (1992). The absolute standard error of the procedure was 0.76%. The set of 30 samples in the calibration set had a mean MEF value of 40.28% and a standard deviation of 5.94%. For the 10 samples in the validation set, the mean and standard deviation were 40.26 and 3.14%, respectively.

TADD index was also selected as a measure of milling quality because it depends on kernel hardness (Lawton and Faubion 1989), an important factor that influences grits yield. The relationship between MEF and TADD for the 40 corn samples from the 1990 crop year is shown in Figure 1. An inverse relationship ($r$ = –0.820) between the two parameters is evident, as a high TADD index is indicative of a softer kernel and lower yields of flaking and brewer's grits. The calibration set had a mean TADD index of 58.15% and a standard deviation of 4.88%. The 19 member validation set had mean and standard deviation values of 57.63 and 4.41%, respectively. The standard error of the laboratory method calculated from the 40 1990 crop year samples was 1.19%.

Results for the prediction of MEF by NIR spectroscopy are shown in Table I. The best calibrations from each combination of regression type and spectral treatment yielded similar calibration statistics ($R$ = 0.85–0.87). However, there were substantial differences in the performance of the calibrations when applied to the validation sample set. The use of derivatives lowered the SEP compared to the values obtained when log 1/*R* measurements were used directly (Table I). Derivatization eliminates the sample-to-sample baseline shifts observed in the log 1/*R* spectra. Calibrations developed by PLS regression provided no substantial improvement in results compared to those developed by multiple linear regression (Table I). In fact, when using derivatized data, PLS calibrations did not perform as well as the simple linear combination of terms obtained by MLR.

Prediction results for TADD index obtained from the validation sample set are given in Table II. Standard errors have been bias corrected as described by Osborne et al (1993) because a small bias was noted between the 1990 and 1991 crop years. The overall SEP representing both crop years was calculated with the bias correction included and therefore characterizes the random error, but not the systematic or constant error, of the NIR method (Workman and Mark 1992). Again, derivatization was found to lower the SEP, with a linear combination of first derivative terms providing the lowest SEP values over both crop years (Table II). The use of PLS regression did not improve the results, as PLS models yielded SEP values for the 1991 crop year that were substantially greater than those obtained from calibrations developed by MLR (Table II). The PLS algorithm apparently included spectral variations in the model that were unique to the 1990 samples, resulting in overfitting. Calibrations developed using MLR had more similar SEP values for the 1990 and 1991 samples, requiring only a slight bias adjustment between the two crop years. SEP values were larger for TADD index than for the MEF. Correlation coefficients between NIR and laboratory measurements for the validation samples were also slightly lower for the TADD index than for the MEF prediction ($r$ = 0.87 and 0.90 for TADD index and MEF, respectively, using the calibrations derived by MLR of first derivative terms).

For measurement of both MEF and TADD index, wavelengths in the 1,100–1,175 nm region were selected by multiple linear regression as the primary indicator wavelengths, as evidenced by large partial $F$-values. A large partial $F$-value obtained upon addition of a wavelength to a regression equation indicates a significant change in the equation's ability to model the data (Workman and Mark 1992). When using first derivative terms, 1,134 nm appears in regression equations for both TADD index and MEF, apparently indicating that the same spectral variation is related to both measurements. Since the use of derivatives, which remove baseline shifts attributable to scattering differences, improved the prediction results, it appears that MEF and TADD index are not simply related to sample-to-sample changes in bulk reflectance. More likely, MEF and TADD index are being related to changes in specific absorption bands. The wavelengths in the 1,110–1,175 nm region are on the shoulder of an absorption band centered at ≈1,200 nm. Absorptions in this region are primarily -CH stretching and -OH combination bands (Osborne et al 1993), and arise

**TABLE I**
**Prediction Results for Milling Evaluation Factor (MEF) Obtained from the Validation Sample Set Using Various Calibrations**

| Regression Type[a] | Spectral Treatment | Wavelength or Number of PLS Terms Used | Correlation Coefficient (r) | Standard Error of Prediction (%) |
|---|---|---|---|---|
| MLR | log 1/*R* | 1,110 nm<br>1,282 nm<br>1,340 nm | 0.88 | 2.34 |
| MLR | 1st derivative[b] | 1,134 nm<br>1,202 nm<br>1,584 nm | 0.90 | 1.55 |
| MLR | 2nd derivative[c] | 1,172 nm<br>1,794 nm | 0.92 | 1.79 |
| PLS | log 1/*R* | 4 factors[d] | 0.90 | 2.28 |
| PLS | 1st derivative[b] | 3 factors[e] | 0.91 | 2.07 |
| PLS | 2nd derivative[c] | 3 factors[f] | 0.91 | 2.06 |

[a] MLR = multiple linear regression, PLS = partial least squares regression.
[b] Segment = 5 points, gap = 5 points.
[c] Segment = 5 points, gap = 5 points.
[d] Spectral range = 1,100–1,500 nm.
[e] Spectral range = 1,100–1,600 nm.
[f] Spectral range = 1,100–1,500 nm.

**TABLE II**
**Standard Errors of Prediction (SEP) Obtained for TADD[a] Index from the Validation Set of 1990 and 1991 Crop Year Samples**

| Regression Type | Spectral Treatment | Wavelength or Number of PLS Terms Used | SEP (%) | | |
|---|---|---|---|---|---|
| | | | Overall | 1990 | 1991 |
| MLR | log 1/*R* | 1,114 nm<br>1,176 nm<br>1,226 nm<br>1,402 nm | 2.84 | 3.08 | 2.72 |
| PLS | log 1/*R* | 7 factors[d] | 3.46 | 2.78 | 4.24 |
| MLR | 1st derivative[e] | 1,134 nm<br>1,204 nm<br>1,658 nm | 2.28 | 2.23 | 2.47 |
| MLR | 2nd derivative[f] | 1,170 nm<br>1,690 nm | 2.61 | 2.87 | 2.45 |

[a] TADD = Tangential Abrasive Dehulling Device.
[b] MLR = multiple linear regression, PLS = partial least squares regression.
[c] 1991 samples were bias-corrected. Overall SEP included this correction for systematic error.
[d] Spectral range = 1,100–1,500 nm.
[e] Segment = 10 points, gap = 15 points.
[f] Segment = 20 points, gap = 15 points.

from the carbohydrate, protein, and lipid components in the sample. It is thought that grain hardness is related to the strength of interactions between the protein and starch fractions (Hoseney 1986), and it may be that variation in the strength of these interactions leads to spectral differences in this wavelength region. Further studies are needed to confirm these preliminary observations.

In summary, our research has shown that near-infrared spectroscopy has the ability to predict dry-milling quality of dent corn with at least sufficient reliability to be used as a tool for rough screening. NIR spectroscopy allows this measurement to be made rapidly and conveniently. Calibration sets with a larger number of samples, grown over multiple crop years and with a wide geographical distribution, are now needed to establish calibrations applicable on a global scale.


## ACKNOWLEDGMENTS

This work was supported in part by a grant from the Nebraska Corn Development, Utilization and Marketing Board.


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[Received September 20, 1995. Accepted June 12, 1996.]