# Relations of Grain Proximate Composition and Physical Properties to Wet-Milling Characteristics of Maize[1]

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## ABSTRACT

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The relations of proximate compositions and physical properties of 27 maize hybrids to laboratory wet-milling characteristics were determined. No single trait accounted for more than 40% of the variation ($r^2$) in starch yield or more than 60% of the variation in protein content of the recovered starch. Merely having higher starch content in the kernel did not increase starch yields ($r = 0.20$). Hybrids with lower protein contents ($r = -0.63$) and higher 1,000-grain weights ($r = 0.43$) yielded more starch, whereas hybrids with higher protein contents ($r = 0.77$) and harder endosperms ($r = 0.58$) gave higher residual protein contents of recovered starch. The best models for predicting starch yields included grain protein content and any one of the following: test weight, absolute density, kernel hardness (Stenvert sample height), or water absorptivity (index or initial rate). The preferred model for starch yield (percentage of starch yield = 58.2 − 3.6 [percentage grain protein] + 0.5 [test weight]) accounted for 61% of the variation. Protein content in starch was a function of grain protein and oil contents. The preferred model for protein content of recovered starch (percentage of protein in starch = −1.28 + 0.23 [percentage grain protein] + 0.13 [percentage grain oil]) accounted for 66% of the observed variation.

The growing importance of wet milling and other maize-processing industries has heightened interest in the relation of grain quality factors to end-use value. Current grading standards do not relate well to estimated end-use value of maize processed by the wet-milling industry (Watson 1987a). Based on grain proximate composition, the values of maize hybrids for wet milling have been estimated to range by as much as 15%. Maize hybrids with higher-than-normal starch contents are believed to have greater end-use values. Factors other than starch content of the grain, however, probably also relate to yields of starch and other high-value products from wet-milled maize. Understanding these relations may lead to a means of evaluating wet-milling potential at the time of first sale and/or purchase by wet-milling companies. It also may lead to improved hybrids yielding greater proportions of high-valued products, or ones that are more amenable to processing with reduced costs.

Watson and Hirata (1954) reported a method for determining millability of steeped grain by visually estimating the amount of starch released when thinly sectioned slices of steeped maize kernels were brushed. No correlation was found between the visual millability score and the yield of starch from wet milling. Freeman and Watson (1969) reported that gluten-starch separation in graduated cylinders was indicative of millability. Maize of high millability yielded sharp delineations between the milky white

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starch layer at the bottom of the cylinder and the less dense, but highly pigmented, gluten layer. These investigators, however, did not establish quantitative relations among the relative proportions, sharpness of delineation between deposits, or starch recovery in wet milling.

Freeman (1973) concluded that test weight, kernel size, stress cracks, and moisture content of maize gave little indication of product yields when wet milled. Likewise, Vojnovich et al (1975) observed no significant correlation between starch recovery and test weight, although they did find a high negative correlation ($r = -0.93$) between starch yield and drying temperature. Brown et al (1979) reported quantitative relations between steeping index (starch yield) and test weight ($r = 0.57$), stress cracks ($r = 0.47$), and viability ($r = 0.54$). They concluded that none of the measures was a good predictor of wet-milling performance.

Watson (1987b) contended, based on his experience, that kernel hardness, density, and breakage susceptibility are important in determining yields of wet-milled products, but he presented no statistical correlations of these properties to product yields. Weller (1987) and Weller et al (1988) related starch yield to starch content ($r = -0.65$), test weight ($r = 0.24$), and ethanol-soluble protein ($r = 0.45$) in the grain. They examined only four hybrids for differences in wet-milling characteristics and, thus, their correlations were not statistically significant. They also observed that starch yield decreased as harvest moisture and drying temperature increased.

Laboratory wet milling is time-consuming and labor-intensive. It is impractical to use such procedures to predict the wet-milling performance of grain at the time of sale. Rapid methods for determining quality factors indicative of wet-milled product yields would be very useful to the wet-milling industry and to breeders (Biss and Cogan 1988). The objective of this research was to determine the relations (correlations) of various grain composition factors and physical properties to yields and compositions of the products recovered from laboratory wet milling.

## MATERIALS AND METHODS

### Sample Collection

During 1987, 184 maize hybrid entries in the Iowa Corn Yield Trials were grown at the Iowa State University Agronomy and Agricultural Engineering Research Center near Ames. As determined by AACC method 44-15A (AACC 1983), moisture contents at harvest ranged from 18.2 to 22.8%. To eliminate any effects of hot-air drying and mechanical shelling on grain properties and wet-milling potential, maize was harvested by hand, forced-air (ambient, 20–25°C) dried on the cob to 13.6–15.6%, and then shelled in a laboratory sheller. The shelled maize (about 4–5 kg per hybrid) was cleaned over a 6.35-mm screen in a Carter-Day dockage tester (CEA, Minneapolis, MN) and stored at 4°C to prevent molding during the two-year period in which this work was done.

### Proximate Composition and Physical Properties

Proximate composition factors examined were grain moisture, crude free fat (oil), protein, and starch contents. Physical properties studied included 1,000-grain weight, test weight, absolute kernel density, breakage susceptibility, kernel hardness, water absorption index, initial water absorption rate, and moisture saturation point. The maize sample set used in this study was the same as that used by Dorsey-Redding et al (1991) to study relations among maize quality factors.

Oil, protein, and starch contents were estimated with a GAC III fixed-filter, near-infrared reflectance (NIR) analyzer (Dickey-john Corp., Auburn, IL). For NIR calibration, ground maize moisture contents were determined according to AACC method 44-15A (AACC 1983). Crude free fat content was calibrated according to AOAC methods 14-084 and 14-085 (AOAC 1984). Protein and starch contents were calibrated according to the Corn Refiners' Association methods A-18 and A-20, respectively (CRA 1986). All composition data were adjusted to 15.5% moisture basis.

Thousand-grain weight was determined for each sample by weighing 100 randomly selected, unbroken kernels to within 0.01 g and multiplying the result by 10. Four replications per sample were made. To correct for differences in moisture contents, 1,000-grain weight determinations were adjusted to 15.5% moisture basis according to the moisture adjustment equation reported by Dorsey-Redding et al (1990).

Test weight was determined according to the USDA Federal Grain Inspection Service method (FGIS 1988). Test weight values were adjusted to 15.5% moisture basis according to the moisture adjustment equation developed by Hall and Hill (1974).

Absolute kernel density was determined according to the air-comparison pycnometer method described by Thompson and Isaacs (1967). About 33 g of whole kernels were weighed to within 0.001 g. Volume determinations were made in triplicate with an air-comparison pycnometer (model 930, Beckman Instruments Inc., St. Louis, MO). Absolute density was adjusted to a 15.5% moisture basis by using the moisture adjustment equation of Dorsey-Redding et al (1990).

Breakage susceptibility, or the potential for kernels to break on impact, was determined with the Wisconsin breakage tester (Cargill, Minneapolis, MN) according to the methods described by Watson and Herum (1986). Breakage susceptibility was reported as the percentage of a 200-g sample passing through the 4.76-mm screen (Gunasekaran and Paulsen 1985). Breakage susceptibility values were adjusted to 15.5% moisture basis by using the moisture adjustment equation of Dutta (1986).

Kernel hardness was determined with the Stenvert Hardness Tester (Pomeranz et al 1985). Both ground-sample height and the time for a 20-g sample to be ground through the 2-mm screen of the Micro-Hammer Mill IV (Glen Mills Inc., Maywood, NJ) were determined. In this study, we encountered occasional clogging of the mill, which we believe made the time data less reliable than the height data. We therefore chose not to use the former. In this test, the ground grain of hybrids with harder endosperm occupies less volume and, thus, shows less height in the 125- × 25-mm standard collection tube. Three replications per sample were made. Stenvert height data were adjusted to 15.5% moisture basis according to the moisture adjustment equation of Dorsey-Redding et al (1990).

Water absorption properties were measured according to three different criteria: water absorption index (WAI), initial water absorption rate (IWAR), and moisture saturation point (MSP). WAI, a measure of the amount of water absorbed by the kernels in a 4-hr period, was determined according to the method of Hsu et al (1983). In this test, 10 kernels were placed in a beaker containing 300 ml of distilled water, which, in turn, was placed in a 30°C water bath. After 4 hr, the surfaces of the kernels were blotted dry, and the weight increase due to water absorption was measured. Two replications per sample were made. WAI data were adjusted to 15.5% moisture basis by using the moisture adjustment equation of Dorsey-Redding et al (1990).

IWAR was determined in duplicate by modifying methods of Hsu et al (1983). In this method, water absorption was measured after 0.5, 1, 2, 3, 4, 6, 8, 10, 12, 24, and 48 hr of steeping maize kernels in a beaker containing 300 ml of distilled water, which, in turn, was placed in a 30°C water bath. The plot of the moisture content versus time curve was adjusted to an initial moisture level of 15.5% (Dorsey-Redding et al 1990). The vast majority of water diffusion into the kernels occurred during the first hour of steeping. The slope of gain in moisture content over the first hour of steeping was used to estimate initial water absorption rate.

MSP, the maximum amount of moisture that the grain can absorb before rupturing the seed coat, was estimated by measuring the amount of water absorbed after 48 hr of steeping maize kernels in a beaker containing 300 ml of distilled water, which, in turn, was placed in a 30°C water bath. Duplicate samples for each hybrid were tested.

### Selection of Hybrids for Wet Milling

Because laboratory wet milling is tedious, not all 184 hybrids were wet milled. Based on the catalog of compositional factors and physical properties for the 184 hybrids, 27 hybrids representing the broadest range of proximate composition (oil, protein, and starch contents) and physical properties (1,000-grain weight, test weight, absolute kernel density, breakage susceptibility, kernel hardness, and water absorption properties) were selected for laboratory wet milling.

### Wet Milling

Each hybrid sample was divided into four subsamples after grain properties were determined, and each subsample (constituting one replication) was laboratory wet milled. The steeping procedure used was modified from the methods of Watson et al (1955) and Krochta et al (1981). Approximately 300 g of maize was steeped for 36 hr in a 600-ml solution containing 1.5% lactic acid and 0.05% SO$_2$, followed by 12 hr in a second 600-ml solution containing 0.5% lactic acid and 0.1% SO$_2$. The steeped grain then was wet milled using procedures previously reported by Steinke and Johnson (1991). All fractions, except for starch, were dried in a forced-air oven for 24 hr at 60°C and then dried in a vacuum oven for 12 hr at 60°C. To evaluate the variation in starch properties due to hybrid and drying temperature for a companion study, two of the starch samples were dried at 45°C and the remaining two at 60°C in both forced-air and vacuum ovens for 12 hr each. After drying, all fractions were weighed for yield and then ground for proximate analysis.

### Proximate Analysis of Wet-Milled Products

All fractions were analyzed in duplicate for moisture, crude free fat, and protein contents. Moisture content was determined by drying 2-g ground samples in a convection oven for 3 hr at 130°C (AOAC 1984, method 14.004). Percentage of yield was calculated on a dry-weight basis (db) for each fraction.

Crude free fat content was determined according to AOAC methods 14-084 and 14-085 (AOAC 1984). Germ fractions were analyzed in duplicate. All other fractions were analyzed once for oil.

Protein contents were determined in duplicate according to the Corn Refiners Association macro-Kjeldahl method A-18 (CRA 1986) and the Tecator Kjeltec system (Tecator, Hoganas, Sweden).

**TABLE I**
**Composition and Physical Properties of 27 Maize Hybrids Selected for Wet Milling**

| Property[a] | Mean | Low | High | SD[b] | LSD[b,c] |
|---|---|---|---|---|---|
| Composition | | | | | |
| Crude free fat, % | 3.4 | 2.8 | 4.0 | 0.3 | ... |
| Protein, % | 8.7 | 7.9 | 9.6 | 0.5 | ... |
| Starch, % | 59.5 | 57.6 | 60.6 | 0.8 | ... |
| Starch, % db | 70.0 | 67.8 | 71.3 | 0.9 | ... |
| Size and density | | | | | |
| 1,000-grain weight, g | 338 | 291 | 428 | 30 | 10 |
| Test weight, lb/bu | 59.9 | 54.7 | 63.6 | 2.2 | ... |
| Absolute density, g/cm$^3$ | 1.26 | 1.20 | 1.30 | 0.03 | 0.002 |
| Kernel strength | | | | | |
| Breakage susceptibility, % | 2.82 | 0.99 | 5.17 | 0.88 | ... |
| Stenvert height, cm | 10.8 | 10.1 | 11.7 | 0.44 | 0.3 |
| Water uptake | | | | | |
| WAI[d] | 0.169 | 0.137 | 0.201 | 0.017 | 0.010 |
| IWAR,[d] hr$^{-1}$ | 0.063 | 0.057 | 0.069 | 0.004 | 0.004 |
| MSP[d] | 0.392 | 0.373 | 0.420 | 0.014 | 0.009 |

[a] Adjusted to 15.5% moisture content, except for starch, which was adjusted to dry basis.

[b] Standard deviations and least significant difference, respectively.

[c] $P < 0.05$, between hybrids.

[d] Water absorption index, initial water absorption rate, and moisture saturation point, respectively.

### Statistical Analysis

Means, standard deviations (SD), and least significant difference (LSD) values were calculated using the SAS general linear models procedure (SAS 1984). Correlation coefficients ($r$) were calculated by means of the SAS correlation (CORR) procedure. Multiple correlations ($r^2$), probability $>|T|$ values, and regression equations were calculated by means of the SAS regression (REG) procedure.

## RESULTS AND DISCUSSION

### Proximate Composition and Physical Properties

Means, SD values, and LSD values for each composition factor and physical property of the 27 samples selected for wet milling are presented in Table I. Wide ranges in proximate composition and physical properties among the 27 selected hybrids were achieved, especially in starch and protein contents, absolute density, Stenvert height, and WAI. Among the 27 selected hybrids, starch contents ranged from 57.6 to 60.6% and protein contents ranged from 7.9 to 9.6%. These great differences in composition were expected to cause significant differences in wet-milling yields of starch and in protein contents of the recovered starch. The selected hybrids also differed greatly in hardness, as indicated by the Stenvert height range of 10.1–11.7 cm (LSD of 0.3 cm). We previously reported on the interrelations of proximate composition factors and physical properties (Dorsey-Redding et al 1991).

### Yields and Recoveries of Wet-Milled Products

The yields of wet-milled products from the 27 maize hybrids differed greatly (Table II). Starch yields (percentage of solids in the original maize) were lower than what is typically recovered by commercial corn wet mills (67.5% db) (Anderson and Watson 1982). This lower yield was mainly due to the 2.7–10.2% of inseparables and the large amounts of total dissolved solids recovered in our procedure. The mean yields of germ, gluten, and fiber recovered were not very different from industry values. The laboratory wet-milling yields obtained were similar to other reports of laboratory wet milling in which batch steeping had been used (Anderson and Watson 1982, Steinke et al 1991). Our laboratory procedures, however, may be more sensitive to differences in maize millability than are commercial practices. Current commercial practices have probably evolved for the poorest wet-milling properties encountered.

Relatively high SD and LSD values (Table II) indicated considerable variation among replications and suggested a long-term need for more precise wet-milling techniques. Variation analysis (Table III) estimated that nearly half of the observed

**TABLE II**
**Yields of Wet-Milled Products Among 27 Hybrids**

| Fraction (% db) | Present Study | | | | | Industry Mean[c] |
|---|---|---|---|---|---|---|
| | Mean | Low | High | SD[a] | LSD[b] | |
| Starch | 56.8 | 50.9 | 60.0 | 2.4 | 4.1 | 67.5 |
| Gluten | 6.7 | 5.8 | 8.4 | 0.6 | 1.0 | 5.8 |
| Fiber | 13.5 | 11.2 | 17.2 | 1.5 | 2.3 | 11.5 |
| Germ | 6.2 | 5.2 | 7.5 | 0.5 | 0.7 | 7.5 |
| Inseparables | 5.1 | 2.7 | 10.2 | 1.6 | 3.3 | ... |
| Steeping solids | 9.2 | 8.1 | 10.0 | 0.5 | 0.9 | ... |
| Wash solids | 3.3 | 2.5 | 3.9 | 0.4 | 0.9 | ... |
| Total dissolved solids[d] | 12.5 | 11.5 | 13.8 | 0.5 | 1.3 | 11.5 |
| Crude starch[e] | 61.9 | 56.4 | 64.6 | 2.0 | 2.6 | ... |
| Mill starch[f] | 68.5 | 64.8 | 70.9 | 1.7 | 2.6 | ... |
| Total solids recovered | 100.7 | 98.8 | 103.0 | 1.0 | 2.4 | 99.8 |

[a] Standard deviation.

[b] Least significant difference. $P < 0.05$, between hybrids.

[c] Anderson and Watson (1982).

[d] Sum of steep solids and wash solids.

[e] Sum of starch and inseparables.

[f] Sum of starch, inseparables, and gluten.

variation in starch yield was accounted for within hybrids and, thus, was due to relatively high procedural variability. Differences in mean yields of wet-milled products among hybrid subsamples (Table IV), for the most part, were not statistically significant. This supports the contention (as shown in Table III) that over half of the observed variations in starch, gluten, fiber, and germ yields were because of differences among selected hybrids rather than because of procedural differences within hybrids, i.e., among hybrid subsamples (replications).

### Proximate Composition of Products

Means, SD values, and LSD values for protein and oil contents of the major wet-milled products are shown in Table V. Protein contents of the recovered starch fractions were higher (0.91–1.46% db) than those normally observed in industry (0.3%). We attribute this to differences in the procedure and hybrids used in this study, compared with those used by industry. Residual protein content in starch was more sensitive to grain properties in our procedure than it seems to be in industrial practices. Mean protein contents of the other products were similar to those observed in previous work (Anderson and Watson 1982, Steinke and Johnson 1991, Steinke et al 1991, Wang and Johnson 1991).

### Single-Factor Correlations

All proximate composition and physical property data were correlated with product yields, starch recovery, and product composition data (Table VI).

*Starch.* Only one grain quality factor (protein) correlated well, i.e., at the 0.01 significance level, with starch yield. The negative correlation ($r = -0.63$) indicated that maize hybrids with high protein contents yield less starch. Presumably, the more protein available to form protein matrices around starch granules, the more difficult it becomes to recover the starch. Surprisingly, poor positive correlation ($r = 0.20$) was observed between NIR starch content and starch yield. Higher starch content should favor higher starch yield, but our results indicate that other properties, such as protein content, are equally or more important determinants of starch yield.

Thousand-grain weight significantly and positively correlated ($r = 0.43$) with starch yield at the 0.05 significance level. Thus, hybrids with larger kernels yielded more starch. This correlation probably results from less seed coat per unit weight of starch, because the ratio of surface area to volume decreases as 1,000-grain weight increases. Starch yield measures the amount of starch recovered in relation to total solids present in the grain. By contrast, starch recovery measures the yield of starch in relation to the amount of starch present in the grain. The recovery of starch was slightly less correlated ($r = -0.56$) with grain protein content than was starch yield ($r = -0.63$), and slightly more correlated ($r = 0.54$) with 1,000-grain weight than was starch yield ($r = 0.43$).

Residual protein levels in starch correlated with four physical properties at the 0.01 significance level. The protein content of the recovered starch was positively correlated ($r = 0.77$) with grain protein content. This correlation indicates that for hybrids with higher protein contents, it is more difficult to achieve clean separations of protein and starch, and that the poor separation characteristics result in higher protein contents in the recovered starch. The protein content in the recovered starch was negatively correlated with breakage susceptibility ($r = -0.58$) and Stenvert sample height ($r = -0.58$). These correlations indicate that hybrids with more resistance to breakage and with harder endosperms are more difficult to wet mill. Protein contents in starch were correlated with two additional physical properties at the 0.05 significance level. Absolute kernel density was positively correlated with protein levels in starch ($r = 0.43$). The correlation indicates that denser hybrids are more difficult to wet mill. Grain oil contents also were positively correlated with protein content of the recovered starch ($r = 0.67$). This correlation suggests that cultivars high in oil are less millable.

*Gluten.* Gluten yields correlated with four quality factors at the 0.01 level of significance. Gluten yields were positively correlated ($r = 0.67$) with grain protein content. Hardness as measured by Stenvert height also was significantly correlated with gluten yield ($r = -0.56$). Because hardness is correlated with protein content ($r = 0.64$, in 1987 data reported by Dorsey-Redding et al [1991]), harder hybrids yield more gluten. Gluten yield also was negatively correlated with 1,000-grain weight ($r = -0.51$). This correlation indicates that hybrids with smaller kernel size yield more gluten. Grain oil content was positively correlated at the 0.05 significance level with gluten yield ($r = 0.43$).

The protein content of gluten was correlated with six compositional and physical properties at the 0.01 significance level. Grain protein content was positively correlated with gluten protein content ($r = 0.58$). This correlation indicates that hybrids with

TABLE III
Variation in Yields of Wet-Milled Products Among 27 Hybrids

| Fraction | CV Within Hybrid (%) | CV Across Hybrids (%) | Percent Variation Due to Differences Among Hybrids |
|---|---|---|---|
| Starch | 5.1 | 8.4 | 50.6 |
| Gluten | 10.9 | 23.2 | 63.0 |
| Fiber | 12.0 | 24.7 | 61.1 |
| Germ | 8.4 | 20.2 | 68.1 |
| Inseparables | 46.7 | 67.6 | 43.8 |
| Steeping solids | 7.1 | 11.3 | 48.1 |

TABLE IV
Mean Yields of Wet-Milled Products Among Subsamples of 27 Hybrids

| Fraction (% db) | Hybrid Subsample 1 | 2 | 3 | 4 | Mean | SD[a] | LSD[b] |
|---|---|---|---|---|---|---|---|
| Starch | 55.7 | 56.4 | 57.5 | 57.5 | 56.8 | 0.9 | 1.6 |
| Gluten | 6.3 | 6.3 | 6.6 | 7.5 | 6.7 | 0.6 | 0.4 |
| Fiber | 14.1 | 14.6 | 12.1 | 13.1 | 13.5 | 1.1 | 0.9 |
| Germ | 5.6 | 6.2 | 6.7 | 6.4 | 6.2 | 0.4 | 0.3 |
| Inseparables | 6.1 | 4.1 | 5.2 | 4.9 | 5.1 | 0.9 | 1.3 |
| Steeping solids | 8.9 | 9.0 | 9.4 | 9.5 | 9.2 | 0.3 | 0.4 |
| Wash solids | 3.1 | 3.4 | 3.4 | 3.5 | 3.3 | 0.2 | 0.3 |
| Total dissolved solids[c] | 12.0 | 12.3 | 12.8 | 13.0 | 12.5 | 0.4 | 0.5 |
| Crude starch[d] | 61.9 | 60.5 | 62.7 | 62.4 | 61.9 | 1.0 | 1.0 |
| Mill starch[e] | 68.2 | 66.7 | 69.3 | 69.9 | 68.5 | 1.4 | 1.0 |
| Total solids recovered | 99.9 | 99.8 | 100.9 | 102.3 | 100.7 | 1.2 | 0.9 |

[a] Standard deviation.
[b] Least significant difference. $P < 0.05$, between hybrid subsamples.
[c] Sum of steep solids and wash solids.
[d] Sum of starch and inseparables.
[e] Sum of starch, inseparables, and gluten.

higher protein content yield gluten with higher protein content. We previously have shown (Dorsey-Redding et al 1991) that grain protein content is significantly correlated ($P < 0.01$) with starch content ($r = -0.35$), breakage susceptibility ($r = -0.42$), absolute density ($r = 0.39$), WAI ($r = -0.29$), and Stenvert height ($r = -0.64$). Thus, it was not surprising to find also that gluten protein content was correlated with breakage susceptibility ($r = -0.39$), absolute density ($r = 0.58$), WAI ($r = -0.53$), Stenvert height ($r = -0.53$), IWAR ($r = -0.60$), and MSP ($r = -0.62$).

*Fiber.* Fiber yield was correlated with two composition factors at the 0.01 significance level. Fiber yield was positively correlated with grain protein content ($r = 0.57$). This correlation indicates that maize hybrids with higher protein contents yield more fiber. Perhaps this correlation was due to poor separation of endosperm from bran caused by increased protein content and/or hardness. Breakage susceptibility was negatively correlated with fiber yield ($r = -0.54$). This correlation suggests that bran structure is related to breakage susceptibility.

*Germ.* Germ yields were not significantly correlated with any

TABLE V
**Protein and Crude Free Fat Contents of Wet-Milled Products**

| Fraction | Present Study Mean | Low | High | SD[a] | LSD[b] | Industry Mean[c] |
|---|---|---|---|---|---|---|
| Protein content, % db | | | | | | |
| Starch | 1.14 | 0.91 | 1.46 | 0.16 | 0.26 | 0.30 |
| Gluten | 45.3 | 35.4 | 53.9 | 3.7 | 6.1 | 65.8 |
| Fiber | 11.5 | 9.8 | 13.7 | 1.0 | 1.2 | 12.0 |
| Germ | 11.9 | 9.9 | 14.0 | 1.0 | 1.1 | 12.0 |
| Inseparables | 3.1 | 2.4 | 4.1 | 0.5 | 1.2 | ... |
| Crude free fat content, % db | | | | | | |
| Starch | 0.07 | 0.04 | 0.09 | 0.01 | 0.04 | ... |
| Gluten | 4.2 | 1.1 | 7.1 | 1.9 | 2.6 | ... |
| Fiber | 3.7 | 2.4 | 5.1 | 0.7 | 1.5 | ... |
| Germ | 53.2 | 46.6 | 59.7 | 3.6 | 4.1 | ... |
| Inseparables | 0.19 | 0.11 | 0.32 | 0.05 | 0.20 | ... |

[a] Standard deviation.
[b] Least significant difference. $P < 0.05$, between hybrids.
[c] Anderson and Watson (1982).

TABLE VI
**Correlation Coefficients ($r$) Between Maize Quality Factors and Yields of Wet-Milled Products**

| | Kernel Composition Factor | | | Physical Property | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Protein | Starch | Crude Free Fat | 1,000-Grain Weight | Test Weight | Absolute Density | Breakage Susceptibility | Stenvert Height | WAI[a] | IWAR[b] | MSP[c] |
| Product yield | | | | | | | | | | | |
| Starch | −0.63**[d,e] | +0.20 | −0.02 | +0.43*[f] | +0.32 | −0.02 | +0.37 | +0.11 | −0.16 | −0.10 | −0.12 |
| Gluten | +0.67** | +0.01 | +0.43* | −0.51** | +0.21 | +0.33 | −0.33 | −0.56** | −0.08 | −0.23 | −0.10 |
| Fiber | +0.57** | −0.20 | +0.14 | −0.13 | −0.05 | +0.19 | −0.54** | −0.27 | +0.29 | −0.08 | +0.07 |
| Germ | +0.13 | −0.15 | +0.03 | +0.13 | +0.01 | −0.14 | −0.01 | +0.08 | −0.26 | +0.18 | +0.01 |
| Steeping solids | +0.58** | −0.27 | +0.18 | −0.14 | +0.16 | +0.44 | −0.16 | −0.36 | −0.22 | −0.25 | −0.36 |
| Total dissolved solids | +0.36 | −0.07 | +0.07 | −0.18 | −0.23 | +0.04 | +0.08 | −0.03 | +0.01 | −0.03 | −0.03 |
| Recovery | | | | | | | | | | | |
| Starch | −0.56** | −0.09 | −0.18 | +0.54* | +0.30 | −0.03 | +0.32 | +0.15 | −0.24 | −0.06 | −0.16 |
| Composition of products | | | | | | | | | | | |
| Protein in starch | +0.77**[g] | −0.12 | +0.46* | −0.24 | +0.28 | +0.43* | −0.58**[h] | −0.58**[i] | −0.03 | −0.35 | −0.18 |
| Protein in gluten | +0.58** | −0.30 | +0.22 | −0.07 | +0.32 | +0.58** | −0.39* | −0.53** | −0.53** | −0.60** | −0.62** |
| Protein in germ | −0.09 | −0.05 | −0.36 | +0.25 | −0.34 | −0.50** | −0.07 | +0.30 | +0.44* | +0.40* | +0.63** |
| Crude free fat in germ | −0.03 | +0.09 | +0.60** | −0.09 | +0.71* | +0.57** | +0.07 | −0.49* | −0.38 | −0.41* | −0.55** |

[a] Water absorption index.
[b] Initial water absorption rate.
[c] Moisture saturation point.
[d] Significant at 0.05 and 0.01 for * and **, respectively.
[e] Line of best fit is starch yield (%) = 84.25 − 3.14 (percentage grain protein); probability of greater than $|T|$ is 0.0004.
[f] Line of best fit is starch yield (%) = 45.24 + 0.03 (1,000-grain weight); probability of greater than $|T|$ is 0.0264.
[g] Line of best fit is protein in starch (%) = −1.07 + 0.25 (percentage grain protein); probability of greater than $|T|$ is 0.0001.
[h] Line of best fit is protein in starch (%) = 1.43 − 0.10 (breakage susceptibility); probability of greater than $|T|$ is 0.0016.
[i] Line of best fit is protein in starch (%) = 3.39 − 0.21 (Stenvert height); probability of greater than $|T|$ is 0.0014.

TABLE VII
**Mathematical Models and Multiple Correlation ($r^2$) Values of Starch Yield to Grain Protein Content and One Other Property**

| Mathematical Model[a] | $r^2$[b] |
|---|---|
| Composition | |
| Starch yield, % = 82.1 − 3.4 (percentage grain protein) + 1.3 (percentage grain crude free fat) | 0.43 |
| Starch yield, % = 85.8 − 3.2 (percentage grain protein) − 0.02 (percentage grain starch) | 0.40 |
| Size and density | |
| Starch yield, % = 74.4 − 2.7 (percentage grain protein) + 0.02 (1,000- grain weight) | 0.45 |
| Starch yield, % = 58.2 − 3.6 (percentage grain protein) + 0.5 (test weight) | 0.61** |
| Starch yield, % = 50.1 − 4.0 (percentage grain protein) + 32.8 (absolute density) | 0.50* |
| Kernel strength | |
| Starch yield, % = 84.5 − 3.2 (percentage grain protein) − 0.02 (breakage susceptibility) | 0.40 |
| Starch yield, % = 119.7 − 4.4 (percentage grain protein) − 2.3 (Stenvert height) | 0.51* |
| Water uptake | |
| Starch yield, % = 94.9 − 3.5 (percentage grain protein) − 45.6 (WAI[c]) | 0.50* |
| Starch yield, % = 103.5 − 3.7 (percentage grain protein) − 231.7 (IWAR[d]) | 0.50* |
| Starch yield, % = 108.1 − 3.5 (percentage grain protein) − 52.1 (MSP[e]) | 0.49 |

[a] Regression equations representing mathematical model of best fit according to $t$-test parameter estimates.
[b] Significant at 0.05 and 0.01 for * and **, respectively.
[c] Water absorption index.
[d] Initial water absorption rate.
[e] Moisture saturation point.

**TABLE VIII**

**Mathematical Models and Multiple Correlation ($r^2$) Values of Protein Content in Starch (Starch Purity) to Grain Protein Content and One Other Property**

| Mathematical Model[a] | $r^2$ |
|---|---|
| Composition | |
| Protein in starch, %[b] $= -1.28 + 0.23$ (percentage grain protein) $+ 0.13$ (percentage grain crude free fat) | 0.66*[c] |
| Protein in starch, % $= -3.19 + 0.27$ (percentage grain protein) $+ 0.03$ (percentage grain starch) | 0.62 |
| Size and density | |
| Protein in starch, % $= -1.16 + 0.26$ (percentage grain protein) $+ 0.01$ (1,000-grain weight) | 0.60 |
| Protein in starch, % $= -1.56 + 0.24$ (percentage grain protein) $+ 0.01$ (test weight) | 0.61 |
| Protein in starch, % $= -1.58 + 0.24$ (percentage grain protein) $+ 0.49$ (absolute density) | 0.60 |
| Kernel strength | |
| Protein in starch, % $= -0.66 + 0.22$ (percentage grain protein) $- 0.03$ (breakage susceptibility) | 0.62 |
| Protein in starch, % $= -0.02 + 0.22$ (percentage grain protein) $- 0.07$ (Stenvert height) | 0.62 |
| Water uptake | |
| Protein in starch, % $= -1.40 + 0.26$ (percentage grain protein) $+ 1.4$ (WAI[d]) | 0.62 |
| Protein in starch, % $= -0.66 + 0.24$ (percentage grain protein) $- 5.0$ (IWAR[e]) | 0.61 |
| Protein in starch, % $= -1.16 + 0.25$ (percentage grain protein) $+ 0.18$ (MSP[f]) | 0.60 |

[a] Regression equations representing mathematical model of best fit according to $t$-test parameter estimates.

[b] Percentage of residual protein content in recovered starch fraction.

[c] Significant at 0.05.

[d] Water absorption index.

[e] Initial water absorption rate.

[f] Moisture saturation point.

of the proximate composition factors or physical properties, including oil content. This finding was surprising because increased size of germs has been attributed to higher oil contents in maize kernels (Watson 1987a).

Four factors correlated with oil content of the germ at the 0.01 significance level. Not surprisingly, oil content of the germ fraction was positively correlated with oil content of the grain ($r = 0.60$). Among the 27 hybrids, higher oil content in the kernel was not related to germ size, but to increased proportion of oil in the germ. Test weight and absolute kernel density also were positively correlated with germ oil contents ($r = 0.71$ and 0.57, respectively). This finding indicates that hybrids producing denser grain yield more oil in the germ. MSP and IWAR were negatively correlated with germ oil content ($r = -0.55$ and $-0.41$, respectively). This finding indicates that kernels with higher oil contents in their germs absorb less water and absorb more slowly. Stenvert height was negatively correlated with germ oil content ($r = -0.49$). This finding indicates that harder hybrids yield germs with more oil.

Germ protein is the major component that leaches from the grain into the steep liquor. Two factors correlated with remaining germ protein contents (after steeping) at the 0.01 significance level. Absolute kernel density was negatively correlated with protein content of the germ ($r = -0.50$). This correlation indicates that there was more leaching of germ protein during steeping from denser hybrids. MSP was correlated with germ protein content ($r = 0.63$), as were WAI ($r = 0.44$) and IWAR ($r = 0.40$).

*Steeping solids and total dissolved solids.* The solids leached during steeping into the steep solution were positively correlated with grain protein content ($r = 0.58$) at the 0.01 significance level. Hybrids containing more protein leach more solids during steeping. Steeping solids also were correlated with absolute kernel density ($r = 0.44$) at the 0.05 significance level. Total dissolved solids from all liquid fractions (steeping solids and starch wash-water solids) were not significantly correlated with any of the composition factors or physical properties tested.

### Multiple Regression

To account for additional variation in starch yields and protein contents of the recovered starch, we considered multiple regression (two and three factors).

*Starch yield.* Starch yield as a function of grain protein content and one additional property was investigated (Table VII). Starch yield was significantly correlated at the 0.01 significance level with grain protein content and test weight. The model of best fit ($r^2 = 0.61$) was

$$\text{Percentage starch yield} = 58.2 - 3.6\,(\text{percentage grain protein}) + 0.5\,(\text{test weight}).$$

Thus, 61% of the variation was accounted for by these two factors, opposed to 40% by grain protein alone. We have accounted for all of the variation between hybrids (Table III), and the remaining variation was due to variation in the wet-milling procedure.

Multiple regression of starch yield to grain protein plus any one of four additional properties, including absolute kernel density ($r^2 = 0.50$), Stenvert height ($r^2 = 0.51$), WAI ($r^2 = 0.50$), or IWAR ($r^2 = 0.50$), were significant at the 0.05 level.

*Protein content of starch.* Multiple correlations of protein contents of the recovered starch to grain protein contents and one other property also were investigated (Table VIII). No additional properties improved the correlation at the 0.01 significance level. However, adding the oil content of the grain into the mathematical model was significant at the 0.05 level, and the multiple correlation coefficient improved to $r^2 = 0.66$. Thus, the model

$$\begin{aligned}&\text{Percentage protein content in recovered starch}\\ &= -1.28 + 0.23\,(\text{percentage grain protein})\\ &\quad + 0.13\,(\text{percentage grain oil})\end{aligned}$$

accounted for 66% of the variation, compared with 60% for the model considering only the protein content of the grain.

Three-factor correlations, in which starch yield was correlated with grain protein plus two other properties, also were examined. No significant improvements in the mathematical model were achieved.

## CONCLUSIONS

Proximate composition factors and physical properties of maize were correlated to yields and protein contents of laboratory wet-milled products. Several of the grain properties tested affected wet-milling performance, indicating significant differences in millability of maize hybrids. Starch yields were functions of percentage grain protein content and any one of the following: test weight, absolute kernel density, Stenvert hardness (ground sample height), WAI, or IWAR. Residual protein content in recovered starch was a function of the protein and oil contents of the grain. Because nearly one half of the observed variation in starch yield was attributed to wet-milling methods, more reliable and reproducible techniques for simulating commercial wet milling are needed to support breeding programs and the industry's quality control efforts.

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