

Effects of Maturity on Grain Quality and Wet-Milling Properties of Two Selected Corn Hybrids

S. D. Jennings,¹ D. J. Myers,^{2,3} L. A. Johnson,⁴ and L. M. Pollak⁵

ABSTRACT

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The effects of maturity on grain quality and wet-milling properties were investigated for two hybrids of corn. Significant differences for hybrid and maturity were observed for all grain quality parameters. Test weight, absolute density, and thousand-grain weight all increased as the corn matured. Kernel hardness increased and breakage susceptibility varied with increased maturity. Water uptake parameters decreased with maturity

of the grain. The starch yield results from wet milling showed that the starch yield increased significantly within each cultivar in the early stages of grain maturity, but there were no significant differences between hybrids. Mathematical models using selected grain quality parameters accurately predicted trends in starch yield for the immature and mature corn samples in this study.

Approximately 9 billion bushels of corn are produced in the United States each year. About 15% of this grain is processed by the wet-milling industry; however, not all grain is suitable for wet milling. Typically, corn that has been graded and classified by the Federal Grain Inspection Service as No. 2 yellow dent corn is used. The standards for No. 2 yellow corn used in determining selling price of the grain are 1) a minimum test weight per bushel of 54 lbs; 2) a maximum limit of 2% heat-damaged kernels and 5% total damaged kernels; and 3) a maximum limit of 3% broken kernels and foreign material. There is considerable uncertainty in the industry with regard to which factors give valuable information about the suitability of the corn for wet milling, as well as the earliest time the grain can be harvested without jeopardizing the yields of high-value wet-milled products.

Grain that has been harvested early due to unfavorable weather conditions, such as the threat of an early frost, may not meet these grading specifications. The question is whether or not this corn is suitable for wet milling. There have been numerous attempts made by a number of researchers to relate grain quality factors to the value of corn for wet milling. In 1950, Braid and coworkers attempted to use a test for viability with 2,3,5 triphenyltetrazolium chloride solution to relate quality factors to the value of corn for wet milling. Their results favorably linked the test method and starch recovery. However, other researchers (Gausman et al 1952; MacMasters et al 1954) refuted the results finding that color test was “less apparent than roughly proportional” and starch recovery was not correlated at all. Freeman (1973) reviewed factors that are not included in the U.S. grading standards that could affect the value of corn for wet milling. These factors included oil content and distribution, oil quality, carotenoid pigment, stress cracks, and grain preservatives. However, no statistical correlations were presented to support this data. Brown et al (1979) attempted to relate a steeping index (a measure of percentage of starch recovery) to quality factors. They found a moderate correlation between stress cracks, viability, and test weight, but concluded that, individually, they did not accurately predict wet-milling performance. Watson (1987) expressed belief, based on experience, in breakage susceptibility, hardness, and density as being important quality factors in determining yields of products from wet milling. However, he did not elaborate on these factors with predictive equations or correlations. The study by Weller et al (1988)

found starch recovery to be a function of starch content, test weight, and ethanol-soluble protein, but found no statistical significance in correlations. Fox et al (1992) attempted to correlate grain proximate composition and physical properties to wet-milling characteristics and found that a model using grain protein content and test weight could account for 61% of the variation for starch yield. A second predictive model using grain protein content and oil content could account for 66% of the observed variation for the protein content of recovered starch, but there was low correlation between starch yield and starch content.

Because the effect of maturity on the quality of corn and the yields of wet-milled products are not well understood, the objectives of this study were 1) to evaluate the quality of corn at immature and mature stages of development using a variety of grain quality parameters; 2) to use the grain proximate composition and a grain quality attribute in a mathematical model and predict the starch yield before milling the grain; and 3) to mill the grain and compare the actual yields to those predicted by the models.

MATERIALS AND METHODS

Sample Collection and Storage

Two corn hybrids from the 1993 crop year were harvested at three stages of maturity as measured by moisture content, milk line, and presence of black layer. These harvests were made in approximately two-week intervals from the Iowa State University Agronomy and Agricultural Engineering Research Center near Ames, IA. Pioneer 3525 was an early maturing hybrid (106 days before flowering [DBF]), and Pioneer 3394 was a late-maturing hybrid (111 DBF). The moisture levels of the samples at harvest was 25–56% as determined by Approved Method 44-15A (AACC 2000). The hand-harvested corn was gently dried on the cob to 11–13% moisture in carts that facilitated the ventilation of room air and then shelled using a customized mechanical sheller (ISU Agronomy and Agricultural Research Farm). The corn samples (2–4 kg) were stored at 4°C throughout the study.

Grain Quality

To determine the effect of maturity on grain quality, seven grain quality parameters were quantitatively analyzed: 1) test weight determined according to the USDA Federal Grain Inspection Service (FGIS 1993); 2) absolute density (Thompson and Isaacs 1967); 3) kernel hardness determined using the Stenvert hardness test (Pomeranz et al 1985); and 4) water absorption index (WAI) developed by Hsu et al (1983). Thousand-grain weight, breakage susceptibility, and moisture saturation point procedures were cited by Fox et al (1992). Moisture adjustment equations reported by Dorsey-Redding et al (1990) were used on thousand-grain weight, absolute density, kernel hardness, and WAI values reported on a 15.5% moisture basis. Test weight was adjusted to 15.5% moisture using the equation of

¹ Proliant Inc., 2325 North Loop Drive, Ames, IA 50010.

² Associate professor, Department of Food Science and Human Nutrition, Iowa State University, Ames, IA 50011.

³ Corresponding author. E-mail: dmyers@iastate.edu.

⁴ Professor-in-charge, Center for Crops Utilization Research, Iowa State University, Ames, IA 50011.

⁵ Research geneticist, USDA-ARS, Department of Agronomy, Iowa State University, Ames, IA 50011.

Hall and Hill (1974). The equation of Dutta (1986) was used to determine breakage susceptibility. All analyses were made in triplicate for each maturity level.

Proximate Composition

Protein, oil, and starch contents for the corn samples were estimated with a near-infrared reflectance (NIR) grain analyzer (Infratec 1225) calibrated against standard methods. Protein and starch contents were calibrated according to CRA methods A-18 and A-20, respectively (CRA 1986). Crude free fat content was calibrated according to AOAC methods 14-084 and 14-085 (AOAC 1984). All composition data were adjusted to a 15.5% moisture basis. All analyses were made in triplicate at each maturity level.

Prediction of Starch Yield and Comparison to Actual Yield

Mathematical models (Fox et al 1992) that use grain protein content and an additional grain quality measurement were used to predict trends in percentage of starch yield of the corn samples before milling. Actual percentages of starch yields were not attainable because the method of starch and gluten separation used in this study was not the same as the procedure used in the study that created the mathematical models.

Wet Milling

Wet milling was performed using procedures previously reported by Steinke and Johnson (1991) with modifications.

Steeping. Steeping solutions for this study were prepared using deionized water. Each of the steepers were placed in a 4L beaker and immersed in a 50°C water bath. Whole corn kernels (≈1 kg) were steeped for 36 hr in a 3L solution of 1.5% lactic acid and 0.05% SO₂, followed by 12 hr in a second 3L solution containing 0.5% lactic acid and 0.1% SO₂.

Germ separation. The steeped grain was first coarsely ground in a Waring blender (Dynamics Corp. of America, New Hartford, CT) to liberate the germ intact. Equal volumes of corn and water were added to the 1.2L blender cup and blended for ≈2 min at one-third speed. A 115V AC variable autotransformer set at 33% was used to control the speed. The coarsely ground slurry was then transferred to a 10-qt bucket, and 1L of distilled water was added to facilitate germ floatation. The slurry was stirred with a spatula and allowed to settle for 2 min. The germ was then skimmed from the surface with a wire screen (20 mesh) and removed from the screen by subtle tapping on the inside of a 800-mL beaker. After all the germ had been

recovered, it was transferred to a U.S. Standard 200 sieve and washed with 50 mL of distilled water. The wash water was then returned to the degerminated slurry. The germ was dried, weighed for yield, and analyzed for moisture, oil, and protein contents.

Fiber separation. The degerminated slurry was blended for a second and final time with ≈400 mL of distilled water in the blender at full speed for 2 min. This fine grinding released the endosperm from the bran and helped release the starch from the protein matrix in the endosperm. Fiber was separated by pouring ≈200 mL of the ground slurry on a U.S. Standard 200 sieve and pan. The sieve and pan were then shaken on a testing sieve shaker (Ro-Tap, W.S. Tyler, Inc., Mentor, OH) with tapping. The fraction remaining on the No. 200 sieve was a mixture of fine and coarse fiber, and the material that passed through the sieve was considered mill starch. The fiber fraction was transferred to a 1L beaker with a rubber spatula. Small portions of the fiber were washed in 2L of distilled water, poured over a 200 sieve, washed in an additional 2L of distilled water, and then scraped onto a piece of nylon cloth with 52-μm openings (Spectra/Mesh, Fisher Scientific, Pittsburgh, PA). As much water as possible was squeezed from the cloth into a pan and then returned to the rest of the wash water until the process was complete. The fiber was removed from the cloth, dried, weighed for yield, and analyzed for moisture, oil, and protein contents. The wash water was combined with the mill starch.

Starch and protein separation. The last step in the milling procedure was the separation of the starch and gluten. This was achieved by starch tabling. The starch table was an I-beam 6 m long and 0.1 m in width, set at 2° pitch. The starch slurry was adjusted to a specific gravity of 1.04 using a hydrometer (Fisher Scientific) and decanting excess water. The slurry was then pumped onto the starch table at 300 mL/min using a peristaltic variable-speed drive system (Cole Parmer, Niles, IL). Separation was achieved based on the difference in density of starch and gluten, with the starch being the heavier of the two. The starch was allowed to settle on the table for ≈10 min. Distilled water (3L) delivered by the pump, and 50 mL of distilled water delivered by a squirt bottle, were used to wash the starch. The gluten and wash water was collected in a bucket at the end of the I-beam. The remaining starch fraction was then dried on the table for 28 hr. Because there was often a buildup of gluten at the end of the table, the last 0.6 m of the table was scraped off separately and labeled as the inseparables fraction. The rest was removed as the starch fraction. Both the starch and the inseparables fractions were weighed for yield and analyzed for moisture, oil, and protein contents. The gluten and water were separated by centrifugation. The mixture was transferred to 250-mL centrifuge bottles and centrifuged for 30 min at 6,000 × g in a refrigerated centrifuge (Sorvall Superspeed

TABLE I
Size and Density of Two Corn Hybrids Harvested
at Three Stages of Development^a

Property	Pioneer 3525	Pioneer 3394	Mean Values ^b
Test weight (lb/bu)			
Harvest 1	51.6	52.6	52.1c
Harvest 2	55.8	57.6	56.7b
Harvest 3	57.5	57.8	57.6a
Cultivar mean ^c	55.0b	56.0a	
Thousand-grain weight (g)			
Harvest 1	175	201	188c
Harvest 2	224	282	253b
Harvest 3	266	317	292a
Cultivar mean ^c	221b	267a	
Absolute density (g/cm ³)			
Harvest 1	1.19	1.22	1.20b
Harvest 2	1.23	1.24	1.24a
Harvest 3	1.23	1.23	1.23a
Cultivar mean ^c	1.22b	1.23a	

^a Harvests 1 and 2 represent corn samples at immature stages in growth. Harvest 3 represents corn at full maturity.

^b Mean values are pooled across both corn hybrids. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

^c Values followed by the same letter in the same row are not significantly different ($P < 0.05$).

TABLE II
Kernel Strength of Two Corn Hybrids Harvested
at Three Stages of Development^a

Property	Pioneer 3525	Pioneer 3394	Mean Values ^b
Breakage susceptibility (%)			
Harvest 1	5.21	3.70	4.46a
Harvest 2	4.59	3.24	3.91b
Harvest 3	4.54	4.14	4.34a
Cultivar mean ^c	4.78a	3.69b	
Stenvert hardness (cm)			
Harvest 1	12.2	11.9	12.0a
Harvest 2	11.7	11.0	11.3b
Harvest 3	11.5	11.1	11.3b
Cultivar mean ^c	11.8a	11.3b	

^a Harvests 1 and 2 represent corn samples at immature stages in growth. Harvest 3 represents corn at full maturity.

^b Mean values are pooled across both corn hybrids. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

^c Values followed by the same letter in the same row are not significantly different ($P < 0.05$).

RC2-B, Newton, CT). After centrifugation, the water was decanted and the protein was transferred from the bottles to a drying pan. The gluten was dried, weighed for yield, and analyzed for moisture, oil, and protein contents. The water was weighed for yield and analyzed for solids content. Both hybrids at each stage of maturity were steeped and milled three times in random order.

Proximate Analysis of Wet-Milled Products

Moisture contents of the whole grain samples were determined in duplicate according to Approved Method 44-15A (AACC 2000). Moisture contents of the specified ground fractions (starch, gluten, fiber, germ, and inseparables) were determined in duplicate according to AOAC method 14.004 (AOAC 1984). Yield and recovery data were calculated on a dry basis. Yields of dissolved solids in liquid fractions (steep 1, steep 2, and wash water) were determined in duplicate by placing a 10-mL fraction of the solution in an oven at 130°C for 16 hr and multiplying the weight difference by the total volume of solution. Crude free fat contents of the ground fractions were determined according to AOAC methods 14-084 and 14-085 (AOAC 1984) using the Goldfisch apparatus. Protein contents of the ground fractions were determined in duplicate according to CRA macro-Kjeldahl method A-18 (CRA 1986) and Tecator Kjeltex system (Tecator, Hoganas, Sweden).

Statistical Analysis

Means and Duncan *t*-test were computed by a two-way analysis of variance (ANOVA) using the general linear models procedure (SAS Institute, Cary, NC).

RESULTS AND DISCUSSION

Means for each of the grain quality parameters are presented in Tables I–II. Significant varietal and harvest differences were observed for all of the grain quality parameters measured.

Size and Density

The late-maturing hybrid (Pioneer 3394) yielded test weight values that were larger than those of the early maturing hybrid (Pioneer 3525). A significant trend was seen within hybrids. As the grain matured, test weight increased (Table I). Weller (1987) observed similar findings. As the moisture of the grain decreased (an indication of maturity increase), the values obtained for test weight increased. Thornton et al (1969) also saw a reduction in test weight resulting from harvesting corn at immature stages of development. According to Watson (1987), starch yield and other wet-milling properties are not appreciably affected by test weight unless it drops below 48 lb/bu. Grain from harvests 2 and 3 met the USDA

test weight standard for No. 2 corn and were comparable to test weight values of the mature grain samples used in research reported by Fox et al (1992). However, grain from harvest 1 in this study would not meet USDA test weight standard for No. 2 corn and would be discounted on this basis.

In this study, Pioneer 3394 yielded a significantly larger seed as measured by thousand-grain weight analysis than did Pioneer 3525 (Table I). A significant difference also was observed within hybrids. As the grain matured, the thousand-grain weight values increased. These findings indicate that as the corn plant is maturing, the kernels are increasing in size.

Between hybrids there was a significant difference in density, with Pioneer 3394 being the denser of the two (Table I). Within each hybrid there was a significant trend. As the grain matured from harvest 1 to harvest 2, absolute density increased. Although the grain became more dense with maturity, the values did not exceed those of average corn (1.260–1.270 g/cm³ (Hurburgh 1991). Because the density of the corn kernel is the sum of the densities of its components (Watson 1987) and more dense components are being deposited as the grain is maturing, these findings were expected.

The results for test weight, thousand-grain weight, and absolute density all can be attributed to the maturation process of the corn kernel. In the early development stages of the kernel, soluble cellular components such as soluble nitrogen, amino acids, sugars, and nucleotides are present in the kernel in large amounts. When dry matter is accumulating and the kernel is physiologically maturing, these components are metabolized into larger polymers such as starch and protein. These changes account for the more dense, large kernel size.

Kernel Strength

Among the early and late maturing hybrids there was a significant difference in breakage susceptibility with early maturing Pioneer 3525 being more susceptible to breakage (Table II). Within hybrids, Pioneer 3525 displayed a decreasing trend as the grain matured, while breakage susceptibility of the late maturing Pioneer 3394 fluctuated from less susceptible at harvest 2 to more susceptible at harvest 3. We believe that moisture content of the grain had a significant effect on these data. These results may be attributed to excess and depleted moisture causing the grain to become more brittle as well as ordinary differences seen in different hybrids.

According to Hurburgh (1991), the average breakage value for hand-harvested, air-dried corn is 3–4%, which was true for the samples in this investigation. These findings, however, are in contrast to those found by Weller (1987), who found moisture (an indicator for maturity) to have no effect on breakage susceptibility as measured by the Wisconsin breakage tester.

The early maturing Pioneer 3525 was significantly harder than the late maturing Pioneer 3394 (Table II). These results can be explained by realizing that hardness is closely related to the ratio of horny to flourey endosperm (Watson 1987), and hybrids vary in proportions of these components. Within hybrids, there was a significant difference in hardness, harvest 1 had a Stenvert hardness value greater than the other harvest 2 or harvest 3. These values are characteristic of soft to medium-soft grain, which is optimum for

TABLE III
Water Uptake of Two Corn Hybrids Harvested at Three Stages of Development^a

Property	Pioneer 3525	Pioneer 3394	Mean Values ^b
Water absorption index			
Harvest 1	0.236	0.265	0.250a
Harvest 2	0.171	0.147	0.159b
Harvest 3	0.160	0.167	0.164b
Cultivar mean ^c	0.189b	0.193a	
Moisture saturation point			
Harvest 1	0.568	0.617	0.591a
Harvest 2	0.440	0.465	0.452b
Harvest 3	0.411	0.418	0.414c
Cultivar mean ^c	0.473b	0.500a	

^a Harvests 1 and 2 represent corn samples at immature stages in growth. Harvest 3 represents corn at full maturity.

^b Mean values are pooled across both corn hybrids. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

^c Values followed by the same letter in the same row are not significantly different ($P < 0.05$).

TABLE IV
Proximate Composition^a for Two Corn Hybrids Harvested at Three Stages of Development^b

Harvest	Protein		Oil		Starch	
	3525	3394	3525	3394	3525	3394
1	7.3	7.5	3.3	3.1	62.6	62.9
2	6.8	7.5	3.1	2.8	62.9	63.1
3	7.3	7.2	2.8	2.6	63.4	64.0

^a Adjusted to 15.5% moisture content.

^b Harvests 1 and 2 represent corn samples at immature stages of growth. Harvest 3 represents corn at full maturity.

wet milling. As the grain matured, the endosperm became more dense and, in theory, would be less desirable for wet milling. These findings can be attributed to maturation of the kernel in size and density.

Water Uptake

In general, the late maturing Pioneer 3394 yielded the largest WAI (Table III). Within hybrids, a significant decrease in WAI was seen between harvest 1 and harvest 2. The WAI values for the immature harvests as well as the mature harvest were comparable to those obtained by Fox et al (1992), who analyzed 27 mature hybrids for this property.

A significantly larger moisture saturation point was obtained from Pioneer 3394 (Table III). Also, there was a significant trend within each of the hybrids wherein as the grain matured, the moisture saturation point decreased. Hsu et al (1983) found water absorption rate and kernel size correlated at $r = -0.53$ and attributed it to small kernels having more surface area per unit mass. The moisture saturation point data coincide with these findings.

The results from the water uptake measurements, water absorption index, and moisture saturation point are due to the maturation of the kernel. Initially, the kernel has more free sugars and amino acids which theoretically can bind more water. As the grain matures, these water-soluble components polymerize, making water binding more difficult.

Predicted Starch Yields and Comparison of Actual Yields

Based on the study by Fox et al (1992), test weight, Stenvert hardness, and WAI, along with grain protein content from the proximate analysis (Table IV), were used to predict starch yields due to the relatively higher values of $r^2 = 0.61, 0.51,$ and $0.50,$ respectively, compared with the other grain quality parameters (Table V). Data in Table V show that higher starch yields (>2% increase) would be expected as the corn matures, particularly between harvest 1 and harvest 2 within each cultivar, and there would not be a difference in starch yield between the late and early maturing hybrids.

Yields of Wet-Milled Products

The starch yield for both hybrids increased as the grain matured (Table VI). The starch yield increased significantly within each cultivar between harvest 1 and harvest 2 but did not increase between harvest 2 and harvest 3. There was no significant difference between

TABLE V
Predicted Starch Yields for Selected Corn Hybrids

Model	Pioneer 3525		Pioneer 3394	
	Predicted	Interpreted	Predicted	Interpreted
Test weight	Starch yield, % = 58.2 - 3.6 (% grain protein ^a) + 0.5 (test weight ^b) $r^2 = 0.61^{***}$			
Harvest 1	57.9	Low	57.5	Low
Harvest 2	61.6	High	60.0	High
Harvest 3	60.7	High	61.2	High
Stenvert hardness	Starch yield, % = 119.7 - 4.4 (% grain protein ^d) - 2.3 (Stenvert height ^b) $r^2 = 0.51^{**}$			
Harvest 1	59.6	Low	59.4	Low
Harvest 2	62.9	High	61.5	High
Harvest 3	61.2	High	62.5	High
WAI	Starch yield, % = 94.9 - 3.5 (% grain protein ^e) - 45.6 (WAI ^b) $r^2 = 0.50^{**}$			
Harvest 1	58.6	Low	56.6	Low
Harvest 2	63.3	High	60.7	High
Harvest 3	62.0	High	62.1	High

^a 7.3 for early maturing and 7.5 for late maturing.

^b Values in Tables I-III.

^c ** and *, significant at $P < 0.05$ and $0.01,$ respectively.

^d 6.8 for Pioneer 3525 and 7.5 for Pioneer 3394.

^e 7.3 for Pioneer 3525 and 7.2 for Pioneer 3394.

cultivars at all three harvests. The trend indicated that starch yield reached a plateau at the second harvest in the early maturing Pioneer 3525, whereas the late maturing Pioneer 3394 seemed to peak at harvest 3 (full maturity). This would be expected because the hybrids are characterized as early and late maturing. Therefore, starch yield would be expected to peak earlier in Pioneer 3525 than in Pioneer 3394. Between hybrids, there were no significant differences in starch yield at $P < 0.05.$ Based on the proximate composition data in Table IV, these results were expected. Starch yields at each of the maturity levels for both hybrids were comparable to the laboratory wet-milling experiment of Eckhoff et al (1993), who used fully matured grain and the starch tabling method for separation of starch and gluten and obtained starch yields averaging 64.8%. Yields also were comparable to industry, which recovers 67.5% starch (Anderson and Watson 1982).

Gluten yield within both hybrids decreased as the grain matured (Table VI), although differences were not significantly different at $P < 0.05.$ Between the two hybrids, there were no significant differences. Gluten yields for all samples within and between hybrids were 6.29-7.43% and were slightly low when compared with the laboratory milling study (9.9%) (Eckhoff et al 1993), but comparable to industry (5.8%) (Anderson and Watson 1982). The gluten yield suggest that protein content in the endosperm reaches its maximum level at harvest 1 and does not increase significantly thereafter. The trend of decreased gluten recovery coupled with the trend of increased starch recovery suggests that the starch level is increasing or maturing in such a manner as to increase the amount of starch recovered, thereby reducing the yield of gluten.

TABLE VI
Yield (% db) of Wet-Milled Products from Corn Harvested at Three Stages of Development^a

Fraction	Pioneer 3525	Pioneer 3394	Mean Values ^b
Starch			
Harvest 1	65.27	65.34	65.30b
Harvest 2	68.77	68.01	68.39a
Harvest 3	67.28	68.69	68.24a,b
Cultivar mean ^c	67.10a	67.35a	
Gluten			
Harvest 1	6.79	7.43	7.1a
Harvest 2	6.29	6.89	6.6a
Harvest 3	6.29	6.31	6.3a
Cultivar mean ^c	6.46a	6.88a	
Fiber			
Harvest 1	11.00	12.31	11.8a
Harvest 2	10.63	11.84	11.2a,b
Harvest 3	9.97	11.08	10.5b
Cultivar mean ^c	10.54b	11.74a	
Germ			
Harvest 1	4.49	4.34	4.4c
Harvest 2	5.36	5.02	5.2a
Harvest 3	4.94	4.75	4.8b
Cultivar mean ^c	4.93a	4.70b	
Inseparables ^d			
Harvest 1	0.8	0.7	0.8a
Harvest 2	0.6	0.6	0.6a
Harvest 3	0.6	0.7	0.7a
Cultivar mean ^c	0.7a	0.7a	
Total yields			
Harvest 1	99.8	102.2	101.0a
Harvest 2	102.1	101.6	101.9a
Harvest 3	99.2	100.0	99.8a
Cultivar mean ^c	100.4a	101.4a	

^a Harvests 1 and 2 represent corn samples at immature stages in growth. Harvest 3 represents corn at full maturity.

^b Mean values are pooled across both corn hybrids. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

^c Values followed by the same letter in the same row are not significantly different ($P < 0.05$).

^d Inseparables fraction from last 0.6 m of starch table.

Within both hybrids, fiber yields decreased with increased maturity in the grain (Table VI). The fiber yields of harvest 1 were significantly higher at $P < 0.05$ from harvest 3. As the other components of the maturing kernel are deposited, the surface area of the fiber becomes proportionately smaller. Between the two hybrids, the early maturing Pioneer 3525 yielded significantly less fiber than did late maturing Pioneer 3394. Visual observation of the two hybrids showed that Pioneer 3394 was always larger, so these results are not totally unexpected. All fiber yields in this study were comparable to the laboratory milling study (9.9) (Eckhoff et al 1993) and industry (11.5%) (Anderson and Watson 1982).

The germ yields within both hybrids were significantly different at $P < 0.05$ (Table VI). Grain from harvest 2 had a larger germ yield than grain from harvests 1 and 3, and harvest 3 produced larger germ yield than harvest 1. The results indicate that the embryo was not fully matured at harvest 1 and reached full maturity by harvest 2. The decrease in yield, therefore, does not suggest a decrease in the size of the germ but possibly a change in the nature of the kernel making the germ more difficult to recover. These changes could be related to the decrease in the total amount of fiber or a change in the endosperm or a change in the germ itself. This hypothesis would have to be verified with further studies. There were no statistical differences in germ yield between the two hybrids. The higher germ yield between the two hybrids probably could be explained by the differences in the hybrids with the early maturing Pioneer 3525 producing larger germ yield than Pioneer 3394. For all samples, germ yields were slightly lower than yields of the laboratory milling study (7.0) (Eckhoff et al 1993) and industry (6.6%) (Anderson and Watson 1982).

Inseparables yields (Table VI) were not significantly affected by harvest or hybrid at $P < 0.05$. This is not a fraction produced in industry, therefore no industry comparison could be made.

TABLE VII
Protein Content (% db) of Wet-Milled Products from Corn Harvested at Three Stages of Development^a

Fraction	Pioneer 3525	Pioneer 3394	Mean Values ^b
Starch			
Harvest 1	0.13	0.20	0.17a
Harvest 2	0.34	0.25	0.29a
Harvest 3	0.14	0.28	0.21a
Cultivar mean ^c	0.20a	0.24a	
Gluten			
Harvest 1	40.9	40.8	40.9a
Harvest 2	44.5	46.6	45.6a
Harvest 3	42.5	42.9	42.0a
Cultivar mean ^c	42.6a	43.0a	
Fiber			
Harvest 1	10.8	9.9	10.4a
Harvest 2	10.6	10.3	10.5a
Harvest 3	11.0	10.1	10.4a
Cultivar mean ^c	10.8a	10.0b	
Germ			
Harvest 1	11.0	11.6	11.3a
Harvest 2	11.2	12.6	11.9a
Harvest 3	11.4	11.7	12.0a
Cultivar mean ^c	11.2b	12.3a	
Inseparables ^d			
Harvest 1	0.17	0.13	0.15a
Harvest 2	0.21	0.64	0.42a
Harvest 3	0.25	0.83	0.30a
Cultivar mean ^c	0.21a	0.37a	

^a Harvests 1 and 2 represent corn samples at immature stages in growth. Harvest 3 represents corn at full maturity.

^b Mean values are pooled across both corn hybrids. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

^c Values followed by the same letter in the same row are not significantly different ($P < 0.05$).

^d Inseparables fraction from last 0.6 m of starch table.

Proximate Composition of Products

Protein contents of the wet-milled products are presented in Table VII. The results show that maturity (time of harvest) did not significantly affect the protein contents of any of the fractions. Protein contents of the starches were typical of those normally observed in industry (0.3%). However, the purity of gluten suffered, yielding lower protein values of 36–49% compared with other laboratory wet-milling studies that yielded protein values of 48–54%. Germ protein was significantly different ($P < 0.05$) for the two hybrids. The late maturing Pioneer 3394 contained more protein than the early maturing Pioneer 3525. Fiber protein content for Pioneer 3525 (10.9%) was significantly different from Pioneer 3394 (10.0%). The difference in germ and fiber protein between the two hybrids may indicate differences between the hybrids or the nature of milling properties between the two that result in different protein contents. Gluten, starch, and inseparables protein contents were not significantly different at $P < 0.05$ for hybrid.

Oil contents of the wet-milled products are presented in Table VIII. Germ oil content was significantly different at $P < 0.05$ for time of harvest. Harvests 1 and 2 produced more crude fat (53.1 and 53.7%, respectively) than harvest 3 (52.1%). The lower oil yield after harvest 3 may help explain the difference in germ yield from harvest 1 to harvest 3. The results suggest that oil content reaches a maximum after harvest 1 and the germ changes by the harvest 3, resulting in a lower germ oil yield. These results may also explain the significant difference in oil content in the gluten fraction between harvests 2 and 3. The significant differences in gluten and fiber oil contents between Pioneer 3525 and Pioneer 3394 is probably due to varietal differences. Fiber, gluten, starch, and inseparables oil contents were not significantly different at $P < 0.05$ for time of harvest or hybrid.

TABLE VIII
Oil Content (% db) of Wet-Milled Products from Corn Harvested at Three Stages of Development^a

Fraction	Pioneer 3525	Pioneer 3394	Mean Values ^b
Starch			
Harvest 1	0.07	0.07	0.07a
Harvest 2	0.06	0.06	0.06a
Harvest 3	0.04	0.10	0.07a
Cultivar mean ^c	0.06a	0.07a	
Gluten			
Harvest 1	4.9	3.4	4.2a
Harvest 2	3.7	2.7	3.2b
Harvest 3	3.2	3.5	3.3b
Cultivar mean ^c	4.0a	3.2b	
Fiber			
Harvest 1	3.1	2.4	2.7a
Harvest 2	3.1	2.6	2.8a
Harvest 3	3.3	2.2	2.8a
Cultivar mean ^c	3.1a	2.4b	
Germ			
Harvest 1	50.4	55.7	53.1a
Harvest 2	54.1	53.4	53.7a
Harvest 3	54.8	49.4	52.1b
Cultivar mean ^c	53.1a	52.8a	
Inseparables ^d			
Harvest 1	0.41	0.38	0.40a
Harvest 2	0.45	0.38	0.41a
Harvest 3	0.37	0.23	0.30a
Cultivar mean ^c	0.41a	0.33a	

^a Harvests 1 and 2 represent corn samples at immature stages in growth. Harvest 3 represents corn at full maturity.

^b Mean values are pooled across both corn hybrids. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

^c Values followed by the same letter in the same row are not significantly different ($P < 0.05$).

^d Inseparables fraction from last 0.6 m of starch table.

Comparison of Predicted Starch Yield to Actual Starch Yield

Starch yields (Table VI) from the actual milling of the grain showed an identical trend to the starch yields predicted by the mathematical model (Table V). Peak starch yield for Pioneer 3525 was observed at harvest 2, while the late maturing Pioneer 3394 peak starch yield was observed at harvest 3 (full maturity).

CONCLUSIONS

All grain quality parameters were significantly ($P < 0.05$) affected by hybrid and maturity. Test weight, thousand-grain weight, and absolute density increased with increased maturity. Late maturing Pioneer 3394 displayed greater values for all size and density parameters. Breakage susceptibility varied within hybrids. Late maturing Pioneer 3394 was less susceptible to breakage than Pioneer 3525. Kernel hardness increased with maturity. Pioneer 3394 yielded a harder grain than Pioneer 3525. Water absorption and moisture saturation point decreased with increased maturity. Pioneer 3394 absorbed more water than Pioneer 3525. Yield and purity of starch was not significantly ($P < 0.05$) affected by hybrid or maturity. The mathematical models were useful in interpreting the wet-milling characteristics of the grain at all three stages of development.

Although differences were observed in the grain quality parameters and some product yields and composition for hybrid and harvest, this study indicates that grain harvested as early as late milk line would not adversely affect the wet-milling industry. Starch yield and purity were comparable to that of industry and other laboratory wet-milling studies. Other coproducts obtained by this process also were comparable for yield and composition. However, based on the USDA quality standards for No. 2 yellow corn, which is typically used in wet milling, the farmer would be discounted for grain harvested as early as late milk line due to its low test weight value alone. Grain harvested as early as the dent stage would meet the test weight specifications for No. 2 yellow corn. Predictive models used in this study were effective at coupling protein composition and a grain quality attribute to predict trends in starch yield for both mature and immature grains before actual milling. Starch yield and purity for all of the grain samples, immature and mature, were not significantly affected by maturity, suggesting that the wet-milling industry could use the grain, which they would purchase at a discounted price due to low test weight, to recover starch and other coproducts comparable to mature grain purchased at full price.

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