Correlation of Starch Recovery with Assorted Quality Factors of Four Corn Hybrids

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ABSTRACT

A laboratory wet-milling process was developed for use in determining starch recovery of yellow dent corn. The effects of harvest moisture and drying air temperature on starch recovery were investigated for four hybrids. A split split-plot experimental design was used to evaluate the effects. Starch recovery was not significantly different among the hybrids; however, it did decrease as both harvest moisture and drying air temperature increased. Starch recovery was correlated with quantitative measures of assorted quality factors. Regression analysis found starch recovery to be a function of starch content, test weight, and ethanol-soluble protein.

The shift from natural drying of ear corn to artificial drying of shelled corn is one of the factors that has made the U.S. farmer one of the most productive and efficient in the world. However, the shift has not taken place without negative effects and corn quality problems related to artificial drying (Foster 1965).

One industry that has been hurt considerably by the vast movement toward artificial drying is the corn wet-milling industry (Wichser 1961). The amount of starch recoverable from a wet-milling process is inversely related to the severity of the drying treatment of the incoming corn. Traditionally, millers have relied on extremely limited and highly empirical evaluations of corn quality during purchasing to screen out corn dried under severe conditions (Freeman 1973). For example, the evaluations may consist of a visual inspection and a review of the information provided on the official grain inspection certificate. Nevertheless, some low-quality corn is purchased, resulting in a reduced production of starch.

To ensure continuing domestic and foreign markets and to improve the efficiency of wet milling, a simple, rapid, and reliable grain quality evaluation procedure is needed (Wichser 1961, Freeman 1973). The procedure should quantitatively relate corn quality to millability. Millability is defined as the ease with which kernel components are separated and purified by the wet milling process. For the purposes of the study, it was assumed that millability was highly correlated with starch recovery.

Wichser (1961) desired a better method than those presently in use for identifying and separating artificially dried corn. He proposed corn being placed into lots according to temperature and other drying conditions used. Freeman (1973) called for simpler and more reliable grain evaluation procedures and inclusion of quality factors not considered in the official grading standards. Both wanted a testing method that would give the wet miller an opportunity to practice selective buying of yellow dent corn.

An early attempt to relate a quality factor with the value of corn for wet milling was undertaken by Baird and co-workers (1950). They concluded that a test for viability with 2,3,5-triphenyltetrazolium chloride solution appeared to offer promise for the determination of industrial suitability of corn as offered for sale. However, a study by Gausman et al (1952) showed the correlation between the 2,3,5-triphenyltetrazolium chloride color test and starch recovery was "less apparent than roughly proportional." MacMasters et al (1954) later concluded that there was no correlation between the color test and starch recovery.

Also in 1954, Watson and Hirata reported a method for determining the potential millability of steeped grain by visually estimating, through magnification, the amount of starch released when thin slices of steeped corn kernels were brushed. Millability scores were obtained for numerous hybrids; however, no correlation was made with a starch recovery process.

Watson and Hirata observed in 1962 that viability of corn, as determined through seed germination, was reduced or destroyed by drying conditions that were less severe than those that adversely affect millability. As a result of this, they concluded that grain with high viability should invariably be suitable for wet milling. However, it should be noted that grain with low viability does not necessarily indicate poor millability, because viability measures some property of the germ whereas millability measures endosperm properties.

In 1964, Holaday reported a method for determining heat damage to dried corn. He also showed that prime starch yield (weight of starch and gluten recovered per weight of preprocessing dry matter) has an inverse relationship to his measure of heat damage, capacitance displacement value.

A study by Heusdens and MacMasters in 1967 found glutamic acid decarboxylase activity, germination, and the 2,3,5-triphenyltetrazolium chloride color test to be of limited use individually in detecting damage in corn caused by artificial drying. Nevertheless, they stated that a combination of the methods could be used to detect drying damage.

Freeman and Watson (1969) reported a simple, 2–3 hr method for demonstrating starch and protein separation in graduated cylinders. The method gives a large starch deposit in the bottom of a cylinder, with sharp delineation between the starch layer and the less dense gluten for corn of high millability. However, they did not establish any quantitative relationships between the height of the deposit and starch recovery from a wet-milling process.

Two tests of corn for starch yield (percent of recovered starch in relation to the processed corn dry matter) were reported by Lasseran (1973). The first method, which required 24 hr to complete, compared extracted ethanol-soluble proteins to starch yield. He found an increasing linear relationship between starch yield and protein solubility until approximately 67%, where the starch yield became essentially constant.

The second corn starch yield method required 30 min. In this method, undenatured, water-soluble, thermosensible proteins were extracted from the corn samples and precipitated in a boiling water bath. Absorbance readings were taken on the supernatants using a colorimeter. Corn dried in the range of ambient temperature to 90° C did not experience a denaturation of water-soluble proteins. However, at some critical temperature between 90 and 100° C, the water-soluble proteins in the corn denatured and lowered the turbidity to some constant value.

Vojnovich et al (1975) observed no significant correlation between starch recovery and test weight. However, they did find a high correlation ($R = +0.93$) between starch recovery and drying temperature. They also claimed that corn suffering high damage from the combine gave a starch recovery comparable to that of corn harvested with little damage. Nevertheless, their results showed mechanical damage has less impact than drying air temperature on starch recovery.

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In 1979, Brown et al reported several quantitative relationships between quality factors and millability of corn. The first related percent starch recovery to a steeping index value through use of a regression equation. Their steeping index was based on a visual inspection of steeped kernel sections to determine the softening of the vitreous endosperm. The index value ranged from 100 to 300. A value of 200 or more for any corn sample indicated satisfactory milling performance in terms of starch recovery and normal starch-gluten separation.

Brown and co-workers (1979) then used the steeping index as a standard measure of starch recovery against which to relate such quality factors as stress cracks, viability, and test weight. They concluded that viability, test weight, and stress crack analysis of dried corn, individually, did not accurately predict wet milling performance. They also stated that taken together the measurements of the quality factors could be used to detect corn with different drying damage levels that adversely affect millability. However, they did not elaborate on this point with theories or prediction equations.

Wight (1981) studied the relationship between various quality factors and wet millability as they are affected by increased drying temperatures. He reported that a decrease in water solubility for any corn protein fraction cannot be used as a rapid detection method for drying damage as it related to starch yield. He also found that germination and stress crack formation are unreliable indicators of decreased starch yield as drying air temperature increases.

None of the previous attempts to develop a testing procedure for evaluating wet-milling quality of incoming corn has been widely accepted. All have lacked rapidity or sufficient reliability. Therefore, this study was initiated to develop a laboratory wet-milling process to accurately and efficiently determine starch recovery for 48 treatments of yellow dent corn; to determine the effects of hybrid, harvest moisture, and drying air temperature of corn on starch recovery; and to correlate starch recovery with quantitative measures of quality factors to develop a rapid and reliable method for predicting starch recovery.

MATERIALS AND METHODS

Four hybrids of shelled, whole kernel yellow dent corn were grown in Champaign County, IL. Samples of the hybrids were harvested at each of three moisture contents and stored at 4° C in sealed containers. The samples were dried within 48 hr after harvesting with one of four drying treatments. Four replicates of each were prepared such that the total number of samples was 192, and each sample weighed approximately 2.5 kg.

Vitreous-to-floury endosperm ratios for the four hybrids were established in personal consultation with R. Troutman (Champaign County Seed Co., St. Joseph, IL), J. Hiser (Illinois Foundation Seeds, Inc., Tolono, IL), and D. G. White (Department of Plant Pathology, University of Illinois, Urbana). Hybrid 1, FR27 × FRMo17, was characterized as having a medium low vitreous-to-floury endosperm ratio. Hybrid 2, B73 × LH38, was characterized as having a low ratio, whereas hybrids 3 (LH51 × LH119) and 4 (FR27 × Va22) were characterized as having medium high and high ratios, respectively.

Harvesting

Each hybrid was hand-picked and hand-shelled. A 37-kg (1.33 bu) sample was harvested at high, medium, and low moisture contents of approximately 30, 24, and 18% (wb), respectively. A Dickey-john GAC II was used to determine moisture content. More accurate harvest moisture contents (Table I) were determined using the air-oven method given below.

Drying

Corn samples were dried with one of four treatments. The dryers, the same as used by Gunasekaran and Paulsen (1985), were equipped with a fan, a heater, and a temperature controller. Temperatures of the drying air were set at ambient, 49, 71, and 93° C, and the rate of air flow was 2.0 m^3/min per cubic meter of corn.

The grain bed, containing about 5 kg of corn, was mixed intermittently during drying to obtain uniform drying. The removable dryer tray with corn was periodically weighed to calculate moisture loss.

After the grain reached 14% (wb) moisture, based on the initial GAC II moisture measurement, each sample was placed in a nylon mesh bag. Each mesh bag was then sealed in a plastic bag and left at ambient room temperature to cool before storage.

After cooling, each sample was removed from the plastic bag and placed in an environmental chamber at 20° C and 70% relative humidity for a minimum of four weeks before further testing.

Quality Factor Measurement

Because the methods used for quality factor measurement and the resulting data were described by Weller et al (1987), they are only briefly outlined here.

Bulk density (test weight) was determined for the corn samples using the U.S. Department of Agriculture Federal Grain Inspection Service (FGIS) standard method (1979).

Stress crack percentages were determined using the Thompson and Foster (1963) method modified by Paulsen and Hill (1985). A sample of 50 whole kernels was tested for each drying treatment. Any kernel with one or more internal fissures was considered stress cracked.

Breakage susceptibility for the various dried corn samples was determined using both a Wisconsin breakage tester (WBT; serial no. C017P) and a model CK-2M Stein breakage tester (SBT). Samples of 200 g of whole, unbroken kernels were used in the WBT, and 100-g samples were used in the SBT.

Breakage susceptibility found using the WBT was determined as the percent ratio of the sample weight passing through the 4.76-mm sieve to the initial sample weight tested (Gunasekaran and Paulsen 1985). SBT test procedures followed those given by the American Association of Cereal Chemists method 55-20 (AACC 1983).

Kernel density was calculated from the weight and volume of 50 kernels. Fifty-kernel volume was determined using 99% (v/v) ethanol in the column test described by Paulsen and Hill (1985).

Resistance was determined with a procedure and ohmmeter similar to that of Whitten and Holaday (1957). The ohmmeter electrode was mounted on the load cell of a Project 10 Universal Materials testing machine and mechanically lowered into a plastic sample cup. Use of the testing machine provided a consistent pressure application of 551.6 kPa (80 psig) on each 250-g corn sample. Each corn sample was connected to a +15 VDC power supply with a 106.8 kΩ resistor in series. Resistance for each sample was calculated by measuring the voltage drop across the resistor and applying Kirchoff's Voltage Law to the circuit.

Capacitance measurements were made using a Motomco model 919 automatic moisture meter. The 250-g samples used were the same well-mixed samples used for resistance measurement. Each sample was placed in the sample cell of the moisture meter, and a capacitance reading in counts was rapidly taken. A count represented 0.013 picofarads.

Capacitance displacement values were calculated by measuring the number of graph divisions along the capacitance axis (x-axis) from a plot of the resistance versus capacitance values for each sample to the linear regression line ($Y = 9.913 - 0.0026X$) of the resistance versus capacitance values for samples dried with ambient

TABLE I
Individual and Mean Harvest Moisture Contents for Each Hybrid as Determined by the Oven Method

Harvest Moisture Level	Observed Harvest Moisture (% wb)				
	Hybrid				
	1	2	3	4	Mean
Low	18.9	17.2	19.5	20.2	19.0
Medium	24.0	21.9	24.2	24.0	23.5
High	33.9	29.6	29.5	29.6	30.7

air at all harvest moistures (Holaday 1964). The plotted capacitance values were meter readings from the Motomco moisture meter in counts. The plotted resistance values were $\log_{10}$ resistance measurements calculated for each corn sample using the 106.8 kΩ resistor and its subsequent voltage drop. Any displacement value calculated at less than zero was corrected to a value of zero. After correction, all the values were referred to as corrected capacitance displacement values.

Germination percentages for the dried corn samples were found using standard analytical method A-24 of the Corn Industries Research Foundation (CRAI 1983). Each sample had 100 whole, unbroken kernels immersed in a sodium hypochlorite solution for 3 min prior to incubation.

Crude protein determinations were made using the Association of Official Analytical Chemists semiautomated method 12 (AOAC 1980). Forty 125-ml volumetric tubes 26 mm in diameter were used with a Techne DG-1 block digester. Digestion time was modified to a 90 min of preheating and 120 min of heating at 410° C in order to obtain total breakdown of organic matter. Sample size was 0.5000 g of ground corn. Percent protein was expressed as 100 times the protein weight (6.25 times the nitrogen weight) divided by the dry matter weight for each sample.

Ethanol-soluble protein determinations were performed using the procedure of Godon and Petit (1971) for estimating the denaturation of protein in corn samples. Extraction of the protein was carried out in 15 ml glass serological tubes with an aqueous solution of 85.5% (v/v) ethanol and 1.25% (v/v) acetic acid. The tubes were sealed, shaken, and laid in a horizontal position in a vertical disk agitator.

The solution containing the ethanol-soluble proteins was recovered after agitation and centrifugation. Protein content determinations were made on the supernatants. The results were expressed as extracted protein weight as a percent of the weight of protein in the original corn sample with all weights on a dry basis.

Near-infrared absorbance was determined for the ground whole kernel corn samples at six wavelengths (2,310, 2,230, 2,180, 2,100, 1,940, and 1,680 nm). A Dickey-john Instalab 860 NIR product analyzer measured the absorbance as $\log_{10}$ reciprocal reflectance. Procedures for the operation and data collection followed instructions of the manufacturer (Dickey-john Corp., Auburn, IL).

Wet-Milling Process

The laboratory-scale wet-milling process was derived by combining and modifying the steeping method of Watson et al (1955) and the milling method of Watson et al (1951).

The steeping medium contained 1.5% (w/w) lactic acid and sufficient potassium metabisulfate (1.63 g/L of steep water) to give a sulfur dioxide concentration of 0.1% (w/w) in tap water. Potassium hydroxide was used to adjust the mixture to pH 3.7. Lactic acid polymers present in the concentrated reagent were eliminated by diluting to 20% (w/w) with distilled water and heating for 12 hr at 95° C prior to use in the steeping water.

Samples of corn containing 350 g of dry matter were placed in 2-qt widemouthed glass jars. The jars were arranged in batteries of eight and immersed in a water bath maintained at 53° C. The mouth of each jar was closed with a no. 14 rubber stopper fitted with two rigid plastic tubes. One tube extended to the bottom of the jar and the other just through the stopper. The longer tube in each jar served as the inlet and the shorter tube as the outlet. A small plastic netting was fitted over the shorter tube to prevent clogging.

The eight jars of a battery were connected together by attaching flexible plastic tubing to the rigid tubes. The tubing ran from a reservoir to the jars, connecting them in series, and then through a small Cole-Palmer model 7553-00 peristaltic pump before returning to the reservoir. Suction was used to fill each battery of eight jars by drawing from 22.7 L (6 gal) of steep water in the reservoir. After filling, the pump was connected to an interval timer to periodically circulate steep water through the grain and reservoir for about 15 min out of every 2 hr for a total steeping time of 40 hr.

After steeping, the steep water was drained from each jar. A 200-g sample of drained steeped corn was weighed for the milling operation. The remaining sample was stored in a plastic bag for moisture determination.

The first step in the milling operation was degermination, which was carried out in a Waring commercial blender model 5011. The agitator blades in the 800-ml bowl had been reversed so as to impart more impacting action and less cutting. The blender motor was operated on the low setting through an autotransformer set at 40% of the 120 V line voltage. The bowl was charged with 250 ml of distilled water for each 200-g corn sample, and blending lasted for 2 min. Slurry from the degermination grind was suitable for germ recovery. However, in the milling operation of this study, the germs were retained with the fiber fraction.

Fiber separation was initiated by pouring the slurry onto a U.S. standard sieve no. 230 (63 μm square mesh spacing) fitted onto an empty 4.73 L (5 qt) plastic container. Coarse material collected on the sieve was washed once with 500 ml of water before it was transferred to a Quaker City model 4E grinding mill.

The mill ground the fraction of steeped corn retained on the no. 230 sieve to physically break down pieces of vitreous endosperm that were softened during steeping but not broken during the degermination grind. Inconsistent grinding pressure between samples would have altered the number of starch granules released from the protein matrix of the endosperm. Therefore, the fine grinding plates of the mill were set to consistently just touch each other and resist movement by hand.

The material in the mill was ground with 500 ml of water added slowly during the grinding procedure. The discharge from the mill was returned to the sieve and washed with 1 L of water. The material remaining on the sieve was considered the fiber fraction. It was removed and dried at 103° C for 72 hr to determine the fiber fraction dry weight. The filtrate through the sieve was considered the mill starch. It was placed in a plastic 3.8 L jar and held for 15 hr at 4° C. During storage, the starch and gluten settled to the bottom of the jar.

The final milling step, separation of the mill starch into starch and gluten fractions, began by adjusting the specific gravity of the mill starch to 7–8° Baumé (1.051–1.058 sp gr). The adjustment was made by decanting off approximately 1,400 ml of the supernatant water in each jar. The starch and gluten were then resuspended in the remaining water to give the desired Baumé solution. This suspension was fed onto the upper end of a starch table in a steady stream. The starch tables were 6.1-m (20-ft) long pieces of 12.7-cm (5-in.) galvanized steel guttering. One end of each gutter was closed and raised 5.08-cm (2-in.) higher than the open end so that a slope of 8.33 mm/m was maintained.

The rate of flow of mill starch (approximately 250 ml/min) allowed starch to settle out and the gluten suspension to flow off at the open end of the table. Consistent flow rates between mill starches were necessary to allow starch granules to settle in the same relative location on the table. After starch was deposited on the table, adhering protein was flushed from the starch surface by passing 1 L of decanted mill starch water over the table at a rate four times faster than the flow rate of the mill starch.

Another washing of the starch on the table followed using the remaining decanted mill starch water diluted to 1,000 ml with distilled water at the faster flow rate. The wash tailings, including all the washings scraped from the last 0.5 m of the gutter, were combined with the gluten overflow for disposal. The starch fraction consisted of what remained on the table. The fraction on the table was allowed to air-dry for at least 45 min to ease its removal. It was removed by scraping towards the effluent end, washing the scrapings out with water, and reslurrying the fraction with a total of 1–3 L of water.

Reslurried starch fractions were placed in tared plastic 3.8-L jars and their weights recorded. Dry weights were calculated for the starch fractions using percent total solids as determined by the procedure described below. Samples of the solids in each starch slurry were prepared by filtering 300 ml of slurry with Whatman no. 50 paper fitted in a Buchner funnel. The samples of the starch slurry solids were then dried at 40° C in an air oven before their starch contents were determined.

Moisture Content

The AACC air oven method 44-15A (AACC 1983) was used to determine the moisture content of harvested corn, dried corn, and drained steeped corn.

Total Solids

A 50-ml sample from each of the starch fraction slurries was precisely placed in a tared aluminum weighing pan for determination of an initial slurry sample weight. After drying at 103° C for 72 hr, the pans were reweighed. Percent total solids was computed as the ratio of slurry sample dry matter to the initial 50-ml slurry sample weight times 100. Total starch fraction dry weight was the percent total solids times the total starch fraction slurry weight.

Starch Content

Starch content was determined with procedures described by Weller (1987). Solubilization of the starch began by mixing 0.5 g of each ground corn sample or each recovered starch fraction with a Tekmar SDT 1810 Tissumizer in 100 ml of 0.5*N* potassium hydroxide for 1 min. The mixture was then allowed to magnetically stir for 30 min before dilution to 200 ml with distilled water. A 25-μl sample from each 200 ml of solution and 25 μl of an amyloglucosidase preparation were injected into a Yellow Springs Instrument model 27 industrial analyzer equipped with a dual injection module for glucose analysis. The analyses followed instructions of the manufacturer (Yellow Springs Instrument Co., Inc., Yellow Springs, OH).

Whole kernel starch contents were calculated as the ratio of grams of starch (0.9 times the glucose weight) per gram of corn dry weight. Starch contents for recovered starch fractions were calculated as the ratio of grams of starch (0.9 times the glucose weight) per grams of starch fraction dry weight.

Starch Recovery

Starch recovery was determined for each 200 g of drained steeped corn sample. The recovery was calculated as the ratio of the total weight of starch recovered from wet milling to the total weight of starch present in the corn. The total weight of the starch recovered for each sample was calculated by multiplying the starch fraction dry weight by the percent starch in the recovered starch fraction. The total weight of starch present in each corn sample was calculated as the product of the percentage of starch content and the total dry matter weight in the 200-g drained steeped corn sample. Solubles lost during steeping were not accounted for in the dry matter weight of the drained steeped corn samples.

RESULTS AND DISCUSSION

Starch Recovery

A split split-plot experimental design was used to evaluate the effect of hybrid, drying air temperature, and harvest moisture on yellow dent corn starch recovery. Orthogonal comparisons were performed on both the main effects and interactions. All statistical statements for significance were made at the 5% probability level.

Corn hybrid 2, a popular commercial hybrid, served as the control in the hybrid comparisons. As the softest hybrid in this study and because of its large content of floury endosperm, hybrid 2 was hypothesized to have the best millability.

The calculation of starch recovery required determination of whole kernel starch contents; these are presented in Table II. The mean starch content of hybrid 2 was 67.4%, which was significantly less than the means of the other three hybrids (Table III). The starch content means for hybrids 1, 3, and 4 were 69.3, 70.0, and 69.3%, respectively.

Harvest moisture had no significant effect on the starch content values among the four hybrids. The means at high, medium, and low harvest moisture levels among the four hybrids were 69.1, 68.9, and 69.0%, respectively. Increasing drying air temperatures increased starch content means among hybrids, linearly, quadratically, and cubicly from 68.6% at 22° C to 70.3% at 93° C.

The starch recovery means for all levels of hybrid, harvest moisture, and drying air temperature are given in Table II. The mean starch recovery for hybrid 2 was 97.6%. It was higher but not significantly different than the means of 95.9, 96.6, and 96.9% for the hybrids 1, 3, and 4, respectively (Table III). A significant linear increase in starch recovery from 96.0 to 97.7% was observed among hybrids as harvest moisture decreased. Increasing drying air temperatures resulted in a significant linear and quadratic decrease in starch recovery among hybrids. Starch recovery decreased from 99.0 to 92.4% as drying air temperature increased from 22 to 93° C.

Normalized starch recoveries were calculated in addition to the previous recoveries due to the significant increases in starch content with increasing drying air temperature. The corresponding hybrid starch content mean was used for each sample rather than its own observed starch content in calculation of the normalized starch recoveries. The normalized starch recovery means are presented in Table II.

Normalized starch recovery means among hybrid and harvest moisture were equivalent to nonnormalized starch recovery means (Table II). The range of the normalized starch recovery means (4.2%) among drying air temperatures was less than the range of the nonnormalized starch recoveries (6.6%). A less precipitous decrease in starch recovery with increasing drying air temperature was also observed with the normalized values (0.059%/ 1° C) when compared to the initial starch recoveries (0.093%/ 1° C). However, the decrease in normalized starch recovery from 98.2 to 94.0% due

TABLE II
Starch Content and Starch Recovery Means for Each Hybrid, Harvest Moisture, and Drying Air Temperature Level

	Starch Quality Factors		
Variable Level	Content (% db)	Recovery (%)	Normalized Recovery (%)
Hybrid			
1	69.3	95.9	95.8
2	67.4	97.6	97.5
3	70.0	96.6	96.4
4	69.3	96.9	96.9
Harvest moisture			
Low	69.0	97.7	97.6
Medium	68.9	96.6	96.3
High	69.1	96.0	96.0
Drying air temperature, °C			
22	68.6	99.0	98.2
49	68.0	98.7	97.3
71	69.2	96.9	97.2
93	70.3	92.4	94.0

TABLE III
Partial Analysis of Variance for the Effects of Hybrid, Harvest Moisture, and Drying Air Temperature on Starch Content, Starch Recovery, and Normalized Starch Recovery Means

		Mean Squares		
Source	df	Content	Recovery	Normalized Recovery
Hybrid (H)[a]	3			
H2 vs. H1[a]	1	83.832*[d]	63.603	62.465*
H2 vs. H3[a]	1	159.032*	22.591	26.055
H2 vs. H4[a]	1	91.104*	9.614	6.767
H*Replicate (R)	12	2.516	21.917	9.689
Harvest moisture (M)[b]	2			
Linear[b]	1	0.193	93.862*	83.013*
Quadratic[b]	1	0.841	3.399	10.111
H*M*R	24	1.335	7.935	5.488
Drying air temperature (D)[c]	3			
Linear[c]	1	94.797*	1141.252*	401.306*
Quadratic[c]	1	35.286*	207.522*	61.654*
Cubic[c]	1	9.304*	3.476	35.137*
H*M*D*R	108	1.676	9.876	6.047

[a] H*R used as error term.
[b] H*M*R used as error term.
[c] H*M*D*R used as error term.
[d] * Significant, 5% level.

TABLE IV
Correlation Coefficients for Yellow Dent Corn Quality Factor Measurements and Starch Recovery

Factor[a]	TWGT	KD	SBT	WBT	EMC	GERM	STSCK	ESP	STRCH	PROT	CAP	RES	F2310	F2230	F2180	F2100	F1940	F1680	CCDV	SRC
TWGT	+1.00																			
KD	+0.73	+1.00																		
SBT	+0.10	+0.05	+1.00																	
WBT	+0.40	+0.13	+0.69	+1.00																
EMC	−0.02	+0.17	−0.47	−0.60	+1.00															
GERM	+0.31	+0.40	−0.50	−0.49	+0.66	+1.00														
STSCK	−0.13	−0.24	+0.46	+0.70	−0.45	−0.58	+1.00													
ESP	+0.11	+0.27	−0.44	−0.39	+0.60	+0.54	−0.32	+1.00												
STRCH	+0.04	−0.09	+0.12	+0.25	−0.23	−0.39	+0.20	−0.24	+1.00											
PROT	−0.41	−0.43	−0.07	−0.26	+0.15	+0.04	−0.09	−0.25	−0.20	+1.00										
CAP	−0.02	+0.19	−0.56	−0.69	+0.87	+0.72	−0.61	+0.51	−0.30	+0.15	+1.00									
RES	+0.09	−0.23	−0.06	+0.19	−0.58	−0.11	+0.16	−0.27	+0.01	0.00	−0.43	+1.00								
F2310	+0.65	+0.49	+0.57	+0.68	−0.35	−0.17	+0.25	−0.32	+0.03	−0.26	−0.41	−0.06	+1.00							
F2230	+0.63	+0.48	+0.58	+0.68	−0.35	−0.18	+0.26	−0.33	+0.03	−0.25	−0.41	−0.08	+1.00	+1.00						
F2180	+0.63	+0.48	+0.57	+0.68	−0.35	−0.17	+0.26	−0.32	+0.03	−0.25	−0.41	−0.07	+1.00	+1.00	+1.00					
F2100	+0.65	+0.49	+0.57	+0.68	−0.35	−0.17	+0.26	−0.32	+0.05	−0.28	−0.41	−0.06	+1.00	+1.00	+1.00	+1.00				
F1940	+0.69	+0.51	+0.46	+0.59	−0.24	−0.04	+0.15	−0.27	+0.02	−0.25	−0.30	−0.06	+0.97	+0.97	+0.97	+0.98	+1.00			
F1680	+0.64	+0.51	+0.56	+0.66	−0.30	−0.14	+0.24	−0.29	0.00	−0.25	−0.37	−0.11	+0.99	+1.00	+0.99	+0.99	+0.97	+1.00		
CCDV	−0.06	+0.07	+0.61	+0.44	−0.28	−0.57	+0.39	−0.26	+0.28	−0.15	−0.52	−0.51	+0.44	+0.45	+0.45	+0.44	+0.35	+0.45	+1.00	
SRC	+0.24	+0.20	−0.31	−0.26	+0.37	+0.50	−0.32	+0.45	−0.65	−0.02	+0.42	+0.01	−0.12	−0.13	−0.13	−0.14	−0.08	−0.10	−0.44	+1.00

[a] TWGT, test weight (kg/m^3); KD, Kernel density (g/cm^3); SBT, Stein breakage susceptibility (%); WBT, Wisconsin breakage susceptiblity (%); EMC, Equilibrium moisture content (% wb); GERM, Germination (%); STSCK, Kernels stress cracked (%); ESP, Ethanol soluble protein (%); STRCH, Starch content (%); PROT, Protein content (%); CAP, Capacitance (pf); RES, Resistance (Ω); F2310, Absorbance at 2,310 nm; F2230, Absorbance at 2,230 nm; F2180, Absorbance at 2,180 nm; F2100, Absorbance at 2,100 nm; F1940, Absorbance at 1,940 nm; F1680, Absorbance at 1,680 nm; CCDV, Corrected capacitance displacement value; SRC, Starch recovery (%).

TABLE V
Analysis of Variance for the Starch Recovery Regression Equation for Lack of Fit

Source	df	MS	*F* Value
Regression[a]	4	582.6579	69.62*[b]
Residual	187		
Lack of fit[c]	43	1.0467	0.10
Pure error	144	10.5558	
Total	191		

[a] Residual MS was error term.
[b] * Significant, 5% level.
[c] Pure error MS was error term.

to increasing drying air temperature was found to be significant (Table III), not only linearly and quadratically, as with the nonnormalized starch recoveries, but also cubicly.

Overall consideration of the statistical analyses indicated that the higher the moisture content and the higher the drying air temperature, the more severe the reduction in recoverable starch. Evidently, the combination of high temperature and high moisture conditions damaged the endosperm protein and prevented its solubilization during steeping such that the starch was not set free during wet milling (Wall et al 1975, Wight 1981).

This protein damage may have been due to a chemical change. Examples of chemical changes such as the formation of hydrogen and nonpolar bonds within the matrix proteins would make the overall structure of the matrix more rigid and interconnected (Aurand and Woods 1973, Watson 1984). Such changes have been alluded to by previous researchers. Watson and Hirata (1962) theorized that a specific protein fraction that releases starch granules when dissolved by steep water is insolubilized by high temperature drying. MacMasters (1962) reported that irreversible biochemical changes analogous to case hardening occur in the protein matrix of the outer endosperm cells during high-temperature drying.

Starch Recovery Prediction

Corn for use by the milling industries is purchased on the basis of various quality factor measurements. No knowledge of the drying or storage history of the corn accompanies it. The decision for its purchase and subsequent use in milling processes must be made based on easily measurable or visual quality factors. The determination of which quality factors highly correlate with wet milling performance is the first step towards improving the purchase decisionmaking process by millers (Freeman 1973).

Table IV presents the correlation matrix for the quality factor and starch recovery measurements made during this study and by Weller et al (1987). The quality factors of starch content, germination, ethanol-soluble protein, corrected capacitance displacement value, capacitance, and equilibrium moisture content had the greatest relative correlation coefficients of −0.65, +0.50, +0.45, −0.44, +0.42, and +0.37, respectively, with nonnormalized starch recovery. Changes in the quantitative measurement of these quality factors, except starch content, were related to protein damage resulting in large extent from increased drying air temperature (Weller et al 1987). The lack of any correlation coefficient with an absolute value greater than 0.70 was due to the varietal differences within any harvest moisture and drying air temperature combination.

The development of a prediction equation for starch recovery based only on quality factor measurements utilized a computerized stepwise regression program. The starch recovery and quality factor measurements used were those reported in this study and by Weller et al (1987). The equation giving the best fit was:

$$Y = 176.599 - 2.177(ST) + 0.038(TW) - 0.137(EP)^2 + 0.07(ST)(EP);$$

where Y was the predicted starch recovery (% of total starch), ST was the whole kernel starch content (% dry basis), TW was the test weight (kg/m^3), and EP was the whole kernel protein fraction soluble in an aqueous solution containing 85.5% (v/v) ethanol and 1.25% (v/v) acetic acid (% of total protein).

"Lack of fit" statistical significance was not found for this equation (Table V); however, the coefficient of multiple determination (R^2) was only 0.598. The significance of hybrid effects on the quality factor measurements of Weller et al (1987) most likely contributed to the low R^2 value.

Many previous attempts, as noted in the introductory paragraphs and in this study have been carried out in an effort to find at least one quality factor that is independent of variety. Considerable evidence has been accumulated to indicate that changes in protein conformation and corn wet millability occur as corn drying air temperatures increase, especially at high harvest moisture (Weller 1987). As research efforts to learn more about the nature of corn proteins continue, the possibility exists for discovery of a specific protein functional group or interprotein relationship that correlates with starch recovery. After this is elucidated, a reliable method can be developed for predicting starch recovery in less than 5 min.

CONCLUSIONS

The development of a laboratory wet-milling process required precision in the release of starch granules from the protein matrix

and in the separation of starch from protein in the mill starch. To achieve the precision, the maintenance of constant and consistent pressure on the grinding plates of the Quaker City mill and consistent flow rate of mill starch onto the starch tables was necessary.

Starch recovery from the corn samples was not significantly different among the hybrids. However, starch recovery decreased as both harvest moisture and drying air temperature increased, as previous investigators, cited in the introductory material, have demonstrated.

The highest correlations between starch recovery and quality factor measurements were found for the quality factors that responded to increased drying air temperatures, especially at high moisture levels. Regression analysis found starch recovery of yellow dent corn to be a function of starch content, test weight, and ethanol-soluble protein. Nevertheless, a rapid and reliable method for predicting starch recovery was not achieved in this study, since test weight was the only quality factor that could be measured in less than 5 min.

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