SOME WET-MILLING PROPERTIES OF ARTIFICIALLY DRIED CORN[1]

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ABSTRACT

Shelled corn artificially dried in two types of dryers, batch and continuous, has been examined for suitability for starch production by use of a laboratory procedure that measures the relative resistance to release of starch in steeped corn in wet-milling. Variables studied included drying temperature, initial grain moisture, relative humidity, and air-flow rate. Corn dried at 180°F. (82.2°C.) or higher showed evidence of reduced millability. Neither initial moisture of grain (batch dryer) nor air-flow rate (continuous dryer) has significant effect on milling results. High relative humidity in the batch dryer increased the degree of damage sustained by the corn dried at 180°F. (82.2°C.).

Viability of the grain was reduced or destroyed by drying conditions less severe than those that adversely affected millability. The temperature which produced a significant drop in viability was found to be a function of the initial moisture of the grain and the relative humidity and flow rate of the drying air. A temperature of 140°F. (60°C.) destroyed the viability of 32%-moisture corn, but 160°F. (71.1°C.) was the lowest temperature that destroyed viability of 21%-moisture corn. Grain dried so as to preserve viability should invariably be suitable for wet-milling.

Majel M. MacMasters and co-workers recently published extensive data which show that shelled corn dried in air at 180°F. (82.2°C.) or higher was damaged with respect to its use in starch production (6). Damage to the corn during drying was indicated by reduced starch yield and increased protein in recovered starch. Their data on residual starch in fiber fractions from the wet-milling tests indicated that more starch was retained in fiber from most corn samples dried at 200°F. (93.3°C.) and many of the samples dried at 180°F. Usually, the first and most apparent effect of processing heat-damaged corn in a wet-milling plant is increase in the amount of the fine-fiber fraction and its starch content. This results in lower starch yield, because of the presence of endosperm particles which apparently are rendered resistant to milling by high temperature during drying. The data presented in this paper were obtained in order to measure endosperm resistance to grinding in two groups of artificially dried corn samples by use of a laboratory wet-milling procedure designed specifically for this purpose (9). Kernel viability and solubles extractable with sulfur-dioxide solution have also been examined in an attempt to find other tests that might correlate with milling results.

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Materials and Methods

Preparation of Corn Samples. Two sets of dried shelled corn samples have been used in this work. The first set was dried in a small-scale batch-type dryer at the University of Illinois, described by Gausman *et al.* (3). The second set was dried in a small-scale continuous-type column dryer used in corn-drying experiments at Cargill, Inc., Minneapolis, Minn. The corn dried in the batch dryer was Illinois hybrid No. 1277 grown at Urbana, Illinois, in 1952. It was picked at two moisture levels, 32 and 21%, shelled and stored at 40°F. (4.4°C.), removed from storage, and dried to final moisture levels of 8 to 12%. Grain at each initial moisture level was dried in air of 15 and 40% r.h. at temperatures of 120°, 140°, 160°, and 180°F. (48.9°, 60°, 71.1°, and 82.2°C.). The 32%-moisture corn was also dried at 200°F. (93.3°C.). Moisture was introduced into the batch dryer to maintain given conditions of relative humidity in the drying air. The levels of 15 and 40% r.h. were chosen to accentuate the effect of humidity, although the higher level would not normally exist in a commercial dryer using heated air.

The corn dried in the continuous dryer was obtained from a carload of commercial corn from the 1956 crop, shipped at 21% moisture. This dryer was a vertical column 6 ft. high, 3 ft. wide, and 6 in. thick, constructed of metal screen. Grain was passed down the column as heated air was passed horizontally through the upper portion of the column and cold air passed through the lower portion. Air was passed through the grain at low, medium, and high air velocities (58–59, 118–127, and 138–181 c.f.m. per bu.), at approximately 150°, 190°, and 220°F. (65.6°, 87.8°, and 104.3°C.). Flow rate of grain down the column was adjusted to yield corn at 14 to 15.5% moisture as in commercial elevator practice.

Temperature of the grain was not measured in either of these experiments. Final grain temperature in the batch dryer was probably close to the air temperature.

Prime Starch Milling Procedure. This method was previously described in detail (9). The corn was steeped 48 hours at 49°C. in a dilute solution of lactic acid and adjusted to pH 4 with potassium hydroxide, containing potassium metabisulfite to give an initial concentration of 0.15% sulfur dioxide. The steeped corn was ground 1 minute in a Waring Blendor equipped with dull blades and operated at 85 volts. The mixture of starch and protein (prime starch fraction) was separated from the coarser fragments by screening and washing the milled grain on silk bolting cloth (openings 60 × 70μ) on a shaker. The filtrate was then passed over a slightly finer cloth (openings $44 \times 57\mu$) called the "cleanup screen," to remove fragments that escaped the first screen. Dry substance yields of cleanup screen residue and prime starch were calculated as a percentage of the dry weight of steeped grain. The prime starch fractions contained 6 to 8% protein and the cleanup residue about 18 to 25% protein.

When adequately steeped corn is ground by this method, germs are liberated with but slight fragmentation; endosperm is sufficiently reduced so that 60 to 70% of the starch is liberated. To express the milling results as a single number that is related to milling experience, an arbitrary millability score was devised:

$$\text{Millability score} = 2(\%\ \text{prime starch} - 25) - (\%\ \text{cleanup residue}) \times 20$$

This formula is based on the experience that prime starch yield seldom falls below 25% with very poorly steeped corn, whereas well-steeped corn may occasionally yield 75% prime starch. The cleanup residue yield is small (0.05 to 1.0%) but is a sensitive index of corn quality. Increase in this value indicates lower millability. Cleanup residue yield is arbitrarily multiplied by 20 and subtracted from the prime starch yield. Millability scores of 65 to 75% for steeped corn are considered essential for an acceptable wet-milling performance.

Viability. Viability was determined by sprouting the kernels for 5 days in a germination cabinet at 80°F. (26.7°C.). Kernels were soaked in 1% calcium hypochlorite solution 3 minutes before they were placed in the germinator.

Solubles. Total solubles, comprising native solubles and solubles formed on steeping with sulfur dioxide, were estimated as follows:

Whole corn (100 g.) was steeped at 49°C. for 48 hours in a medium initially containing 0.15% sulfur dioxide and 0.05% lactic acid. The steeped grain and its steepwater were ground in a Waring Blendor at full speed for 5 minutes. Grist was filtered on Whatman No. 12 fluted filter paper and the filtrate was analyzed for dry substance and nitrogen.

Results and Discussion

Batch-Dried Corn. Viability and solubles extraction data are given in Tables IA and IB. Our data confirm those of MacMasters and coworkers (2,5,6), that viability is the most sensitive index of grain damage resulting from improper drying conditions. Samples dried from the initial moisture level of 21% were rendered essentially nonviable when dried at 180° and 160°F. (82.2° and 71.1°C.). Grains dried from initial moisture levels of 32% were nonviable after drying at 140°F.

TABLE IA

VIABILITY AND SOLUBLES OF CORN DRIED FROM 32% INITIAL MOISTURE IN A BATCH DRYER

Relative Humidity	Drying Conditions			Viability	Solubles	
	Final Moisture	Air Temperature	Drying Time		Total Dry Substance	Protein (N × 6.25)
%	%	°F	*hours*	%	*% d b*	*% d b*
40	10.3	120	8.25	39	8.7	4.3
	10.1	140	4.0	0	8.3	4.2
	10.3	160	2.66	0	7.9	3.7
	10.1	180	2.5	0	7.2	2.9
	10.0	200	1.5	0	6.7	2.2
15	11.0	120	7.5	75	7.5	4.3
	11.0	140	4.0	1	8.2	3.9
	10.8	160	2.5	0	8.3	3.7
	10.8	180	2.5	0	7.7	3.4
	11.0	200	1.5	0	7.4	2.6

TABLE IB

VIABILITY AND SOLUBLES OF CORN DRIED FROM 21% INITIAL MOISTURE IN A BATCH DRYER

Relative Humidity	Drying Conditions			Viability	Solubles	
	Final Moisture	Air Temperature	Drying Time		Total Dry Substance	Protein (N × 6.25)
%	%	°F	*hours*	%	*% d b*	*% d b*
40	11.8	120	6.1	94	10.7	5.1
	11.5	140	3.5	70	8.9	3.7
	11.0	160	2.33	0	9.1	4.3
	8.0	180	1.5	0	8.0	3.9
15	8.6	120	7.25	95	9.3	4.3
	8.5	140	3.0	90	8.9	4.0
	8.8	160	2.0	4	9.0	3.7
	8.8	180	1.17	0	8.4	3.5

(60°C.) and higher. Therefore, corn harvested with picker-sheller equipment (22–30% moisture) (7) may lose viability when dried with heated air at lower temperatures than corn picked with a conventional corn picker and shelled at 16–22% moisture. Drying corn with air of high relative humidity was in general more damaging to viability than drying at lower humidity. This effect was noticeable with grain at both the 32 and 21% initial moisture levels.

Total dry substance and total protein extractable during steeping showed downward trends with increasing drying temperature. The loss in soluble protein was largely responsible for the lower dry substance values. The trend toward lower soluble protein values can be seen in all four groups of samples in Tables IA and IB. The greatest percentage reduction was found in corn dried at 40% r.h., indicating that relative humidity was of greater effect in this instance than initial moisture.

The decrease in soluble protein with increased drying temperatures suggests that heat-denaturation of endosperm protein is involved in damage to corn dried at high temperatures. We know that the action of sulfur dioxide in releasing starch from corn endosperm (8) in steeping is directly related to protein solubilization. It is possible that high temperature insolubilizes a specific protein fraction that normally releases starch granules when it dissolves in sulfur dioxide solutions (1). Determination of soluble protein is probably not a useful test for detecting heat-damaged corn, because soluble protein does not show a sharp break at any specific temperature. Furthermore, no absolute value could be assigned to undamaged corn, because of differences in initial soluble protein levels for different lots of sound corn. McGuire and Earle (4) reported similar conclusions.

Milling data for the batch-dried corn, given in Tables IIA and IIB, were treated in two ways for evaluation. The yields of prime starch and cleanup screen residue were arbitrarily combined, as already described,

TABLE IIA

PRIME STARCH MILLING RESULTS FOR CORN DRIED FROM 32% MOISTURE IN THE BATCH DRYER

RELATIVE HUMIDITY	DRYING TEMPERATURE	PRIME STARCH YIELD [a]	RESIDUE YIELD [a]	MILLABILITY SCORE
%	°F	%	%	%
40	120	67.0	0.63	71.4
	140	67.6	0.65	72.2
	160	65.0	0.77	64.6
	180	58.0	1.13	43.4
	200	41.2	1.11	10.2
15	120	65.8	0.59	70.4
	140	65.3	0.63	68.0
	160	65.3	0.69	66.8
	180	66.0	0.62	64.6
	200	46.3	1.19	18.8

Analysis of variance data

SOURCE	dF	MEAN SQUARE: Prime Starch	MEAN SQUARE: Residue
Temperature	4	376.25**[b]	0.2000*[b]
Relative humidity (r.h.)	1	16.56 NS[b]	0.0555 NS[b]
Temperature × r.h.	4	17.30**[c]	0.0421*[c]
Error	10	2.53	0.0097

[a] Each value is a mean of duplicate millings. At 5% significance level, LSD for prime starch yield = 6.8%; for residue yield, 0.41% for comparison of any two values in the table.
[b] Tested against sum of error and interaction mean squares.
[c] Tested against error variance.

TABLE IIB

PRIME STARCH MILLING RESULTS FOR CORN DRIED FROM 21% MOISTURE IN THE BATCH DRYER

Relative Humidity	Final Moisture	Drying Air Temperature	Prime Starch Yield [a]	Residue Yield [a]	Millability Score
%	%	°F	%	%	%
40	11.8	120	65.7	0.60	69.4
	11.5	140	65.0	0.69	66.2
	11.0	160	62.8	0.70	61.6
	8.0	180	57.7	1.11	43.2
15	8.6	120	64.6	0.75	64.2
	8.5	140	65.7	0.73	66.8
	8.8	160	64.2	0.72	64.0
	8.8	180	64.3	0.98	59.0

Analysis of variance data

Source	dF	Mean Squares: Prime Starch	Mean Squares: Residue
Temperature	3	15.544 NS[b]	0.1235*[b]
Relative humidity (r.h.)	1	14.440 NS[b]	0.0015 NS[b]
Temperature × r.h.	3	10.501*[c]	0.0143*[c]
Error	8	2.362	0.0027

[a] Each value is a mean of duplicate millings. At 5% significance level, LSD for prime starch yield = 6.6%, residue yield, 0.22%. for comparison of any two values.
[b] Tested against sum of error and interaction mean square.
[c] Tested against error variance.

to give an over-all millability score; the prime starch and cleanup screen residue yield values were each subjected to an analysis of variance. A summary of the statistical analysis is appended to each table. Drying the high-moisture corn (32%) with air of 180° or 200°F. and 40% r.h. significantly reduced prime starch yield and increased residue yield when compared with corn dried at the three lower temperatures. At 15% r.h., only 200°F. air had a significant effect.

A similar effect was observed in the drying of corn of 21% moisture content. Air of 40% r.h. at 180°F. significantly reduced prime starch yield and increased residue yield. At 15% r.h. only the residue yield, the sensitive index of damage, was significantly affected. The millability scores support these findings.

In the analysis of variance, the temperature × relative humidity interaction was statistically significant when tested against error mean square. When the mean squares of temperature and relative humidity were tested against the sum of error and interaction mean squares, only temperature was found to be significant at the 5% level. Therefore, there is no true interaction effect between temperature and relative humidity.

The millability scores, although not analyzed statistically, show rather clearly the tendency toward reduced milling quality for all grain samples dried at 180°F. (82.2°C.) and higher. Although yield data do not show significant effects at 160°F. (71.1°C.), the two 40% r.h. sets exhibit drops in millability score at this temperature. The initial moisture level of the grain did not seem to influence the millability results as it did viability. This is not surprising, since viability measures some property of the germ while millability measures endosperm properties.

Final moisture level sought was 11%, but five of the samples were accidentally dried to 8.0–8.8% moisture (Table IB). Both the 8 and 11% moisture levels are lower than usually desired for commercial drying, but final moisture did not appear to have a significant effect on grain properties studied. Drying time varied inversely with the air temperature. It was not possible to determine the effect of drying time independent of drying temperature, but it is possible that some of the effects ascribed to temperature were due to a time-temperature interaction. For example, high drying temperature may require little time to bring the grain to the desired average moisture level, but the gradient of moisture from exterior to interior of the kernel will be greater than in slow, low-temperature drying. Diffusion of moisture in high-temperature drying cannot keep up with evaporation, and the outer layers will be overdried and overheated with respect to the interior portion of the kernel. Starch and residue fractions were examined for starch granules that had been gelatinized by overheating. Both samples dried at 200°F. showed severe gelatinization. Presence of this type of damage in a wet-milling process quickly slows grind rate because screens and filters become plugged, and yields low-quality product starch.

Continuous Dryer. Milling and viability results obtained with grain dried in the continuous dryer are shown in Table III. Another variable has been added in this experiment: air-flow rate through the grain. Relative humidity was below 10%. Viability again proved to be the most sensitive index for detecting damage to the kernel caused by drying conditions. Air temperature in the range of 150°F. (65.6°C.) caused no damage at low air-flow rate, but medium and high air-flow rates each successively reduced viability by 10%. All corn was damaged with respect to viability at air temperatures in the range of 190° and 220°F. (87.8° and 104.3°C.). However, damage was least at the low air-flow level.

Data on analysis of variance again show that temperature was statistically significant in its effect on both prime starch and cleanup screen residue yields. The mean squares for air-flow rate were not significant

TABLE III

DRYING CONDITIONS, VIABILITY, AND MILLABILITY OF CORN DRIED FROM 21% INITIAL MOISTURE IN THE CONTINUOUS DRYER

DRYING TEMPERATURE [a]	AIR FLOW	TIME IN DRYER SECTION [b]	FINAL GRAIN MOISTURE	FINAL GRAIN VIABILITY [c]	PRIME STARCH YIELD [d]	RESIDUE YIELD [d]	MILLABILITY SCORE
°F	*c f m/bu*	*minute*	*%*	*%*	*% d b*	*% d b*	*%*
			High Air Flow Rate				
149	181	49	14.4	62	63.1	0.09	74.4
193	153	37	14.3	3	58.9	0.14	65.0
221	138	32	14.2	0	55.4	0.19	57.0
			Medium Air Flow Rate				
147	118	57	15.1	72	61.7	0.11	71.2
191	126	39	14.2	5	58.1	0.12	63.8
220	127	32	14.4	0	53.9	0.13	55.2
			Low Air Flow Rate				
150	58	79	15.1	82	62.8	0.08	74.0
188	59	49	14.6	39	59.6	0.10	67.2
218	59	42	15.5	29	57.4	0.13	62.2

Analysis of variance data

SOURCE	dF	MEAN SQUARES: Prime Starch	MEAN SQUARES: Residue
Temperature	2	72.162**[e]	0.00449*[e]
Air-flow rate (AFR)	2	6.310 NS[e]	0.00174NS[e]
Temperature × AFR	4	0.983 NS[f]	0.000893**[f]
Error	9	0.920	0.0000884

[a] Temperature of air entering grain column.
[b] Retention time in cooling section was one-third of time in drying section.
[c] Initial viability, 81%.
[d] Each value is a mean of duplicate millings. At 5% significance level LSD for prime starch yield = 5.9%, LSD for residue yield = 0.04% for comparison of any two values.
[e] Tested against error.
[f] Tested against sum of error and interaction mean squares.

when tested against the sum of error and interaction mean squares. As has been noted previously, residue yield is usually a more sensitive index of grain damage. In this series, residue yield values showed particularly good agreement between duplicate runs. At high air-flow rate, both prime starch and residue yield were significantly altered by drying at 193° and 221°F. (89.5° and 105°C.) as compared with 149°F. (65.1°C.). The sharp reductions in millability scores with each increase in temperature indicate that both the air temperature levels, 190° and 220°F. (87.8° and 104.3°C.), caused detectable damage to milling properties of this corn. No gelatinized starch granules were seen in the fractions from these corn samples, probably because of lower initial moisture content than in the batch series.

Flow rate of grain down the column, as measured by average retention time (Table III), was faster at high temperatures and high air-flow rates than at low temperatures and low air-flow rates, in order to deliver grain from the column at 14 to 15.5% moisture. The effect of average retention time of grain in the column probably has an important effect on final grain quality.

Conclusions

A laboratory method of measuring wet-milling properties of corn has shown that corn dried at 180°F. (82.2°C.) or higher is less suitable for wet-milling than corn dried at lower temperatures. Initial grain moisture, and flow rate of drying air in these experiments, did not influence milling results significantly. Very high relative humidity of the drying air tended to increase severity of damage at 180°F. (82.2°C.). Reduction in the amount of prime starch obtained after milling of steeped corn and an increase in the amount of cleanup screen residue were criteria of damage. Some effects may seem to be small; but, in a commercial wet-milling plant, equipment is usually operated at or near capacity and even small changes in volume of a particular fraction are quickly felt.

MacMasters *et al.* (6) have reported that corn samples grown in different seasons differ in response to conditions used in artificial drying. Considering the numerous factors involved in drying shelled corn and the many types of dryers in use, it is impossible to specify one air temperature that is critical in drying corn for starch manufacture. However, in general, our data and those of MacMasters *et al.* indicate that 160°F. (71.1°C.) should be maximum. Since kernel viability is evidently more easily altered by drying conditions than other properties examined, corn dried so as to preserve viability should invariably be suitable for starch manufacture.

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