WET-MILLING HIGH-AMYLOSE CORN CONTAINING 49- AND 57-PERCENT-AMYLOSE STARCH[1]

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ABSTRACT

High-amylose corn with starch containing 49 and 57% amylose can be wet-milled, with some modification in processing conditions, to produce a starch of slightly higher protein content, and in somewhat lesser yield, than is usually obtained from ordinary corn. A 64.4% yield of starch containing 0.51% protein was recovered from ordinary corn, as compared with a 43.5% yield of starch containing 0.7% protein from high-amylose corn (57%). An unusually large swelling of the corn kernels was observed during the steeping of high-amylose corn (57%). These kernels exhibited an increase of 128% of their original dry volume, as compared with a 63% increase in volume observed during the steeping of ordinary corn.

Interest in the potential industrial utilization of amylose and high-amylose starches has been increasing over the past few years. Considerable research is being carried out in this field by govermental, educational, and industrial laboratories. Cooperative work of the Northern Utilization Research and Development Division with a commercial corn breeder, and with corn breeders in Production Research, Agricultural Research Service, U. S. Department of Agriculture, stationed at the Agricultural Experiment Station of the University of Missouri, has resulted in the development of corn with starch containing from 50 to 80% amylose, as compared with the 24–27% present in ordinary corn starch (6,11). Generally, the amount of corn available with starch containing above 60% amylose has been limited to small quantities of breeding stock; considerable amounts of field-type corn in the 50–60% range have been grown.

During the wet-milling of a number of small samples of high-amylose corn to recover starch for evaluation studies, processing difficulties were encountered in the separation of starch and gluten. The smaller size and more irregular shape of the high-amylose starch granules made them more difficult to separate from the gluten, resulting in the recovery of starch with a high protein content (2).

This paper discusses the wet-milling characteristics of corn samples with starch containing about 49 and 57% amylose and compares them with those of an ordinary dent corn.

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Materials and Methods

Materials. Samples of high-amylose dent-type corn, from both the 1957 and 1958 crops, were used in these tests. A sample of ordinary dent-type corn grown in 1956 was used for comparison.

Processing Methods. All experiments were first carried out on a laboratory scale and then confirmed in the pilot plant.

In the laboratory experiments, 1,500 g. of corn were steeped in 2,800 ml. of distilled water containing 0.25% sulfur dioxide at 125°F. After 48 hours of steeping, the water was drained, the grain was coarsely ground in a Quaker City[2] drug mill, and the germ was recovered by simple flotation. The fibrous material remaining was finely ground in the drug mill. The resulting slurry was screened first on a 0.039-in. perforated copper sieve to remove coarse fibers and then on a 200-mesh stainless-steel sieve to remove fine fibers. The final slurry, the millstarch, was tabled to separate the starch from the gluten. This procedure has been described by Zipf *et al.* (10).

In the pilot-plant experiments, 2 bu. of grain were steeped in tapwater containing sulfur dioxide. After steeping was completed, the water was drained, and the corn was ground in a Bauer mill equipped with jaw-toothed plates to crack the kernel and free the germ. The germ was recovered by flotation, and the remaining material was ground in a Rietz disintegrator equipped with a 1/16-in. round, perforated screen. Coarse fibers were removed from the slurry by passing it over a Rotex gyratory shaker equipped with a 26-mesh screen; fine fibers were recovered by passing it over the same shaker equipped with a 200-mesh screen. The resulting millstarch was separated into starch and gluten by tabling. The pilot-plant equipment and procedures have been described by Anderson (3).

Analytical Methods. The moisture of the corn and of the various recovered fractions was determined by drying a sample for 4 hours at 110°C., under a vacuum of 28 in. of mercury. Protein ($N \times 6.25$) was determined by an improved Kjeldahl method for nitrate-free samples (1). Starch content was determined polarimetrically by the procedure of Earle and Milner (7). Apparent amylose content of the corn and of the separated starch was determined by the iodine sorption method of Bates, French, and Rundle (5), as modified by Wilson, Schoch, and Hudson (8). The reported amylose figures in the high-amylose corn are generally high, as it has been shown that the amount of material which can be isolated as amylose from this corn is significantly lower (9). Fat analysis was carried out according to the official

[2] Mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

method of analysis of the AOAC (4). Sulfur dioxide in the steep-water was determined by titration with iodine solution using starch as the indicator. Solubles and sugar analyses were conducted according to the laboratory methods of AACC (1). All recovery and analytical data presented are on a moisture-free basis (M.F.B.).

Results and Discussion

Establishment of Optimum Conditions and Procedure. The described general processing procedure for wet-milling cereal grains has been established over the years by the performance of many experiments on corn, wheat, and grain sorghum. After several samples of high-amylose corn had been milled it was found that some modification had to be made. In particular, a change in the starch-gluten separation was needed, since a good recovery of high-purity starch could not be obtained.

In the investigation of the starch-gluten separation, variables studied were the pitch of the starch table, specific gravity of the millstarch, and the effect of double tabling (Table I). The effect of increasing the table pitch, while the other variables remained constant, was to improve the purity of the starch. When the millstarch was tabled at a pitch of ½ in. per 20 ft., the protein content of the resulting starch was 4.3%. There was substantial improvement in the purity of the starch, with little sacrifice in recovery, when the millstarch was tabled at a table pitch of 1 in. per 20 ft. Raising the table to 2 in. per

TABLE I
EFFECT OF CERTAIN VARIABLES ON THE STARCH-GLUTEN SEPARATION OF HIGH-AMYLOSE CORN (49%)

Variable	Pitch of Table			Specific Gravity of Millstarch		Double Tabling of Starch	
Specific gravity of millstarch, ° Baumé at 60°F.	3.5	3.5	3.5	3.5	6.0	3.5	3.5
Pitch of table, in./20 ft.	½	1	2	2	2	1	2
Recovery of starch,[a] % M.F.B.[b]	88.2	85.1	83.6	83.6	83.2	...	80.0
Purity of recovered starch, % protein, M.F.B.	4.3	1.33	1.05	1.05	1.12	...	0.53
Purity of recovered gluten, % protein, M.F.B.	33.4	37.5	33.1	33.1	33.5	32.2	...

[a] Recovery of starch based on amount of starch present in corn.
[b] Moisture-free basis.

20 ft. resulted in still more improvement in the starch purity, while maintaining a good starch recovery. As the pitch of the table is increased, the smaller starch granules go off the table with the gluten and the protein content of the gluten remains about the same.

Variation of the specific gravity of the millstarch between 3.5° and 6.0° Baumé at 60°F. caused only minor differences in the starch purity and recovery. However, at 3.5° Baumé a slightly better starch was recovered, and this specific gravity was selected as optimum for the remaining work.

Starch of good purity was recovered by tabling the millstarch twice. The first tabling was carried out at a table pitch of 1 in. per 20 ft. Gluten containing 32.2% protein was recovered from this table. The starch was then reslurried and retabled at a pitch of 2 in. per 20 ft., yielding starch with a protein content of 0.53%. Starch recovery by double tabling was about 5% less than obtained by single tabling. The overflow from the second tabling, the squeegee starch, had a protein content of about 17%. Double tabling results in obtaining a good grade of starch with fair recoveries from high-amylose corn containing starch having 49% amylose.

Comparison of Processing Characteristics of High-Amylose and Ordinary Dent-Type Corn. Processing characteristics of two samples

TABLE II

COMPOSITION OF CORN USED IN WET-MILLING EXPERIMENTS
(Moisture-free basis)

COMPOSITION	ORDINARY CORN	HIGH-AMYLOSE CORN (49%)	HIGH-AMYLOSE CORN (57%)
Crop year	1956	1957	1958
Apparent amylose content of starch, %	24	49	57
Protein, %	10.1	12.6	13.9
Fat, %	4.5	6.7	7.1
Starch, %	73.8	62.5	60.9
Solubles, %	5.6	7.3	8.6
Total sugars, %	2.3	3.2	2.6

of high-amylose corn and a sample of ordinary corn were compared on a laboratory scale. The composition of the different corn samples is given in Table II. The protein, oil, and solubles content of the corn increased and the starch content decreased as the amount of amylose present in the starch became greater.

Steeping characteristics of the samples showed some differences when the steeping of the corn was followed for the complete steeping time. This was accomplished by analyzing, at intervals, the steeped

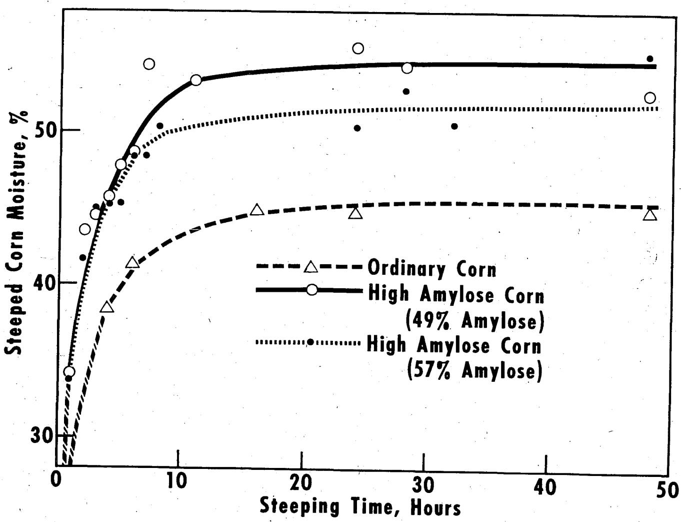

Fig. 1. Effect of steeping time on the steeped corn moisture.

corn for moisture and also the steepwater for pH and sulfur dioxide content. The steeped corn moisture curve from high-amylose corn followed the characteristic water absorption curves obtained from the steeping of ordinary corn, as shown in Fig. 1. However, the high-amylose corn absorbed from 6 to 8% more water in about the same amount of time as did the ordinary corn. As illustrated in Fig. 2, the pH of the steepwater obtained from the three corn samples followed similar curves, except that the pH of the steepwater from ordinary

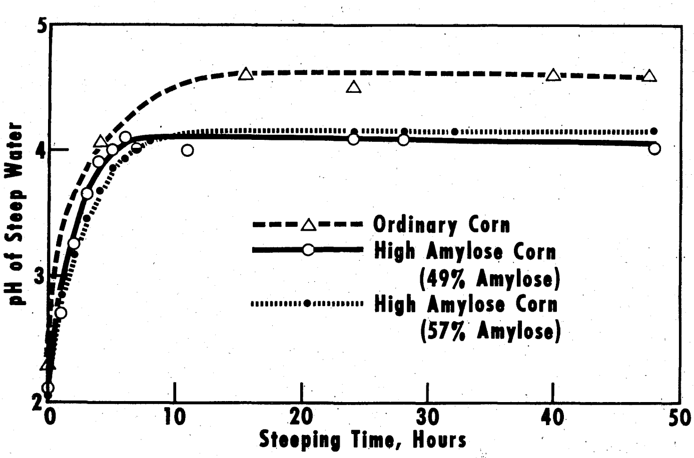

Fig. 2. Effect of steeping time on the pH of the steepwater.

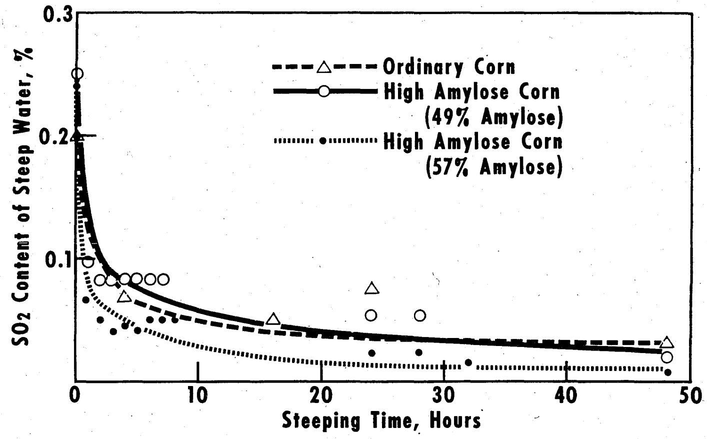

Fig. 3. Effect of steeping time on the sulfur dioxide content of the steepwater.

corn was somewhat higher than from high-amylose corn. The disappearance of sulfur dioxide from the steepwater was about the same for the three samples (Fig. 3). The yield of solids and their protein content in the steepwater were about the same for all three samples, averaging 4.3 and 30.8%, respectively. This comparison and the comparison of other processing characteristics of the three different corn samples are given in Table III. The amount of reducing sugars in the steepwater varied from 0.76% from ordinary corn steepwater to about 2% in the steepwater from high-amylose corn.

One significant difference was the amount of swelling of the high-amylose corn kernel as compared with ordinary corn. During steeping, high-amylose corn (57%) exhibited an increase of 128% of its original dry volume, whereas high-amylose corn (49%) had an increase of 100%, and ordinary corn only 63%. This degree of swelling would decrease the amount of high-amylose corn that could be steeped in tanks of a given size, which might force a reduction in the daily grind in a commercial plant.

Germ yield from the processing of both samples of high-amylose corn amounted to 6.5%, whereas germ yield from ordinary corn was 4.7%. This increase is undoubtedly due to the larger germ present in high-amylose corn. Oil content of the germ was about the same for the three samples; however, recovery of total oil from the high-amylose corn germ was slightly less than from ordinary corn — 51% as compared with 55.5%.

There were no significant differences in the manner in which the coarse or fine fibers from either high-amylose or ordinary corn responded during processing. The amount of fibers recovered was about the same, and the protein and starch contents of the fiber fractions were similar.

The most significant differences were observed during the starch-gluten separation. The yield of starch from a single tabling of mill-starch from ordinary corn was 64.4% (87.3% on a starch basis), and the starch contained 0.51% protein. This is an optimum result for the

TABLE III

COMPARISON OF THE PROCESSING OF THREE DIFFERENT SAMPLES OF CORN

	Ordinary Corn	High-Amylose Corn (49%)	High-Amylose Corn (57%)
Steepwater			
Yield[a] of solids, % M.F.B.[b]	4.2	5.1	3.6
Protein content, % M.F.B.	31.2	30.4	31.0
Reducing sugars, % M.F.B.	0.76	1.9	2.0
Steeped corn			
Average final moisture, %	46	54	52
Volume increase, %	63	100	128
Germ			
Yield, % M.F.B.	4.7	6.5	6.5
Crude oil content, % M.F.B.	53.0	52.9	56.0
Recovery of total oil, %	55.5	51.4	51.0
Coarse fiber			
Yield, % M.F.B.	12.8	11.1	12.6
Protein content, % M.F.B.	14.9	12.7	10.4
Starch content, % M.F.B.	19.8	12.4	12.4
Fine fiber			
Yield, % M.F.B.	6.2	6.4	4.9
Protein content, % M.F.B.	23.8	22.2	21.1
Starch content, % M.F.B.	30.6	31.8	30.1
Starch			
Yield, % M.F.B.	64.4	50.0	43.5
Recovery,[c] %	87.3	80.0	71.4
Protein content, % M.F.B.	0.51	0.53	0.7
Gluten			
Yield, % M.F.B.	7.8	5.3	15.5
Protein content, % M.F.B.	44.2	30.0	33.1
Starch content, % M.F.B.	....	52.7	52.3
Squeegee starch[d]			
Yield, % M.F.B.	....	3.5	4.9
Protein content, % M.F.B.	....	18.9	5.0
Starch content, % M.F.B.	....	71.6	94.4
Process waters			
Yield of solids, % M.F.B.	2.5	6.1	6.9
Protein content, % M.F.B.	40.0	37.0	44.5

[a] Yield based on original weight of dry corn.
[b] Moisture-free basis.
[c] Recovery of starch based on starch present in corn.
[d] Squeegee starch is result of second tabling.

general batch procedure followed in these studies. It would be expected that better results would be obtained in a commercial operation using countercurrent steeping and centrifuges or hydroclones for the starch-gluten separation.

In contrast, double tabling of millstarch from high-amylose corn, both the 49 and 57% variety, resulted in lower yields, 50 and 43.5%, respectively. This loss is partially due to the smaller amount of starch originally present in high-amylose corn, but is probably caused primarily by the presence of small and irregularly shaped starch granules. On the basis of experience with ordinary corn, it seems improbable that the higher protein content of high-amylose corn is an appreciable factor affecting the starch-gluten separation. The loss is further emphasized by the amount of high-amylose starch recovered, based on the starch in the corn. The starch recovery from 49%-amylose corn was 80.0%, and from the 57% material only 71.4% was recovered. The 0.53% protein content of the starch recovered from the 49%-amylose corn compares favorably with starch from ordinary corn. However, the starch obtained from 57%-amylose corn contained slightly more protein (0.7%); this, coupled with its lower yield, illustrates the difficulty involved in separating starch and gluten from corn with an increased amylose content.

The yield and protein content of the gluten recovered from the ordinary corn were 7.8 and 44.2%, respectively. This is of the magnitude usually obtained for a commercial tabling operation. The yield of glutens from high-amylose corn varied from 5.3 to 15.5%. They contained only 30 to 35% protein and over 50% starch. The "squeegee" starch, which was overflow or "gluten" from the second tabling of high-amylose starch, had very high starch contents, as expected.

The solids present in the process waters from wet-milling ordinary corn amounted to 2.5% of the corn processed and contained 40% protein. The process waters from high-amylose corn, 49 and 57%, contained 6.1 and 6.9% solids, respectively, and their protein contents also averaged about 40%. The recovery of a greater quantity of solids in process waters from the two high-amylose corns can be attributed to their higher soluble content and also to the presence of very small starch granules which did not settle out in the various settling operations.

Two-bushel lots of each of the three corn samples were processed in the pilot plant to confirm the data and results obtained in the laboratory studies. The observations made during the larger-scale runs were quite similar to those made in the laboratory. While starch recovered from ordinary corn had a protein content of 0.47%, starch from both the 49- and 57%-amylose corn contained about 0.6% protein.

Similar comparative investigations will be made as plant breeders provide samples of corn that contains starch with even higher percentages of amylose than corn used in this study.

Acknowledgment

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