

Wet Milling of Maize Grits

S. R. ECKHOFF,¹ W. V. JAYASENA,² and C. K. SPILLMAN³

ABSTRACT

Cereal Chem. 70(3):257-259

Maize grits obtained from the standard dry milling of whole corn kernels were studied as the raw material for wet milling. One size of corn grits (those that pass through a no. 5 and over a no. 10 screen), two levels of SO₂ concentration (0.1 and 0.2%), and four levels of steeping time (4, 6, 8, and 10 hr) were used. The steep time could be reduced to 6 hr, and the SO₂ concentration could be reduced to 0.1% in the wet milling of corn grits without any significant reduction in starch yield or increase in protein content of starch. Starch recovery of up to 88.9% (db) was

achieved from the grits in the study, compared to a 87.4% (db) recovery from the control samples (48 hr, 0.2% SO₂, whole kernels). The protein content of starch was as low as 0.5% for the 10-hr steep. The highest and lowest values recorded for apparent viscosity of starch among treatments were 462 and 377 Brabender units, respectively. Both of these values were significantly lower than that of the apparent viscosity of starch obtained from wet milling of whole kernels, which was 490 Brabender units.

During steeping, corn kernels are soaked in dilute sulfurous acid at 48–53°C for 24–48 hr to get optimum separation of components (Kerr 1950). A number of studies have attempted to find a steeping method that would decrease steeping time by increasing the rate of water and SO₂ penetration into the corn kernel (Cox et al 1944, Wagoner 1948, Watson et al 1951, Chu 1961, Watson and Sanders 1961, Fan et al 1965, Wahl and Franzke 1970, Krochta et al 1981, Roushdi et al 1981, and Hassanean and Wahed 1986).

One way to increase the diffusion rate of water is to steep grits instead of whole corn kernels. This allows sulfurous acid to diffuse more rapidly and completely into the protein matrix, reducing the time and cost of steeping. Powell and McGeorge (1975) applied for a patent on a combined wet- and dry-milling process that steeps endosperm fractions. They suggested that steep time could be reduced to as little as 0.5 hr for fine flour. The patent did not discuss the specific procedure used to perform the steeping nor evaluate the resultant starch quality.

Eckhoff and Tso (1991) steeped grits in sulfurous acid for 10 hr as a means of evaluating the effect of high-temperature drying on endogenous protease activity. They were able to increase starch recovery rates with the addition of protease. However, they looked at only one SO₂ level and one steep time and did not look at optimum parameters for steeping grits.

The objectives of this study were to determine the appropriate SO₂ level and steeping time for wet milling grits and to determine the quality of the resulting starch as measured by Brabender Amylograph and by protein level.

MATERIALS AND METHODS

Sample Preparation

Corn (DeKalb 636 hybrid) was cleaned using a grain cleaner (model 6F6, Kice Metal Products, Wichita, KS). Using procedures similar to Eckhoff and Tso (1991), the moisture content was adjusted to approximately 21% in three steps: 12–15% for 16 hr; 15–18% for 1 hr; 18–21% for 30 min. Approximately 150 kg of the cleaned corn was degerminated using a Beall no. 0 degerminator (Union Iron Works, Decatur, IL). After degermination, the pericarp was aspirated off, and germs were recovered using an Eriez H1-V1 model F gravity table (Eriez Magnetics, Erie, PA). The remaining endosperm pieces were dried in a continuous flow dryer (model C1-12-6-RS, Aeroglide Corp., Raleigh, NC). Grits that passed through a no. 5 screen and were retained on a no. 10 screen of a dry granular separator (model

2-18-24, Forsbergs, Inc., Thief River Falls, MN) were used in the steeping tests.

Sixteen representative 1,000-g grit samples were prepared using a Bournier divider (Seedburo Equipment Co., Chicago, IL). The moisture content (11.2%, db) was determined in triplicate (40 g) by the standard air-oven method (103°C, 72 hr). Standard laboratory analytical procedures (AACC 1983) were followed to determine crude protein (8.02%), fiber (0.28%), and oil (1.36%) content in duplicate. A Yellow Springs Instrument, model 27 (Yellow Springs, OH), was used to determine starch content according to the method described by Budke (1984).

Wet-Milling Procedure

A solution of concentrated sulfur dioxide was prepared by bubbling SO₂ gas through distilled water. The SO₂ content of the solution was determined using an idometric-titration procedure similar to that described by Eckhoff and Okos (1983). The solution was diluted with distilled water to a volume of 2,000 ml to get the desired concentration of SO₂ in the steep water.

The laboratory wet-milling procedure of Anderson (1963) was used with some modification as reported by Jayasena (1988). Each sample was steeped in a batch-type steeping tank with continuous steep water recirculation through a Precision model 291 (Precision Scientific, Chicago, IL) temperature-controlled water bath set to maintain a constant temperature of 50°C. A Straub model 4E grinding mill (Quaker City Mill, Seedburo Equipment) was used to finely grind the steep corn grits. The steep water was drained after steeping, as in a usual batch-steeping process of whole corn kernels, and was mixed with the starch-gluten slurry before tabling.

Fiber separation was performed by hand working the fiber over a 200-mesh stainless steel screen for approximately 4 hr. Specific gravity of the remaining starch slurry was adjusted to 1.04 (Kerr 1950) by adding a small amount (~100 g) of starch wet-milled in a practice run from the same hybrid of corn. The starch separation was done on a 6.4-m table, constructed according to the specifications reported by Anderson (1963), at a slurry flow rate of 300 ml/min. A Whatman no. 4 filter with a vacuum pump was used to recover gluten from the table overflow water. A fixed amount of distilled water was used in the study at each step: 2 L of water for grinding, 5 L for fiber washing, and 1 L for starch washing.

The starch, gluten, and fiber were dried in a forced-air convection oven for 24 hr at 50°C. Moisture content of each product was determined by drying a small sample of the dried product at 120°C for 2.5 hr (Anderson 1963). The apparent viscosity of the resulting starch was measured in Brabender Units (BU) using a starch solution of 7% dry solids prepared by mixing 32.2 g of starch (db) with 460 ml of buffer solution (AACC method 22-10). The starch was heated from 35 to 95°C at a temperature increase of 1.5°C/min in Brabender Viskograph-E (C. W. Brabender, South Hackensack, NJ), maintained at 95°C for 30 min, and then cooled to 50°C. The maximum viscosity and the temperature at the maximum viscosity were recorded.

¹Associate professor, Department of Agricultural Engineering, University of Illinois, Urbana-Champaign.

²Graduate student, Department of Grain Science and Industry, Kansas State University, Manhattan.

³Professor, Department of Agricultural Engineering, Kansas State University, Manhattan.

Statistical Analysis

The experimental design was a duplicated full-factorial design using four levels of steeping time (4, 6, 8, and 10 hr) and two levels of SO₂ concentration (0.1 and 0.2%). Analysis of variance and Duncan's multiple range tests were used for data analysis (SAS 1989).

RESULTS AND DISCUSSION

Starch Recovery

Steeping of grits was accomplished in as short as 4 hr, with starch yields comparable to the 48 hr steep (Table I). Starch yield increased (76.8–77.9% and 76.9–77.9%) with increasing steeping from 4 to 10 hr. There was no statistical difference in starch yield between samples steeped with 1,000 ppm or 2,000 ppm.

The highest numerical starch yield (77.9%, db) from steeping grits was obtained at 10 hr, although there was no significant difference in starch yield between 8 and 10 hr. This indicates that a steep time of 8 hr and a 0.1% SO₂ concentration could be used without any significant decrease in starch yield. The lowest starch yield (76.8%, db) was recorded at a steep time of 4 hr and a 0.1% SO₂ concentration. The starch yield increased with increasing steeping time for both the 0.1 and 0.2% SO₂ concentrations.

Starch recovery levels (Table I) were lower (87–89%) than those obtained in industry (92–94%). However, the average starch recovery reported by Anderson (1963) using a laboratory wet milling of whole kernels was 87.9%. The starch recovery from corn grits was higher than the recovery for the 48-hr steep of

whole kernels at all steeping times except the 4-hr treatment. Sulfur dioxide is in direct contact with endosperm proteins during steeping of corn grits. This increases the rate of protein disintegration, even at low SO₂ concentrations, resulting in higher starch recovery.

Gluten Recovery

Higher gluten yield was recorded at the shorter steeping times (Table I), indicating a loss of starch into the gluten fraction. The gluten yield of all treatments (grit samples) was higher than that of the control because of germ and pericarp removal from the grit samples.

Fiber and Soluble Recovery

The lowest fiber content was observed at 8 and 10 hr of steeping for both 0.1 and 0.2% SO₂ concentrations (Table I), indicating lower residual starch in the fine fiber. The solids content of process water (filtrate solids) did not show any significant difference in any of the treatments, although the 48-hr steep also had an additional 3% dry solids lost with the steep water (not shown in Table I). In grit steeping, the water used for steeping was also used as process water.

Starch Quality

The lowest starch protein content (0.49%, db) was for starch from the 10-hr steep with 0.2% SO₂ concentration (Table II). The highest protein content of 0.73 and 0.70% (db) were observed in the treatments with 4-hr steeping. The protein content of starch from wet milling of corn grits at 6, 8, and 10 hr of steeping were statistically similar to that of starch from whole kernel wet milling (Table II). In all cases, the protein content of the starch was greater than industry standards where levels of 0.35–0.50% are anticipated. Higher levels of protein in the starch are expected when tabling the starch on a 6.4-m table and hand separating the fiber. Industry uses countercurrent washing of the starch to remove the additional protein. Results from this study are comparable to results presented by Anderson (1963).

The steeping time and SO₂ concentration could be reduced to 6 hr and 0.1%, respectively, without any significant difference in starch protein content. A marked decrease in protein content, about 23%, could be achieved when steep time increased from 4 to 6 hr (Table II). No significant decrease in protein content was observed when steep time increased from 6 to 10 hr. The protein content of starch was not significantly different for the 0.1 and 0.2% SO₂ concentrations at any of the steep times tested.

The highest apparent viscosity of 490 BU was recorded for the starch obtained from the control (wet-milled whole corn kernels) sample (Table III). The apparent viscosity of starch from the 0.1% SO₂ concentration and 4-hr steeping treatment was 462 BU, which was the highest value from any starch produced from

TABLE I
Starch Yield and Recovery from Wet Milling of Corn Grits at Different Steep Times and SO₂ Concentrations

Steeping Time (hr)	SO ₂ (%)	Starch Yield (% db) ^a	Starch Recovery (% db)	Gluten Yield (% db)	Fiber Yield (% db)	Filtrate Solid Yield (% db)
4	0.1	76.8 b	87.3 d	11.1 a	6.7 a	2.7 a
6	0.1	77.3 ab	88.1 c	10.7 ab	6.6 b	2.6 a
8	0.1	77.6 a	88.5 ab	10.4 b	6.3 d	2.6 a
10	0.1	77.9 a	88.8 a	10.4 b	6.3 d	2.6 a
4	0.2	76.9 b	87.5 d	11.1 a	6.6 a	2.7 a
6	0.2	77.4 ab	88.2 bc	10.8 ab	6.4 c	2.6 a
8	0.2	77.8 a	88.8 a	10.3 b	6.3 d	2.6 a
10	0.2	77.9 a	88.9 a	10.3 b	6.3 d	2.6 a
Control ^b	0.2	65.1 c	87.4 d	7.7 c		
LSD ^c ($\alpha = 0.05$)		0.67	0.29	0.57	0.20	0.04

^aValues with same letters between rows are not significantly different based upon Duncan's multiple range test with $\alpha = 0.05$.

^bWet milling of whole corn kernels using a 48-hr steep.

^cLeast significant difference based upon analysis of variance test.

TABLE II
Protein Content of Starch Obtained from Wet Milling of Corn Grits

Steeping Time (hr)	SO ₂ Concentration (%)	Protein Content of Starch (% db) ^a
4	0.1	0.73 a
6	0.1	0.54 b
8	0.1	0.51 b
10	0.1	0.50 b
4	0.2	0.70 a
6	0.2	0.55 b
8	0.2	0.49 b
10	0.2	0.57 b
Control ^b	0.2	0.57 b
LSD ^c ($\alpha = 0.05$)		0.09

^aValues with same letters between rows are not significantly different based upon Duncan's multiple range test with $\alpha = 0.05$.

^bWet-milled whole corn kernels with 48-hr steep.

^cLeast significant difference based upon analysis of variance test.

TABLE III
Apparent Viscosity of Starch Obtained from Wet Milling of Corn Grits

Steeping Time (hr)	SO ₂ Concentration (%)	Apparent Viscosity (BU) ^{a,b}	Temperature at Max. Viscosity (°C) ^b
4	0.1	462 b	92.3 b
6	0.1	428 c	92.7 ab
8	0.1	420 c	92.2 b
10	0.1	410 cd	92.6 b
4	0.2	393 de	92.3 b
6	0.2	388 c	92.8 ab
8	0.2	384 c	92.2 a
10	0.2	377 c	92.7 ab
Control ^c	0.2	490 a	93.3 a
LSD ^d ($\alpha = 0.05$)	...	18.9	0.58

^aBrabender units.

^bValues with same letters between rows are not significantly different based on Duncan's multiple range test with $\alpha = 0.05$.

^cWet-milled whole corn kernels with 48-hr steep.

^dLeast significant difference based upon analysis of variance test.

grits. The apparent viscosity decreased to 377 BU for 10-hr steeping (Table III). Starch consistency decreases with increasing steeping time and SO₂ level. The temperature of the starch at maximum viscosity showed no significant differences for test conditions.

The low apparent viscosity for starch in grit steeping as compared to whole kernel steeping is not surprising because the grits are more readily exposed to the lower pH of the steep water. Also, the buffering capacity of the grits is more readily overwhelmed by the increased diffusional flux due to the smaller particle size. The increased exposure to SO₂ would lower pH in the grits and could result in acid thinning of the starch and a concomitant decrease in Brabender peak values. It is also possible that, during dry milling, some of the starch granules were damaged by the mechanical forces in the degerminator.

CONCLUSIONS

The steep time could be reduced to 6 hr and the SO₂ concentration could be reduced to 0.1% in wet milling of corn grits without any significant reduction in starch yield or any increase in protein content of starch. Starch recovery as high as 88.9% (db) could be achieved in wet milling of corn at a 10-hr steep as compared to 87.4% for wet milling of whole corn kernels. The protein content of starch in this study compared well with that of starch obtained from whole corn kernels. The SO₂ concentrations of 0.1 or 0.2% did not have any significant effect on either average starch yield or protein content of starch. Because of the increased exposure of the endosperm to SO₂, resulting in acid thinning of the starch, the apparent viscosity of starch obtained from the corn grits was significantly lower than that of starch from the wet milling of whole corn kernels.

LITERATURE CITED

AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1983. Approved Methods of the AACC. Method 22-10, approved May 1960, revised October 1982; Method 30-20, approved April 1961, revised October 1975, reviewed October 1982; Method 32-10, approved October 1981,

revised October 1982; Method 46-13, approved October 1976, reviewed October 1982, revised October 1986. The Association: St. Paul, MN.

ANDERSON, R. A. 1963. Wet-milling properties of grains: Bench-scale study. *Cereal Sci. Today* 8:191.

BUDKE, C. S. 1984. Determination of total available glucose in corn base material. *J. Agric. Food Chem.* 32:34.

CHU, P. S. 1961. Diffusion of water in kernels of corn and sorghum. M.S. thesis. Kansas State University, Manhattan, KS.

COX, M. J., MACMASTERS, M. M., and HILBERT, G. E. 1944. Effect of the sulfuric acid steep in corn wet milling. *Cereal Chem.* 21:447.

ECKHOFF, S. R., and OKOS, M. R. 1983. A direct titrimetric method for the rapid estimation of water extractable sulfur dioxide in corn. *J. Agric. Food Chem.* 31:826.

ECKHOFF, S. R., and TSO, C. C. 1991. Starch recovery from steeped corn grits as affected by drying temperature and added commercial protease. *Cereal Chem.* 68:319.

FAN, L.-T., CHEN, H.-C. SHELLENBERGER, J. A., and CHUNG, D. S. 1965. Comparison of the rates of absorption of water by corn kernels with and without dissolved sulfur dioxide. *Cereal Chem.* 42:385.

HASSANEAN, A., and WAHED, A. A. 1986. A new method to shorten the steeping period of corn grains. *Starch/Staerke* 38:417.

JAYASENA, W. V. 1988. Optimum steeping parameters of corn grits. M.S. thesis. Kansas State University, Manhattan, KS.

KERR, W. R. 1950. *Chemistry and Industry of Starch*, 2nd ed. Academic Press: New York.

KROCHTA, J. M., LOOK, K. T., and WONG, L. G. 1981. Modification of corn wet milling steeping conditions to reduce energy consumption. *J. Food Process. Preserv.* 5:39.

POWELL, L. P., and MCGEORGE, G. G. 1975. Process for recovery of starch and corn oil from corn. U.S. patent 3,909,288.

ROUSHDI, M., GHAL, Y., and CAIRO, A. H. 1981. Factors improving the steeping process of corn grains. *Starch/Staerke* 33:49.

SAS INSTITUTE. 1989. *SAS User's Guide*. The Institute: Cary, NC.

WAGONER, J. A. 1948. Microscopical observations and procedures used in a study of industrial corn steeping. *Cereal Chem.* 25:354.

WAHL, G., and FRANZKE, C. 1970. Biochemical-technological studies on the wet-processing of corn. Part 2: The changes in the composition of the steeping water during the steeping process. *Starch/Staerke* 22:64.

WATSON, S. A., WILLIAMS, C. B., and WAKELY, R. D. 1951. Laboratory steeping procedures used in a wet milling research program. *Cereal Chem.* 28:105.

WATSON, S. A., and SANDERS, E. H. 1961. Steeping studies with corn endosperm sections. *Cereal Chem.* 38:22.

[Received June 23, 1992. Accepted December 2, 1992.]