Starch Recovery from Steeped Corn Grits as Affected by Drying Temperature and Added Commercial Protease

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Millions of tons of starch are produced annually in the United States by corn wet milling for subsequent processing into syrup, ethanol, chemicals, and other products. The wet-milling process steeps whole kernels countercurrently in a 0.1-0.2% sulfurous acid solution at 50-55° C for 24-38 hr to soften the kernels, inhibit the growth of microorganisms, remove solubles, and break down the disulfide bonds in the protein matrix to facilitate the release of starch. Steeping is a capital-intensive, time-consuming step in the process because of the resistance of the seed coat and aleurone layer to water and sulfur dioxide absorption (Cox et al 1944). Watson and Sanders (1961) showed that starch release could be achieved within 2-6 hr of steeping if diffusion limitations were removed. Jayasena (1988) found that starch recovery and protein in starch were comparable in large corn grits steeped with 0.1% sulfurous acid for 6 hr and in whole kernels steeped with 0.2% sulfurous acid for 48 hr.

One of the most important factors influencing wet-milling qualities of corn is the drying temperature. High-temperature drying lowers the yield and quality of starch (Brown et al 1981). Le Bras (1982) showed that starch yield decreases significantly as the drying temperature increases beyond 90°C. Cox et al (1944) and Watson and Sanders (1961) reported similar results. Generally, temperatures above the gelatinization temperature of starch (approximately 57°C) have a negative effect on starch yield and/or starch quality.

Proteolytic enzymes (proteases) are capable of hydrolyzing peptide bonds of proteins and polypeptides. Proteolytic activity during steeping is generally due to the activation of endogenous enzymes by sulfur dioxide, and these proteases can affect the release of corn starch in the first phase of steeping (Wahl 1971). Spanheimer et al (1972) studied the effect of protease mixtures on corn grits (30% moisture content) as a pretreatment for air classification. They reported that all protease treatments solubilized a portion of the protein matrix and weakened grit structure. Kerpisci (1988) found that the specific activity of endogenous corn protease decreased linearly as drying temperature increased and correlated to a decrease in starch yield.

The objective of this study was to apply commercial protease to corn dried at high and low temperatures (HTD corn and LTD corn, respectively) to investigate the effects on wet-milling properties and on the yield and quality of starch.

MATERIALS AND METHODS

Yellow dent corn (FR27 × FRMo17), a relatively soft endosperm hybrid, was dried at 30, 80, and 120°C from an initial moisture content of approximately 30% to 14.5-15% (wet basis). Samples of corn dried at low (30°C) and high (a 50-50% mixture of the corn dried at 80 and 120°C) temperatures were dry milled to make grits. The corn dried at 80 and 120°C was mixed to provide a sufficient quantity of corn for the test. The corn was tempered by adding distilled water to 2,500 g of corn in a sealed plastic

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bag. Moisture was increased to 21%, held for 1 hr 45 min, and then increased to 24% and held for 15 min before degermination. The bag was shaken frequently to ensure uniform moisture absorption by the corn kernels.

A horizontal rotor degerminator was operated at 1,750 rpm. Details of the degerminator were reported by Brekke et al (1972). The overs of a No. 3 1/2 (Tyler number) sieve was run through the degerminator again. After ambient drying for 24 hr, the through-stock was aspirated twice with an aspirator (model 6DT4, Kice Metal Products Co. Inc., Wichita, KS) to remove fiber. Flour and fines were separated from the fiber using a No. 10 sieve. Germ was then removed from the grits by hand separation. Corn grits were sized using a laboratory sifter (shop No. 130-U, Great Western Manufacturing Co., Leavenworth, KS) by sifting for 2 min. The grits that passed through a No. 5 sieve but held on a No. 10 sieve were used in this study.

Grits (750 g) from HTD and LTD corn were steeped with 1,400 ml of sulfurous acid solution (0.1%, based on the weight of the steepwater) at 50°C for 10 hr with and without added commercial protease. The initial pH of the steepwater was approximately 2.0, and it increased to 4.0 within 2 hr and to 4.5 at the end of the 10-hr steeping. The optimum pH range for the activity of the commercial protease (Rhozyme P11, Genencor, Inc., South San Francisco, CA) is 5-9, according to data provided by the manufacturer. The enzyme was added into the steepwater at a rate of 0.1% (based on the weight of the corn grits) 4 hr after the start of steeping to reduce the influence of low pH on enzyme activity. Two replicates were run for each condition.

The wet-milling procedure of Anderson (1963) was followed to separate fiber, starch, and gluten. The steeped grits were finely ground using a Quaker City mill in the presence of steepwater. The steepwater was carried forward as process water rather than being discarded. The wet-milled products, fiber, gluten, and starch were dried at 49°C for 24 hr for storage, and three 20-g samples of each fraction were dried at 103°C for 2 hr in an oven to determine the total dry weight of each fraction by calculation. Proximate analyses of all dry- and wet-milled fraction samples were performed by an outside commercial laboratory. Proximate analysis was performed using AOAC method 5 for crude fat (AOAC 1974), 7.003 for moisture, 7.074 for acid-detergent fiber, 7.009 for ash, and 7.033 for crude protein (AOAC 1984). Method 14.067 (AOAC 1984), which is based on nitrogen analysis, was used for determining protein content; the conversion factor used was 6.25. The nitrogen-free extract fraction, mainly carbohydrates, was calculated by subtraction of other fractions. Statistical analysis was done with Duncan's multiple range test (SAS 1982).

RESULTS AND DISCUSSION

The specific protease activities of the corn samples were 0.00293 and 0.00453 (units per minute per milliliter per milligram) for HTD and LTD corn, respectively, as determined by the procedure of Kerpisci (1988). The specific activity of LTD corn was approximately 1.5 times that of HTD corn.

The compositions of corn grits used for wet milling were determined by proximate analyses. The dry-weight percentages

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TABLE I **Yields and Protein Contents of Wet-Milled Corn Grit Products**

Treatment	Fiber (%, db)		Gluten (%, db)		Starch (%, db)	
	Yield	Protein Content	Yield	Protein Content	Yield	Protein Content
LTD ^a						
With protease	6.1 b ^b	28.9 a	8.1 b	48.2 a	80.5 a	0.3 b
Without protease	8.6 a	29.3 a	8.8 b	42.7 b	77.7 b	0.2 b
HTD .						
With protease	6.8 b	27.8 a	8.6 b	44.8 b	78.4 b	0.0 c
Without protease	8.8 a	28.7 a	11.6 a	29.4 c	74.1 c	0.6 a

^aLTD = corn dried at low temperature (30°C), HTD = corn dried at high temperature (80°C and 120°C).

of crude protein, crude fat, acid-detergent fiber, ash, and nitrogenfree extract fraction were 6.8, 1.5, 2.5, 0.3, and 89.2%, respectively, for LTD corn grits and 7.0, 0.9, 1.8, 0.3, and 89.8%, respectively, for HTD corn grits. Major differences existed in the fractions of crude fat (1.5 vs. 0.9%) and acid-detergent fiber (2.5 vs. 1.8%).

Effect of Protease on Product Yields

Protease significantly increased starch yield both in HTD corn (from 74.1 to 78.4%) and in LTD corn (from 77.7 to 80.5%) (Table I). Fiber yields decreased in HTD corn (from 8.8 to 6.8%) and in LTD corn (from 8.6 to 6.1%). Protease addition resulted in a decreased gluten yield in HTD corn (from 11.6 to 8.6%) but had no obvious effect on the gluten yield in LTD corn. This indicates that the increase in starch yield was mainly due to a more complete starch recovery from the fiber fraction for LTD corn and from fiber and gluten fractions for HTD corn. The use of protease successfully restored the losses in wet-milled starch caused by high-temperature drying.

Effect of Drying Temperature on Product Yields

LTD corn recovered 3.6 percentage points more starch than did HTD corn, and a difference of 2.1 percentage points was maintained after the addition of protease. However, drying temperature still showed a great influence on wet-milling properties of corn, especially the starch-gluten separation, as shown in Table I. HTD corn yielded 2.8 percentage points more gluten than LTD corn without enzyme treatment, but the gluten fraction of LTD corn had approximately 10% more protein. The major nonprotein component of gluten is starch.

One of the theories as to why high-temperature drying decreases starch yield involves the thermal denaturation of protease in the endosperm (Wahl 1970, Wall et al 1975, Kerpisci 1988). Our data support this theory and show that supplemental protease could greatly improve starch yield. The present study shows that removing the diffusion barriers before steeping with protease can shorten steep time and increase starch yield.

Protein Content of Wet-Milling Products

Protein content is considered an indication of quality of wetmilled products. No significant difference was observed in protein content of fiber (Table I), and no relationship was found for protein content of starch, except that HTD corn grits treated with the addition of protease had significantly lower value than did all other treatments. The protein content of this starch was only 0.04\%, which is considerably lower than that of starch from a normal wet-milling process (approximately 0.3%). This observed benefit for HTD corn probably is due to the more severe stress cracking that results from high-temperature drying, increasing the accessibility of the protein matrix to the enzyme. It may also be due to some change in the protein configuration. Protein solubility changes as a result of high-temperature drying. Such changes are the bases of the turbidity test and the ethanol solubility tests that corn wet millers have used.

The protein content of gluten was significantly improved by the addition of protease (5.5 percentage points for LTD corn and 15.4 percentage points for HTD corn). High-temperature drying, as shown, reduced protein content in gluten by 13.3 percentage points, but adding protease could increase the protein content to the level of that for LTD corn without protease.

CONCLUSIONS

The addition of protease had a significant effect on starch recovery for both HTD and LTD corn. It improved the starch yield of HTD corn by more complete starch-fiber and starchgluten separations; but it improved the starch yield of LTD corn only by better starch-fiber separation. High drying temperatures result in low starch yield mainly because of the increased difficulty in starch-gluten separation, which also results in low quality of wet-milled products. However, great improvement can be achieved by adding protease.

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LITERATURE CITED

- ANDERSON, R. A. 1963. Wet-milling properties of grains: Bench-scale study. Cereal Sci. Today 8:190-195.
- ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS. 1984. Official Methods of Analysis, 14th ed. Methods 7.003, 7.009, 7.033, 7.074, 14.067. The Association: Washington, DC.
- BREKKE, O. I., PEPLINSKI, A. J., GRIFFIN, E. I., and ELLIS, J. J. 1972. Dry milling of corn attacked by southern leaf blight. Cereal Chem. 49:466
- BROWN, R. B., FULFORD, G. N., OTTEN, L., and DAYNARD, T. B. 1981. Note on the suitability of corn exposed to high drying temperatures at different moisture contents. Cereal Chem. 58:75-76.
- COX, M. J. McMASTERS, M. M., and HILBERT, G. E. 1944. Effect of the sulfurous acid steep in corn wet milling. Cereal Chem. 21:447-465
- JAYASENA, W. V. 1988. Optimum steeping parameters of corn grits. M.S. thesis. Kansas State University, Manhattan, KS.
- KERPISCI, M. R. 1988. Effect of variety and drying temperature on proteolytic enzyme activity of yellow dent corn. M.S. thesis. Kansas State University, Manhattan.
- LE BRAS, A. 1982. Maize drying conditions and its resulting quality for wet-milling industry. Pages 95-125 in: Maize: Recent Progress in Chemistry and Technology. Academic Press Inc.: Orlando, FL.
- RANDALL, E. L. 1974. Improved method for fat and oil analysis by a new process of extraction. J. Assoc. Off. Anal. Chem. 57(5):1166-1168.
- SAS INSTITUTE. 1982. SAS User's Guide: Statistics, Version edition, p. 137. The Institute: Cary, NC.
- SPANHEIMER, J., FREEMAN, J. E., HEADLEY, V. E., and HEADY, R. E. 1972. Air classification of corn grits. 1. Softening grits with enzymes and chemicals. Cereal Chem. 49:131-141.
- WAHL, V. G. 1970. Biochemical-technological studies on wet-processing of maize. Part 4. The qualitative comparison of the corn-hydrolase system and the activity of particular enzymes and enzyme groups. Starch/Staerke 22:232-242.
- WAHL, V. G. 1971. Biochemical-technological studies on wet-processing of maize. Part 7. Model test for the determination of hydrolytic reactions by maize enzymes during the technical maize steeping process. Starch/ Staerke 23:212-218.
- WALL, J. S., JAMES, C., and DONALDSON, G. L. 1975. Corn proteins: Chemical and physical changes during drying of grain. Cereal Chem. 52:779-789.
- WATSON, S. A., and SANDERS, E. H. 1961. Steeping studies with corn endosperm section. Cereal Chem. 38:22-33.

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^bMean comparisons followed by the same letter are not significantly different in the same column, according to Duncan's multiple range test (P < 0.05).