

NOTE

Pilot-Plant Wet-Milling Process for Producing Corn Gluten Meal¹S. WU,² D. J. MYERS,^{2,3} L. A. JOHNSON,^{2,4} S. R. FOX,² and S. K. SINGH²**ABSTRACT**

Cereal Chem. 74(3):264–267

A pilot-plant wet-milling process was specially used to produce corn gluten meal (CGM) with a relatively high protein content (49.8% from Pioneer 3394 and 53.7% from Wilson D110). The protein content of the CGM obtained from a starch table in our pilot-plant procedure was similar to the results obtained by using the hydrocyclone method. Wilson

D110 has a higher protein content in its corn kernels, producing higher yield and protein content gluten than the Pioneer 3394, which has lower protein content in the corn. The effects of drying method and temperature on the color of CGM were also discussed.

The amount of corn processed by the wet-milling industry has grown rapidly as the demand for sweeteners and fuel-grade ethanol has increased. Of 7.4 billion bushels of corn produced in 1995, 20.6% was processed by wet-milling (Lugar 1996). As a coproduct of wet-milling, the amount of corn gluten meal (CGM) produced has also increased. It was estimated that $\approx 1,031$ million kg (2,269 million lb.) of CGM was produced in 1995 (Urbanic 1996).

Both laboratory-scale and pilot-plant-scale wet-milling processes can be used to evaluate the wet-milling characteristics of corn and the effect of processing techniques on product yield and quality (Watson et al 1951; Anderson 1957, 1963; Eckhoff et al 1993). A pilot-plant study is the process of choice when large samples of starch or other by-products are required for further study, or when data are required to generate information needed for plant scale-up. Rubens (1990) described a pilot-plant wet-milling process that more closely parallels commercial milling operations. For dent corn, Rubens' process had a starch yield of 58.8% with 0.63% protein and a gluten yield of 7.6% with 53.8% protein.

Because high starch recovery is the primary goal of wet-milling, there is little attention on the milling process to improve the quality of protein in the CGM. However, the severity of artificial drying can affect gluten yield and protein quality. MacMasters and his coworkers (1959) reported that the protein content in gluten decreased, and starch recovery decreased, as the corn drying temperature increased from ambient temperature to 93.3°C. The amounts of salt-soluble proteins and the proteins dissolved in 0.01*N* KOH solution significantly decreased when corn was dried at high temperature (93.3 or 143°C) (McGuire and Earle 1958, Wall et al 1975).

Commercially, gluten meal is then dried by flash, rotary, or steam-heated tubular dryers (Blanchard 1992). Flash dryers have a relatively short dryer retention time (several seconds); they proc-

ess less material and have less thermal efficiency than the other dryers. Rotary dryers are relatively inexpensive and simple but are cumbersome and difficult to control, so that scorching of gluten is common. Steam tubular dryers are more energy efficient than flash dryers and less expensive. Therefore, the steam tubular system has been the dryer of choice in recent years. Drying temperatures <400°C are recommended to avoid a dark-colored product, burnt particles, and offensive odor or haze in the dryer exhaust (May 1987).

Neumann et al (1984) reported that wet CGM contained more salt-soluble proteins than does dried CGM, but the amount of alcohol-soluble proteins was only slightly different between wet and dried CGM obtained from a large commercial plant. Because drying CGM has been shown to influence CGM quality, the objectives of the research were: 1) to modify a pilot-plant wet-milling process to obtain CGM in relatively high yield and protein content, and 2) to evaluate the influence of hybrid and drying process on CGM color.

MATERIALS AND METHODS**Maize**

Two varieties of yellow dent maize grown in 1994 were selected based on protein content and availability. One hybrid, Wilson D110, had 10.2% (db) protein and 1.31 density, and the other hybrid, Pioneer 3394, had 8.6% (db) and 1.28 density. The harvested kernels were dried at ambient temperature in a bin with an air system to 13–14% mc and stored at 4.4°C.

Wet-Milling Procedure

The pilot-plant design and procedure were based on the process design of Anderson (1957) and Rubens (1990), with the goal of obtaining gluten with a protein content higher than that usually obtained in laboratory milling and similar to that achieved in commercial production. A 265-L conical bottom steeping tank (model 70-gallon JOVC, Process/Storage System, Charlevoix, MI) was used with a hot-water jacket system to control the steep-water temperature at 48–50°C. Corn kernels (20 kg) were steeped for 40 hr in a 50-L solution containing 0.2% sulfur dioxide and 0.44% lactic acid. Degermination was accomplished by first grinding in a Sprout-Bauer 0.35-m dice mill (Reliance Electric Co., Cleveland, OH) at a speed of 900 rpm, with a suitable plate gap based on kernel size, and a feed speed of 300 rpm. A continuous stream of distilled water at the rate of 5 L/min was supplied to prevent clogging and reduce heat buildup. The specific gravity of the ground corn was then adjusted to 1.04–1.05 to facilitate germ floatation. The germs were separated by hand with a wire-mesh

¹Journal Paper, J-16804 of the Iowa Agriculture and Home Economics Experiment Station, Ames, IA, 50011. Project no. 0161. Research supported by the Iowa Corn Promotion Board, Center for Crops Utilization Research and the Iowa Agriculture and Home Economics Experiment Station.

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screen and washed through a 60-mesh (250- μ m opening size) screen with three 3-L portions of water. The washwater was transferred back to the degerminated slurry, and a second grinding was performed (Stephan Food Processing Technology, Germany) with 0.005- and 0.02-mm knife gaps at 3,200 rpm. After the second grind, the fiber was separated on a separator (model K30-1-SS, Kason Corporation, Linden, NJ) with a 0.76-m dia., 200-mesh (75- μ m opening size) stainless-steel screen. The separated fiber was washed with 150 L of fresh distilled water at a 5 L/min rate through the 200-mesh screen.

The starch and gluten were separated from the remaining mill starch fraction by means of a starch table, 6.1 m long and 0.51 m wide with a slope of 0.54° (0.057 m pitch) for the entire length. The specific gravity of the mill starch slurry was adjusted to 1.04 using the fiber washwater, and the slurry was pumped onto the table at a rate of 1,000 mL/min. The gluten fraction was collected in a bucket at the distal end of the table. Distilled water (\approx 35 L) was used to wash the tabled starch and collected as the washwater fraction. The gluten collected from the table overflow was stored in a cooler (5°C) for future processing.

The fiber and germ fractions were dried in a forced-air oven for 48 hr at 50°C. The starch was air-dried on the table at room temperature with an air fan for 24 hr, and then dried in a forced-air oven for 48 hr at 50°C.

Gluten Treatment

After the gluten slurry was allowed to settle by storing at 5°C overnight (\approx 15 hr), it was concentrated to 60–80 L by siphoning off the supernatant. Based on the efficiency of a vacuum-drum filter, most (80% by volume) of the slurry was dewatered by using a vacuum-drum filter (Filtration Engineers, East Moline, IL) with a belt covering the filter surface (polypropylene, 3–8 μ m retention) for the purpose of building up the cake. The resulting gluten cake (53–60% mc) was divided into three parts and dried in a forced-air oven at 50, 100, and 150°C until the moisture content of gluten was <10%.

The remaining (20%) gluten slurry was allowed to settle again and concentrated to a level of 1.8–2.0 % solids by siphoning off the supernatant. Half of the concentrated slurry was dried with a spray drier and the other half with a freeze drier. Spray-drying was performed with a Pulvis mini-spray (model GA-31, Yamato Scientific Co., Tokyo, Japan) with an inlet temperature of 120°C, an outlet temperature of 60°C, and 7.5–8.0 mL/min sample feed rate. Freeze drying was performed in a freeze drier (model Ultra 35 SL, Virtis Company, Inc., Gardener, NY). A 50.8 \times 30.5 \times 2.5 cm³ stainless-steel box filled half deep with gluten slurry was pre-frozen at –20.5°C before it was placed into the freeze drier. Freeze drying was performed at 80 millitorr vacuum and 25–27°C shelf temperature for 72 hr.

The milling procedure and gluten drying processes were replicated three times for each hybrid. All the dried CGM was stored at 4°C until use.

Sample Analysis

The initial moisture, starch, protein, and oil contents of maize kernels were determined by a GAC III fixed-filter, near-infrared reflectance (NIR) analyzer (Dickey-John Corp., Auburn, IL). The starch, protein, and oil data were reported on a dry basis (db).

The wet-milling fractions, dried germ, fiber, starch, steepwater, starch washwater, and the first concentrated gluten slurry were analyzed for proximate composition. Moisture contents of the wet-milling fractions were determined by drying a 2.00-g sample in a convection oven for 3 hr at 130°C (method 44-15A, AACC 1995). Crude fat was determined by using the Goldfisch method (method 14-084 and 14-085, AOAC 1984). Protein contents were determined by measuring total nitrogen content using the Kjeldahl method and a protein conversion factor of 6.25 (method A-18, CRA 1986). The yields of wet-milling fractions (starch, fiber,

gluten, germ, steepwater solids, and washwater solids) were determined as the percentages of initial maize dry solids. The recovery of starch (or protein) was calculated as the ratio of the total weight of starch (or protein) recovered from wet-milling to the total weight of starch (or protein) present in the corn.

The moisture content of the dried corn gluten meal was determined by the Karl Fischer method (method E203-75, ASTM 1975). The color of the CGM samples was measured with the HunterLab Labscan (Hunter, VA).

Statistical Analysis

The general linear model and the test of least significant difference (LSD) at the 5% level were used to evaluate means.

RESULTS AND DISCUSSION

Yields and Recoveries of Wet-Milling Products

Table I shows the proximate analysis data of the two selected hybrids used to produce CGM. The Wilson D110 had higher protein and oil content and lower starch content than did Pioneer 3394.

The starch yields of two hybrids were low (Table II) compared with the typical values of plant-scale (67.5%) (Johnson 1991) and lab-scale (59.2–65.1%) milling (Watson and Yahl 1967), but they were similar to the pilot-plant results of Rubens (1990). Pioneer 3394 produced higher starch yield than did Wilson D110. The

TABLE I
Proximate Compositions of Maize Hybrids

Composition	Pioneer 3394	Wilson D110
Moisture (%)	14.0	13.9
Starch (% db)	72.6	71.5
Protein (% db)	8.6	10.2
Oil (% db)	3.4	4.5

TABLE II
Yields, Protein and Oil Contents, and Recoveries of Wet-Milled Products^a

Product	Pioneer 3394	Wilson D110	Rubens ^b
Starch			
Yield (% db)	60.4 \pm 0.7	57.9 \pm 2.0	58.8
Starch Recovery (%)	82.8	80.5	
Protein (% db)	0.49 \pm 0.10	0.46 \pm 0.04	0.63
Protein recovery (%)	3.5	2.6	
Oil (% db)	0.06 \pm 0.02	0.06 \pm 0.05	
Gluten			
Yield (% db)	6.0 \pm 0.2	7.4 \pm 0.2	7.6
Protein (% db)	49.8 \pm 1.0	53.7 \pm 1.8	53.8
Protein recovery (%)	35.0	39.0	
Oil (% db)	11.0 \pm 1.7	7.0 \pm 0.8	
Fiber			
Yield (% db)	21.5 \pm 3.2	18.6 \pm 1.5	21.8
Protein (% db)	8.9 \pm 0.2	10.8 \pm 0.4	
Protein recovery (%)	22.2	19.7	
Oil (% db)	3.0 \pm 0.4	2.1 \pm 0.4	
Germ			
Yield (% db)	5.9 \pm 0.1	6.6 \pm 0.4	10.5
Protein (% db)	12.6 \pm 0.1	14.4 \pm 0.4	
Protein recovery (%)	8.6	9.3	
Oil (% db)	36.0 \pm 1.4	47.0 \pm 0.5	
Steepwater			
Yield (% db)	4.4 \pm 0.07	5.3 \pm 0.2	5.1
Protein (% db)	34.7 \pm 0.4	34.5 \pm 1.6	
Protein recovery (%)	17.8	17.8	
Wash water			
Yield (% db)	0.4 \pm 0.09	0.6 \pm 0.03	
Protein (% db)	7.7 \pm 1.2	14.5 \pm 5.6	
Protein recovery (%)	0.36	0.80	
Total solids recovery (%)	98.6 \pm 3.37	96.3 \pm 0.18	

^a All data are the means of three millings.

^b Rubens 1990.

protein contents of the starch for both hybrids were <0.5%. Even though the protein contents were >0.3%, typical values in commercial starch, the quality of starch produced in our procedure was better than that produced by Rubens (0.63%).

Wilson D110 maize produced significantly higher ($P < 0.05$) gluten yield with a significantly higher ($P < 0.05$) protein content than did Pioneer 3394. These results were also anticipated because Wilson D110 maize had a higher protein content. The gluten yields and protein contents obtained by our pilot-plant procedure were similar to the results of Rubens, although Rubens used a hydrocyclone procedure compared to our starch table procedure to separate gluten and starch. With tabling as the separation method for starch and gluten, the yield of our gluten was lower than yields of previous laboratory studies (6.9–7.9%), but the protein content of gluten was higher than that reported (40.8–46.0%) (Watson and Yahl 1967, Eckhoff et al 1993).

Although the fiber yields of our process were close to the fiber yields of Rubens (1990), it was higher than the typical industrial value of 11.5% (Johnson 1991). The germ yield of our procedure was low because germs, especially broken germs, were not easily skimmed out by hand. The residue from the broken germs in the second grind is believed to have caused the relatively high oil content in the Pioneer 3394 gluten (11.0% db). The oil content in the gluten produced by an industrial wet-milling plant is ≈7% db (Johnson 1991).

Our process yielded more steepwater solids than the lab-scale (3.9–4.0%) wet-milling process did (Watson and Yahl 1967). Wilson D110 produced significantly higher steepwater yields than did Pioneer 3394. There was no significant difference in the steepwater protein contents of the two hybrids (34.5 and 34.7%); however, these results were lower than those of lab-scale (58.6–63.4%) milling (Watson and Yahl 1967). The low protein loss in the steepwater may have contributed to the high protein content in the gluten. The washwater fraction was specifically produced for the production of a high-quality starch and high protein content gluten. The yields of washwater were low, 0.4–0.6%, with a protein content of 7.7% for Pioneer 3394 and 14.5% for Wilson D110. Comparable data reported by Watson and Yahl (1967) had 6.7% yield and 13.4% protein content for lab-scale experiments.

Effect of Drying on CGM Color

The colors of CGM dried with different drying methods were significantly different (Table III). As oven temperature increased, the *L* value (lightness) significantly decreased, and *a* value (+*a* value = red color) significantly increased for both hybrids. From 50 to 100°C, the *L* value decreased about 10 units, and *a* value increased over 2 units; whereas increasing the drying temperature

from 100 to 150°C, decreased the *L* value by about 3.5 units for Wilson CGM and 1.7 units for Pioneer CGM, and increased the *a* value by 0.7 and 0.4 units, respectively. The color change in CGM from 50 to 100°C was much greater than from 100 to 150°C. The *b* value (+*b* value = yellow color) also varied among treatments, but the variation in the *b* value was less than for the other two parameters. Compared with the oven drying, the freeze- and spray-dried CGMs were lighter and the intensity of red was lower because the CGM was subjected to less heat during freeze- and spray-drying than in oven-drying. The spray-dried CGM was lighter and had less red color than the freeze-dried CGM because it was subjected to less heat relative to freeze-drying. In spray-drying, the gluten was dried in only a few seconds at a temperature range of 120–60°C, compared with 72 hr at 25–27°C shelf temperature in the freeze dryer. The CGM from Pioneer 3394 was more red and yellow in color than the CGM of Wilson D110 because the hybrid Pioneer 3394 maize kernels were more red and yellow than the Wilson D110. The higher crude fat content with attendant pigments might also play a role in the darker color of Pioneer 3394 CGM.

CONCLUSIONS

The pilot-plant procedure successfully produced gluten with a high protein content compared with that of laboratory-scale milling. Maize hybrids with different initial protein contents affect gluten yield and protein content of CGM. The color difference of CGM was attributed to the drying method and maize hybrid. As the temperature increases, the lightness of the CGM decreases, and red color intensities increase.

ACKNOWLEDGMENTS

Special thanks to Charles Hurburgh and the Iowa State University Grain Quality Lab for providing maize and the NIR proximate composition analysis of corn. Thanks also to Mark Ruber for his cooperation in the pilot plant.

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TABLE III
Colors of Dried Corn Gluten Meal (CGM) Samples^a

CGM	Color ^b		
	<i>L</i>	<i>a</i>	<i>b</i>
Wilson D110			
Oven-dried			
50°C	74.35a	2.32a	23.41a
100°C	64.14b	4.74b	24.48bc
150°C	60.65c	5.46c	24.70bd
Freeze-dried	77.82d	1.88d	24.91d
Spray-dried	78.69d	1.15e	24.17b
Pioneer 3394			
Oven-dried			
50°C	66.94e	6.40f	26.02e
100°C	59.57fc	8.52g	25.48f
150°C	57.87g	8.98h	25.46f
Freeze-dried	76.31h	4.00i	27.09g
Spray-dried	77.78hd	2.93j	27.19g

^a Means of three millings.

^b Data in same column with different letters are significantly different at $P < 0.05$.

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[Received May 28, 1996. Accepted January 19, 1997.]