

Recovery of Starch and Protein from Wet-Milled Corn Fiber

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Physical and chemical methods were used to recover starch and protein from wet-milled corn fiber. A single milling of the fiber produced an 18% yield of mill starch. By separating the mill starch with a starch table, 68% of this material was recovered as starch with a protein contamination of 0.66%. Milling increased fine fiber from 4.5% in the start-

ing material to 11.5% after a single grind. Successive additional milling passes modestly increased the mill starch and fine fiber yields with a corresponding decrease in the coarse fiber yield. Pretreatment with combinations of lactic and sulfurous acids had only a small effect on the distribution and composition of the recovered fractions.

Fiber is an important coproduct of the corn wet-milling process. Currently, corn fiber forms the main component of corn gluten feed, a 21% protein animal feed sold almost exclusively in Europe. Combined with steeping solubles, stillage, and germ residue, corn gluten feed accounts for 20–24% of the wet-milled raw corn mass. On a per pound basis, however, this material is the least valuable of the wet-milled products and generally sells for less than the price of unprocessed corn. Corn fiber has also been tested and marketed as dietary bran (Anonymous 1985, Sosulski and Wu 1988, Artz et al 1990), but poor food functionality and concerns about aflatoxin have limited its use as a commodity ingredient. Consequently, alternative uses for this material are being sought. Recent reports have identified potentially valuable components in fiber (Doner and Hicks 1996, Moreau et al 1996) and have studied the potential of using this material as an ion-exchange resin (Wing 1996). Methods are also being developed to convert the fiber's cellulosic and hemicellulosic fractions into fermentable sugars and related compounds (Carlson 1994; Esquivel et al 1994; Leathers and Gupta 1994, 1996; Picataggio and Finkelstein 1996).

The corn wet-milling process is effective at separating the components of the kernel. Greater than 90% of the starch, 85% of the oil, and over 50% of the protein, the last as a high-value gluten meal, is recovered. Much of the remaining starch, protein, and oil is separated with the fiber. After its initial screening from the milled slurry, the fiber is washed and abraded over several additional screens to help release bound starch before being dewatered by centrifugation or pressing. On a dry basis (db), dewatered fiber contains 18–25% starch and 10–13% protein. This relatively high starch content suggests that additional recovery might be feasible.

The objective of this research was to determine the potential of recovering additional starch and protein from corn fiber. Additional milling of fiber was studied using a laboratory-scale plate mill. The production of fine fiber was also monitored because this material can create difficulties in processing and drying (May 1987). To test that the chemical events that occur during corn steeping were complete, lactic and sulfurous acid pretreatments were also studied.

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MATERIALS AND METHODS**Sample Collection and Handling**

Pekin Energy Co. (Pekin, IL) supplied corn fiber, collected immediately downstream of the dewatering presses. The samples were stored frozen because initial experiments indicated that no significant differences in fraction yield or composition were obtained due to the freezing. Individual bags of fiber were thawed as needed. Once thawed, the material could be maintained refrigerated for two to three weeks before microbial degradation became noticeable. Unused sample was discarded after this period.

The starting material can be classified into coarse and fine fiber fractions, free starch, protein, and solubles. To estimate the distribution of the starting material, a 70-g sample was washed thoroughly over a 40-mesh sieve with a gentle stream of water (8 L total volume). The retained material was considered to be coarse fiber. The filtrate was then drained through a 325-mesh sieve and was again washed (4 L total volume). This retained material was considered to be fine fiber. For each bag of fiber sample used, moisture, starch, and protein were measured for the bulk material as well as for the individual coarse and fine fiber fractions.

Fiber Milling and Fraction Separation

Fiber (70 g) and water (250 mL) were mixed to form a slurry and were processed as illustrated in Figure 1. The processing consisted of three basic steps (milling, screening, and centrifuging) and yielded four process fractions. The slurry was milled one to four times with a plate mill (Quaker City model 4E, The Straub Co., Warminster, PA) fitted with fine grinding plates (preconditioned by grinding for 100 hr with a gentle stream of water) and operated as has been described for laboratory-scale wet-milling (Eckhoff et al 1996). Clean water (100 mL) was used to help move fiber through the mill during each grind, except for the third and fourth passes of the three and four grind experiments. For these passes, 100 mL of previously decanted slurry water was used to limit the total volume of added water. After grinding, the mill was disassembled, and the mill hopper, grinding plates, and other fiber-exposed surfaces were washed with 400 mL of clean water. This water was collected separately and used in coarse fiber washing.

The washing procedure was designed to thoroughly abrade the milled fiber and to separate coarse and fine fiber fractions. After milling, the slurry was poured over a 40-mesh sieve placed in the bottom of a bucket with the liquid level just covering the fiber. The assembly was shaken for 5 min on a sieve shaker (model RX-86, W. S. Taylor, Cleveland, OH). After allowing the residual liquid to drain into the bucket, the sieve and retained fiber were placed in a second bucket. The wash water previously used to clean the mill was added, and the assembly was shaken again. This procedure was repeated a third time with clean water. The

retained coarse fiber and sieve were rinsed with a final 100 mL of water, which was collected in the third bucket. The coarse fiber was quantitatively transferred into aluminum pans for drying. To recover fine fiber, the first filtrate from the 40-mesh screenings was poured over a 325-mesh sieve placed in the bottom of a bucket and was shaken for 5 min with the sieve shaker. After draining, the retained fine fiber and sieve were placed in a second bucket. This procedure was repeated using each less concentrated coarse fiber filtrate over the same sieve. A final fourth washing was accomplished with 400 mL of fresh water. The retained fine fiber and sieve were washed with a final 100 mL of clean water, and the fine fiber was transferred onto a glass petri dish for drying. To separate mill starch (starch and protein) from solubles, the four 325-mesh filtrates were combined and centrifuged (Sorvall, model RC-5B, Newtown, CT) at $8,000 \times g$ for 15 min. Steinke and Johnson (1991) used a similar procedure for laboratory-scale wet-milling, except that in this work the starch and protein were not separated. To determine solubles, the total volume of the decanted supernatant was measured, and three 100-mL aliquots were transferred into aluminum pans for drying. All fractions were dried initially at 50°C for 24 hr. A portion of each fiber

fraction, the mill starch, and the soluble aliquots were then dried for 2 hr at 130°C to determine dry masses and yields. The remaining coarse and fine fiber samples were used to measure starch and protein. All experiments were conducted in triplicate.

Chemical Pretreatment

For some experiments, the fiber was chemically pretreated before milling. Fiber (70 g) was weighed into a 500-mL screw-cap volumetric flask and was combined with 350 mL of one of the following solutions: 0.5% lactic acid, 0.4% sulfurous acid (0.59 g sodium metabisulfite per 100 mL of water), or 0.5% lactic and 0.4% sulfurous acids. Lactic acid was from Fisher Scientific Co. (Fair Lawn, NJ) and sodium metabisulfite was from J. T. Baker, Inc. (Phillipsburg, NJ). The flasks were agitated for 2 hr in an orbital waterbath (Labline, model 3540, Melrose Park, IL) operated at 50°C and 200 rpm. After treatment, the slurry was processed as above with the solutions used as process water.

Protein and Starch Analysis

The dried fiber fractions were analyzed for protein and starch. Nitrogen was determined using a nitrogen analyzer (Leco, model FP-428, St. Joseph, MI). A conversion factor of 6.25 was used to estimate protein. Starch was measured by the enzymatic assay of McCleary et al (1994) without the pullulanase step. Glucoamylase, glucose oxidase, and peroxidase were from Sigma Chemical Co. (St. Louis, MO) and α -amylase was a gift from Novo Nordisk BioChem (Danbury, CT). Both analytical procedures were conducted in duplicate with the average of the determinations taken as the result.

Separation of Starch and Protein

The mill starch fractions from four one-grind experiments were combined to obtain sufficient material to separate with a starch table. The combined mill starch slurry was allowed to settle for 30 min, and approximately half of the volume was carefully decanted to yield a concentrated fraction and a diluted fraction. The slurry was tabled on a 5.08 cm \times 2.44 m rectangular aluminum channel operated as described by Eckhoff et al (1996). In succession, the concentrated fraction, the diluted fraction, and 1 L of clean water were pumped onto the table. Recovered starch was scrapped into an aluminum pan, dried at 50°C for 24 hr, and weighed. A 2-g portion was then dried at 130°C for 2 hr to determine the yield of

TABLE I
Yields (% db) of Fiber Fractions, Mill Starch, and Soluble Material from Milled Corn Fiber^a

Fraction	Starting Material	Number of Milling Passes				
		0 ^b	1	2	3	4
Coarse fiber	87.8 (1.7)	74.0a (0.8)	65.8b (1.4)	63.2c (0.2)	62.6c (1.4)	60.1d (0.1)
Fine fiber	4.5 (0.6)	13.5a (0.7)	11.8b (1.4)	12.8a,b (1.4)	13.6a,b (1.7)	14.5a (0.3)
Mill starch		9.7a (0.4)	18.0b (0.4)	20.3c (0.3)	20.7c (0.5)	21.2c (0.8)
Solubles		2.9a (0.1)	3.1a,b (0.2)	3.3b (0.1)	3.3b (0.1)	3.4b (0.3)
Total yield		100.2 (1.7)	98.8 (0.3)	99.8 (1.5)	100.2 (1.1)	99.2 (0.5)

^a Standard deviations are given in parentheses. Letters represent analysis of variance for all possible mean comparisons. Variances have been arcsin-transformed to improve the implicit assumption of equal variances. Different letters in each row indicate a significant difference at $\alpha = 0.05$.

^b Fiber washing only.

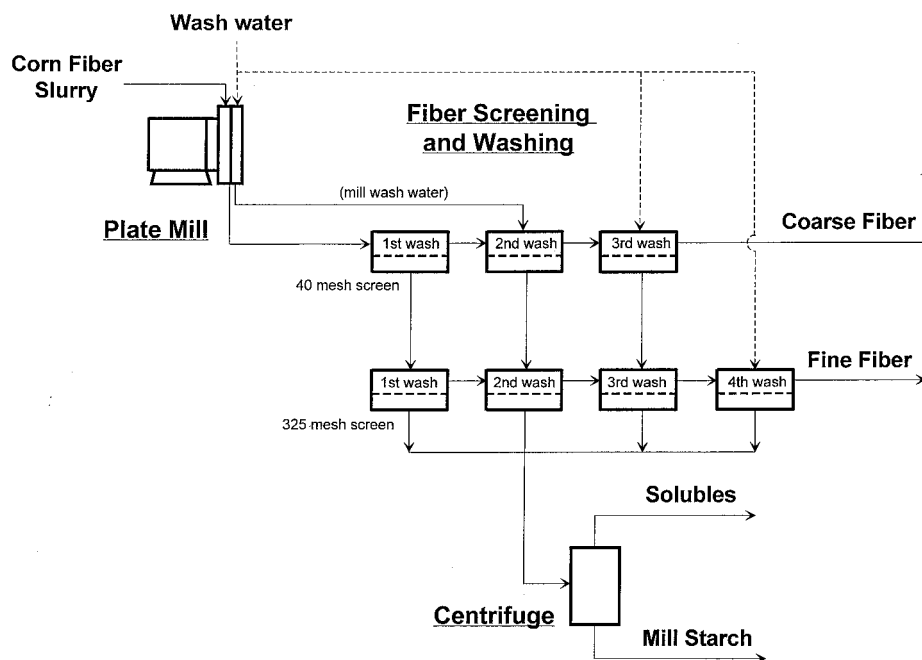


Fig. 1. Milling and separation steps for laboratory-scale processing of corn fiber.

dry matter. The total volume of the table overflow was measured, and three 100-mL aliquots were dried at 50°C for 24 hr followed by 130°C for 2 hr to determine the yield of protein plus solubles. Due to the large volume of table overflow and the difficulties of filtering protein, no attempt was made to separate the protein from the solubles.

RESULTS AND DISCUSSION

Fractionation of the starting material yielded 87.8% coarse fiber and 4.5% fine fiber (Table I). The remaining material was free starch (5–6%), protein (1.2%), part of which may be soluble, and other soluble material. Coarse fiber consists of material primarily from the kernel pericarp, while the fine fiber fraction consists of cell-wall material from the endosperm region and small pieces of pericarp (Eckhoff and Tso 1990). Fine fiber will also include small protein-starch complexes (Watson et al 1955). The initial high level of free starch in the starting material is due to starch freed in the screening process that partitions with the fiber during dewatering as well as starch freed by the abrasion and shearing of the fiber by the dewatering presses. The bulk starch and protein concentrations in the starting material were $18.9 \pm 1.1\%$ and $12.4 \pm 0.4\%$, respectively.

Yields of the four fractions are shown in Table I. A single milling of the fiber produced 65.8% coarse fiber, 18.0% mill starch, 11.8% fine fiber, and 3.1% solubles. Each additional milling pass resulted in a small additional increase in the yields of mill starch and fine fiber with a corresponding decrease in the yield of coarse fiber. After four grinding passes, the yields were 60.1% coarse fiber, 14.5% fine fiber, 21.2% mill starch, and 3.4% solubles. Washing the fiber without milling (0 grind treatment) produced a comparable amount of fine fiber (13.5%) but only half the yield of mill starch (9.7%).

Bound starch in the combined fiber fractions decreased from 13.9% in the starting material to 5.3% after a single grinding pass to 3.2% after four passes (Table II). Based on the initial starch content of 18.9%, a single milling of the material yielded 78% of the starch as mill starch. Although some statistical anomalies exist in the data, in general, additional milling passes gave coarse and fine fiber fractions with successively less starch. Of the two fiber fractions, fine fiber always had higher starch levels than coarse fiber, although in terms of total unrecovered starch, more starch was associated with the coarse fiber. Treating the fiber by washing alone produced coarse and fine fiber fractions with somewhat less starch than in the starting fractions. By material balance, approximately half the starch freed from the coarse fiber by this treatment remained as starch-protein and starch-protein-fiber particles that separate with the fine fiber. A large decrease in the starch content of both fiber fractions occurred when a single milling pass was included in the processing.

Protein levels in the individual fiber fractions decreased modestly with increased milling (Table III). Of the initial protein in the

sample (12.4%), 64% remained with the fiber after one additional grind and 58% remained after four grinds. For all washing and milling treatments, fine fiber had more protein (22–25%) than coarse fiber (7–11%). As for the starch distribution, more total protein remained with the coarse fiber than with the fine fiber.

Chemical pretreatments were conducted at 50°C for 2 hr because these conditions were reported to release most of the starch from cut endosperm sections of dent corn (Watson and Sanders 1961). A two-pass milling treatment was used for these experiments. Treatment with lactic acid or sulfurous acid individually did not significantly increase the mill starch yield, but treatment with both acids did increase the yield slightly (Table IV). The chemical treatments did not significantly reduce the residual starch in the coarse or combined fiber fractions beyond that of milling alone (Table V). The fine fiber fraction, though, appeared to have somewhat higher levels of starch, which may be related to the significant reduction in the protein content of this fraction particularly when the chemical treatment included sulfurous acid (Table VI). The reduction in fine fiber protein was large enough to result in a significant reduction in the protein of the combined coarse and fine fibers (Table VI). Because the reduction in fiber protein did not correlate with a reduction in fiber starch, the remaining starch ($\approx 15\text{--}20\%$ of the initial fiber starch) is likely very tightly bound. This portion of the starch will be difficult to recover as granules.

TABLE III
Residual Fiber Protein (% db) from Milled Corn Fiber^a

Fraction	Starting Material	Number of Milling Passes				
		0 ^b	1	2	3	4
Coarse fiber	10.7 (0.5)	8.3a (0.2)	7.6b (0.5)	7.0c (0.2)	7.1c (0.04)	6.8d (0.2)
Fine fiber	23.3	24.3a,b (0.4)	25.2a (0.8)	23.5b,c (0.3)	23.0c (0.6)	21.8d (0.1)
Combined average	11.3	10.8a (0.2)	10.3b (0.2)	9.8b,c (0.3)	9.9c (0.2)	9.7c (0.2)

^a Standard deviations are given in parentheses. Letters represent analysis of variance for all possible mean comparisons. Variances have been arcsin-transformed to improve the implicit assumption of equal variances. Different letters in each row indicate a significant difference at $\alpha = 0.05$.

^b Fiber washing only.

TABLE IV
Yields of Fiber Fractions, Mill Starch, and Soluble Material (% db) from Chemically Treated and Milled Corn Fiber^a

Fraction	Chemical Pretreatment (50°C, 2 hr) ^b			
	Untreated	LA	SO ₂	LA + SO ₂
Coarse fiber	63.2a (0.2)	63.0a (2.0)	63.8a (1.8)	63.4a (1.0)
Fine fiber	12.8a (1.4)	13.6a (1.4)	13.0a (1.0)	12.2a (0.6)
Mill starch	20.3a (0.3)	19.7a (0.5)	20.6a (0.6)	21.8b (0.5)
Solubles ^c	3.3a (0.1)	4.4b (0.3)	8.1c (0.5)	10.3d (0.4)
Total yield ^d	99.8 (1.5)	100.8 (2.0)	105.5 (1.4)	107.7 (1.3)
Theoretical yield ^e	100	107	107	113

^a All samples were milled twice. Standard deviations are given in parentheses. Letters represent analysis of variance for all possible mean comparisons. Variances have been arcsin-transformed to improve the implicit assumption of equal variances. Different letters in each row indicate a significant difference at $\alpha = 0.05$.

^b LA = 0.5% lactic acid; SO₂ = 0.4% sulfurous acid.

^c Includes mass of solution chemicals.

^d Total dry mass recovered/initial dry mass of fiber $\times 100\%$.

^e Initial dry mass of fiber plus added chemicals/initial dry mass of fiber $\times 100\%$.

TABLE II
Residual Fiber Starch (% db) from Milled Corn Fiber^a

Fraction	Starting Material	Number of Milling Passes				
		0 ^b	1	2	3	4
Coarse fiber	12.9 (0.4)	8.2a (1.4)	3.8b (1.2)	2.5b,c (0.4)	2.4c (1.3)	1.5c (0.2)
Fine fiber	33.1	29.5a (1.4)	13.2b (2.1)	9.8c (1.6)	8.3c (1.4)	10.4b,c (1.3)
Combined average	13.9	11.5a (1.1)	5.3b (0.6)	3.7c (0.1)	3.4c (1.0)	3.2c (0.3)

^a Standard deviations are given in parentheses. Letters represent analysis of variance for all possible mean comparisons. Variances have been arcsin-transformed to improve the implicit assumption of equal variances. Different letters in each row indicate a significant difference at $\alpha = 0.05$.

^b Fiber washing only.

In the four single-grind experiments combined to recover starch, the yield of starch, protein, and solubles was 20.8%, similar to the $21.1 \pm 0.6\%$ yield (Table I) for the mill starch and solubles obtained in the initial work. By tabling, 68% of this mill starch was recovered as starch. Therefore, 53% ($0.68 \times 78\%$) of the starch that would have left the wet-milling process with the fiber was recovered. Assuming a wet-milling fiber yield of 13% (May 1987), recovery of this material would increase the overall wet-milling starch yield by $\approx 1.3\%$.

Protein contamination of the separated starch was 0.66%, greater than the 0.3–0.54% range of values typically obtained from starch tabling in laboratory wet-milling procedures (Singh and Eckhoff 1996) and greater than the 0.3–0.35% values typically achieved with hydrocyclones in industrial operations. However, adding this recovered starch to the bulk of the wet-milled starch would increase the protein contamination of the combined product by $<0.01\%$. The greater protein content of the starch was not surprising due to the increased likelihood that small tightly bound complexes of protein and starch would also be recovered by the separation scheme. With continued milling, small particles of fiber can also separate with the mill starch. While the presence of fine fiber in mill starch can detrimentally affect the separation of starch from protein, a thorough abrading of the fiber during washing has been shown to reduce these problems (Eckhoff and Tso 1990).

By material balance, protein in the recovered gluten fraction is estimated to be between 25 and 50%. The wide range of uncertainty is due to the unknown amount of protein contained in the solubles. Laboratory wet-milling procedures also produce gluten fractions with similar protein concentrations. In comparison, the industrial process produces a 60% protein gluten fraction. This difference in industrial and laboratory processing occurs because of the use of starch tables in the laboratory-scale processes. This technique generally yields a high quality starch fraction but with a reduced starch yield and a less concentrated protein fraction (Eckhoff et al 1993). The presence of fine fiber in the mill starch would also contribute to the protein reduction because this material would tend to separate with the gluten fraction. Conservatively assuming that the protein concentration of the recovered gluten fraction is 25% and that the wet-milling yield of gluten meal is 5.7% (May 1987), the reduction in the protein concentration of the combined gluten product would be $<0.5\%$. The gluten yield would increase by $\approx 0.1\%$.

Based on laboratory and pilot wet-milling studies, it is not clear if hydrocyclone processing of the recovered mill starch would improve the starch-protein separation. A recent, kilogram-scale comparative study concluded that hydrocyclone separation of mill starch did not yield as pure a starch fraction as tabling (Singh and Eckhoff 1995). In contrast, industrially produced starch separated with hydrocyclones typically has a protein concentration equal to the best values achieved by laboratory-scale tabling. These con-

flicting results are likely due to differences in the configuration and operation of the hydrocyclones.

The experiment described in this article was not designed to exactly represent a modified milling scheme. The fiber used in this study was extensively washed over screens and dewatered in the plant before being recovered. Ideally, additional milling would occur after a single screening of the material exiting the third grind but before the intensive fiber washing steps. While it was possible to obtain material after the first screening, it was impracticable to transport or store this wet material. Consequently, dewatered fiber from further downstream was used, and it is assumed that the starch already freed and recovered during the process fiber washing would also be recovered by adding a milling step prior to these washing steps. This presupposes that starch recovered during the process fiber washing steps is less tightly bound than the starch left with the dewatered fiber. Because abrading the fiber would likely be less critical after intensive milling, the number of washing steps may be reduced in a modified process. A second concern is that the results of this study were achieved with a laboratory-scale 10.2-cm diameter plate mill operated at 39 rpm, and milling intensity was studied by repeated grinding passes. In comparison, typical plant third-grind mills have plate diameters of 91–127 cm and operate at higher velocities. Hence, local plate velocities and contact times are different between the two types of mills. Further work may be needed to test for differences that result from the operation of laboratory- and production-scale mills.

Alternative product distributions may be possible because of the additional milling. The simplest option is to recover the mill starch and combine the remaining fractions with the steeping solubles, etc., to form a feed product similar to corn gluten feed. Although some additional protein is recovered from the fiber by the intensive milling, the final protein concentration of this feed would be 2–3% higher because of the greater reduction in starch mass. The digestible energy of this product, however, would be lower. Assuming the recovered starch and protein is worth three times the price of the initial fiber, this option increases the value of the fiber fraction of the kernel by 36% ($0.18 \times 3 + 0.82 \times 1$). A second option is to separate the fine fiber. This fiber fraction has a protein content of $\approx 25\%$ double that of the untreated fiber. Combining the fine fiber with the steeping solubles ($\approx 46\%$ protein) gives a $\approx 40\%$ protein meal and a coarse fiber residue with 7.5% protein. If the 40% protein meal can be marketed for double the price of corn gluten feed and the coarse fiber sold for half, then an 17% increase in fiber value is obtained ($0.18 \times 3 + 0.15 \times 2 + 0.66 \times 0.5$) and the value of the solubles is doubled. The increased value of the fiber plus solubles fraction is 46%. While this appears more favorable, it assumes that the fine fiber and solubles can be combined without an added binder that would dilute the protein concentration.

TABLE V
Residual Fiber Starch (% db) from Chemically Treated and Milled Corn Fiber^a

Fraction	Chemical Pretreatment (50°C, 2 hr) ^b			
	Untreated	LA	SO ₂	LA + SO ₂
Coarse fiber	2.5a (0.4)	2.5a (0.4)	2.2a (0.9)	2.4a (0.5)
Fine fiber	9.8a (1.6)	14.3b (1.2)	13.1b (1.9)	12.3a (0.8)
Combined average	3.7a (0.1)	4.6a (0.5)	4.0a (0.6)	4.0a (0.6)

^a All samples were milled twice. Standard deviations are given in parentheses. Letters represent analysis of variance for all possible mean comparisons. Variances have been arcsin-transformed to improve the implicit assumption of equal variances. Different letters in each row indicate a significant difference at $\alpha = 0.05$.

^b LA = 0.5% lactic acid; SO₂ = 0.4% sulfurous acid.

TABLE VI
Residual Fiber Protein (% db) from Chemically Treated and Milled Corn Fiber^a

Fraction	Chemical Pretreatment (50°C, 2 hr) ^b			
	Untreated	LA	SO ₂	LA + SO ₂
Coarse fiber	7.0a (0.2)	6.8a (0.4)	6.5a (0.2)	6.4a (0.2)
Fine fiber	23.5a (0.3)	23.1a (0.3)	21.7b (0.2)	22.0b (0.2)
Combined average	9.8a (0.3)	9.7a (0.4)	9.1b (0.2)	8.9b (0.2)

^a All samples were milled twice. Standard deviations are given in parentheses. Letters represent analysis of variance for all possible mean comparisons. Variances have been arcsin-transformed to improve the implicit assumption of equal variances. Different letters in each row indicate a significant difference at $\alpha = 0.05$.

^b LA = 0.5% lactic acid; SO₂ = 0.4% sulfurous acid.

The costs associated with recovering additional starch by added milling include the capital and operating costs of an additional mill or, alternatively, the operating costs of driving the third-grind mill harder. The cost difference of these two options is difficult to access. Because the additional mill would grind only the residual fiber, the required capacity of this mill is approximately a fifth of the current third-grind capacity. Hence, the cost of operating this mill will be considerably less than hard milling the entire degermed slurry with full capacity third-grind mills. The lower operating costs may compensate for the added capital cost of this option. Drying costs will be incrementally higher because of the recovered starch and protein as well as the increased fine fiber. In addition, recovery of fine fiber would also entail some modified drying processes, as the isolated material cannot be mechanically dewatered.

CONCLUSIONS

Approximately half of the starch currently lost to the wet-milling fiber fraction was recovered by additional milling. This corresponds to an overall increased starch yield of $\approx 1.3\%$. Adding the starch recovered from the process to the bulk starch will not detrimentally affect the starch quality. Milling also increased the production of fine fiber. This material with a protein content of $\approx 25\%$ can be combined with the protein-rich steep water to form a $\approx 40\%$ feed product with higher value. The remaining coarse fiber contained $< 8\%$ protein and $< 6\%$ starch. Secondary steeping of the fiber with combinations of sulfuric and lactic acids did not materially improve the starch recovery.

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