Wet Milling and Separation of Wheat Distillers' Grains with Solubles into Dietary Fiber and Protein Fractions

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ABSTRACT

Wheat-based stillage from a fuel ethanol plant was wet milled by sonication, abrasive stone milling, and blade grinding before fractionation by sieving and centrifugation into distillers' grains (DG), centrifuged solids (CS), and thin stillage. Blade grinding was the most effective method for separating protein and fiber in stillage, with 40% of the fiber being concentrated in DG and over 70% of protein in CS. The DG, which contained over 70% of total dietary fiber, exhibited high water- and oil-absorption capacities and had potential as a fiber ingredient in foods. The CS fraction contained 60% protein and was lower in functionality than DG. Further studies are required to identify potential uses for the fractions and the economic feasibility of the process.

Fuel ethanol plants produce large volumes of a dilute slurry of grain mash by-products in which soluble and insoluble protein and fiber are the predominant components. This stillage is marketed as distillers' grains with (DGS) or without (DG) solubles, often after drying. The fiber serves a useful role in cattle feeds with respect to bypass protein but reduces protein use by monogastric animals. Procedures are required for separation of dietary fiber from protein in DG in order to enhance the market potential for stillage components. The current demand for dietary fiber in human nutrition may provide the economic base for the stillage fractionation processes, including the drying of products and effluent disposal.

Corn is the principal grain used in the mash bills of fuel ethanol plants, but DG and DGS contained high levels of crude fat in a study by Wu et al (1981). The polyunsaturated nature of corn DG lipids indicated that storage stability would be a serious problem in the food applications (Lee et al 1991). Wheat-based stillage products contained only one third of the lipid found in corn DG, and hard wheat DG or DGS had more crude protein when calculated by the same nitrogen-to-protein conversion factor (Wu et al 1984, Rasco et 1987). Sieving and centrifugation of commercial wheat stillages provided a DG fraction containing 47% crude protein and 36% dietary fiber, and centrifuged solids (CS) containing 55% crude protein and 24% dietary fiber (Lee et al 1991). The degree of separation of protein and fiber was insufficient for direct industrial application, but it appeared that further processing of DG might release more protein into the low-fiber CS fraction. Therefore, the objective of the present study was to mechanically disintegrate the insoluble components in fresh wheat-based stillage, followed by sieving and centrifugation to obtain a greater yield of CS fraction but with minimal increase on stillage solubles (SS) or volume. The yields and chemical compositions of the resulting DG, CS, and SS fractions are reported in this article.

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MATERIALS AND METHODS

Fresh stillage was collected from the beer still of a fuel ethanol plant (Mohawk Oil Co., Minnedosa, MB) when Canadian Prairie Spring wheat was the sole grain ingredient in the mash. The crude protein and dietary fiber contents of the grain were 16.2 and 13.5%, respectively (Lee et al 1991). The stillage was sampled for determination of moisture and dry matter before processing or storage at −20°C.

Initial fractionation of the stillage was done by the scheme of Wu et al (1981), in which stillage was first screened on a 210-μm mesh sieve (Spectrum Medical Industries, Los Angeles, CA), and the solid fraction on the sieve was retained as DG. The liquid fraction was centrifuged at 10,000 $\times g$ for 10 min to obtain CS, with the residual supernatant being thin stillage. A second sample of stillage was processed in the same manner, except that the DG on the sieve were washed twice with distilled water, and the washings were added to the liquid fraction before centrifugation. In a commercial operation, thin stillage could be recycled via the washing step, but the effect of this treatment on the materials balance of the process has not been determined. All products of the fractionations were analyzed directly or freeze-dried and stored at −20°C until sampled for further analysis.

Comminution of the stillage by sonication was conducted on a batch basis using a sonifier (Model W-375, Heat Systems-Ultrasonics, Plainview, NY). The probe of the sonifier was inserted to a depth of 2.5 cm in a 500-g sample of stillage (11.7% solids content) contained in a 1-L glass beaker. The sonifier was operated at a frequency of 20 kHz and a power output of 80 W for 1, 3, 5, or 8 min. After treatment, the stillage was sieved and centrifuged as described above, including the two washings of DG.

Abrasive grinding was done on a continuous wet mill (Model 225, Morehouse Industries, Fullerton, CA) equipped with a stone rotor and stator plates (60 grit, 5-cm diameter). With the rotor operating at 3,500 rpm, 500-g samples of stillage were passed through the wet mill, and the ground stillage was then fractionated in to DG, CS, and SS as before. In the next treatment, the unwashed DG were reground with 100 ml of fresh distilled water before fractionation. A third regrinding of unwashed DG was done to provide three degrees of grinding on the wet mill.

A Waring Blendor was used to reduce the size of stillage particles by the cutting action of revolving blades operated at 20,000 rpm. For one treatment, 500 g of stillage was ground for 2 min before sieving in a 210-μm sieve, washing twice, and centrifuging to obtain DG, CS, and SS. In the next treatment, the DG, after sieving but before washings, were returned for a second 2-min grinding in 100 ml of fresh distilled water before fractionation. Similarly, another sample was processed, so that the DG were subjected to three grindings before final fractionation. In a separate experiment, the throughs of the 210-μm sieve for blade-ground CS (three passes) were further screened on 150-, 100-, and 70-μm sieves. After sieving, the solid fractions were washed twice with 50 ml of distilled water each. The combined liquid fractions were centrifuged at 10,000 $\times$ g for 19 min to obtain the <70-μm solids and SS.

Proximate constituents in DG, CS, and thin stillage or SS were determined by the approved methods of the AACC (1983), using the nitrogen (N)-to-protein conversion factor of 5.7. Starch was measured as glucose on an industrial analyzer (Model 27, Yellow Springs [Ohio] Instrument Co.) after hydrolysis with α-amylase and amyloglucosidase (Budke 1984). Total dietary fiber analysis was conducted by the Prosky et al (1985) procedure, except that protein correction was also based on N $\times$ 5.7 rather than 6.25. Water-oil absorption capacities were determined by the centrifuge procedures described by Sosulski (1962) and Lin

TABLE I
Effect of Sieving, Washing, and Sonication of Wheat Stillage on Yield and Composition of Distillers' Grains (DG), Centrifuged Solids (CS), and Stillage Solubles (SS) (%, db)[a]

Treatment of Stillage	Proportional Yield				Dietary Fiber Concentration			Crude Protein Concentration		
	DG	CS	SS	Total	DG	CS	SS	DG	CS	SS
Cheesecloth[b]	74.0 a	5.0 e	20.9 b	99.9	36.3 e	24.1 a	16.3 a	45.2 a	51.4 b	22.3 a
Sieved	56.4 b	22.2 d	20.5 b	99.1	37.3 e	22.6 ab	16.3 a	41.1 b	58.1 a	22.4 a
Sieved and washed	42.7 c	27.4 c	27.4 a	97.5	42.1 d	21.2 bc	15.6 a	41.3 b	58.5 a	23.1 a
Sonicated										
1 min	39.9 cd	29.1 bc	27.4 a	96.4	43.7 cd	21.5 bc	16.9 a	40.4 bc	57.6 a	23.2 a
3 min	37.1 de	30.9 b	27.6 a	95.6	46.1 bc	20.8 c	16.5 a	39.5 bcd	58.1 a	22.8 a
5 min	35.0 ef	34.2 a	26.8 a	96.0	48.8 a	20.0 c	16.5 a	37.6 cd	58.5 a	23.0 a
8 min	33.3 f	35.3 a	27.6 a	96.2	48.1 ab	20.1 c	16.9 a	36.7 d	58.5 a	23.5 a

[a]Means with the same letters are not significantly different at $P < 0.05$; n = 3.
[b]Data from Lee et al 1991.

TABLE II
Effect of Grinding Treatments on Proportional Yields of Components in Stillage on an As-Is Basis (% of Original Stillage)[a]

Grinding Treatment	Distillers' Grains	Centrifuged Solids	Thin Stillage	Total
Sieve and wash	25.0 a	11.4 e	94.3 d	130.7
Stone mill				
1X[b]	25.5 a	14.6 d	97.0 d	137.1
2X	22.4 b	17.5 c	127.8 c	167.7
3X	20.0 bc	18.6 c	153.8 b	192.4
Blade grinder				
1X	17.6 cd	17.4 c	96.2 d	131.2
2X	15.5 d	21.5 a	131.8 c	168.8
3X	13.5 d	23.3 a	173.2 a	210.0

[a]Means with the same letters are not significantly different at $P < 0.05$, n = 3.
[b]X = number of grindings.

TABLE III
Effect of Grinding Treatments on Proportional Yields of Components in Stillage on a Dry-Weight Basis (%)[a]

Grinding Treatment	Distillers' Grains	Centrifuged Solids	Stillage Solubles	Total
Sieve and wash	42.7 a	27.4 d	27.4 b	97.5
Stone mill				
1X[b]	36.8 b	33.3 c	27.4 b	97.5
2X	28.2 c	39.3 b	30.8 a	98.3
3X	23.9 d	42.7 b	31.6 a	98.2
Blade grinder				
1X	28.7 c	40.6 b	29.6 ab	98.9
2X	22.0 d	46.7 a	29.7 ab	98.4
3X	18.2 e	49.6 a	30.9 a	98.7

[a]Means with the same letters are not significantly different at $P < 0.05$, n = 3.
[b]X = number of grindings.

TABLE IV
Effect of Grinding Treatments on the Chemical Composition and Recovery of Dietary Fiber and Crude Protein in Distillers' Grains (DG), Centrifuged Solids (CS), and Stillage Solubles (SS) (%, db)[a,b]

Grinding Treatment	Dietary Fiber Concentration			Crude Protein Concentration			Dietary Fiber Recovery			Crude Protein Recovery		
	DG	CS	SS	DG	CS	SS	DG	CS	SS	DG	CS	SS
Sieved and washed	42.1 f	21.2 ab	15.6 a	41.3 a	58.5 a	23.1 a	56.8 a	18.3 e	13.5 b	42.1 a	38.3 f	15.1 b
Stone mill												
1X[c]	47.0 e	19.9 b	16.1 a	38.3 b	57.5 a	23.2 a	54.6 a	20.9 d	13.9 b	33.6 b	45.7 e	15.2 b
2X	53.7 d	20.4 b	15.9 a	34.0 c	58.9 a	23.3 a	47.8 b	25.3 c	15.4 a	22.9 c	55.2 d	17.1 a
3X	59.4 c	20.8 ab	15.5 a	30.1 d	59.5 a	23.5 a	44.8 c	28.0 b	15.5 a	17.2 d	60.6 c	17.7 a
Blade grinder												
1X	53.2 d	22.1 a	16.0 a	33.8 c	60.3 a	22.8 a	48.2 b	28.3 b	14.9 a	23.2 c	58.4 c	16.1 ab
2X	64.9 b	20.0 b	16.7 a	25.4 e	59.3 a	22.9 a	45.0 c	29.5 b	15.6 a	13.3 e	66.1 b	16.2 ab
3X	71.7 a	20.6 ab	15.9 a	19.9 f	59.2 a	23.3 a	41.2 d	32.2 a	15.5 a	8.6 f	70.1 a	17.2 a

[a]The original distillers' grains with solubles contained 31.7% dietary fiber and 41.9% crude protein (Lee et al 1991).
[b]Means with the same letters are not significantly different at $P < 0.05$, n = 3.
[c]X = number of grindings.

et al (1974). The data on fraction yield and chemical composition were analyzed statistically using the Statistical Analysis System (1985).

RESULTS AND DISCUSSION

Filtration and Screening

Lee et al (1991) found that the fresh stillage sample from the fuel ethanol plant that was mashing CPS wheat contained 11.7% DGS. After filtration of the stillage on cheesecloth, and centrifugation, the DGS yielded only 5.0% CS, as compared to 74.0% DG, dry basis (Table I). Fractionation of the same stillage on the 210-μm sieve increased the CS yield to 22.2% while increasing CS protein concentration from 51.4 to 58.1%. Washing the DG transferred about one quarter of the DG into CS and SS in particular, and enriched the DG fiber level to 42.1%.

Sonication

When the duration of sonication was increased progressively from 1 to 8 min, the proportion of DG decreased, with essentially all of the displaced solids appearing in CS (Table I). Dietary fiber in DG increased to 48.8% after sonication for 5 min, at which stage the CS yield was 34.2%, and the CS protein content was 58.5%. In general, irrespective of time, sonication did not increase the yield of SS or losses of protein or dietary fiber into SS, compared with the sieving and washing procedure.

Grinding

A materials balance of the sieved and ground products of the component extraction is given in Tables II and III. On an as-is basis, sieving and washing yielded 25.0 and 11.4% by weight of DG and CS, respectively, but thin stillage became 94.3% of the original stillage weight because of the two washings. The grinding treatments were conducted one (1X), two (2X), or three (3X) times, and each additional grind added incrementally to the volume of thin stillage. Moisture contents of the DG were 80–86% and in CS were 72–75%, with the solids load in thin stillage being only 2–3.5%.

Three passes of DG through the stone mill progressively reduced DG yield and increased the proportion of CS and SS (Tables II and III). On a dry basis the CS yield was 42.7% of total dry matter but, unfortunately, the SS increased from 27.4 to 31.6%. The blade-grinder treatments, which provided 2, 4, or 6 min of continuous cutting action, decreased DG yield to less than half the control weight, and CS yield increased to 49.6% of the dry matter.

In terms of chemical composition, dietary fiber in DG increased from 42.1 to 59.4% as a result of stone milling (3X), but blade grindings were more effective and brought the fiber concentration in DG to 71.7% (Table IV). Of course, recovery of dietary fiber in the DG fraction decreased from 56.8% for the control sieved sample to 44.8% for stone milling or 41.2% for blade grinding.

With respect to crude protein in the CS fractions, the increases in concentration due to processing the DG were quite small, but recovery of protein increased markedly (Table IV). Stone milling (3X) raised the protein recovery in CS from 38.2 to 60.6%, and the 6 min of blade grinding recovered 70.1% in this fraction. Losses of crude protein and dietary fiber into the SS fraction increased only about 2 percentage points over that of the control.

To determine whether a greater recovery of fiber in DG or protein in CS could be achieved by sieving DG at a finer mesh, the throughs from the 210-μm sieve were sieved on 150-, 100-, and 70-μm sieves (Table V). It was observed that dietary fiber percentage decreased and protein concentration increased with reduction in particle size. Relative to CS, the 150-μm sieve retained a small fraction, 3.6% of DGS, which was higher in dietary fiber and lower in protein. But incorporation of this fraction into DG would have lowered the DG fiber content significantly and increased the percentage of DG protein. Thus, there appeared to be no advantage in using a finer sieve than 210 μm or more than a single sieve size for the fractionation of DGS.

The dietary fiber level of 71.7% in blade-ground DG was significantly higher than that in other DG samples and commercial wheat bran (Table VI). In addition, the processed DG samples had much less ash than wheat bran and sieved (unwashed) DG. Also, lipid levels remained below 4.0%.

No significant differences were noted in protein content among the various processed CS samples, which averaged 59.1%, and dietary fiber levels were uniformly low at 20.5% (Table VI). A significant portion of ash and lipid in DGS segregated into the CS fraction, which averaged 3.5 and 5.6%, respectively.

The water and oil absorptions of the control (unwashed) DG were similar to the values obtained for commercial wheat bran (Table VI). On the other hand, the absorption values for the blade-ground DG were, respectively, about 40 and 60% higher than those of the controls. Water absorptions in the CS samples were lower than those in DG, and oil absorptions decreased in proportion to the degree of processing, being particularly low in blade-ground CS. This indicated that the fiber-rich fraction

TABLE V
Fractionation of Throughs of 210-μm Sieve for Blade-Ground Centrifuged Solids (Three Passes) on Progressively Finer Sieves (%, db)[a]

Fraction	Recovery of Centrifuged Solids	Dietary Fiber	Crude Protein	Fiber Recovery	Protein Recovery
Centrifuged solids	49.6	20.6	59.2	32.2	70.1
150-μm sieve	3.6 d	26.4 a	57.8 c	3.0 c	5.0 d
100-μm sieve	6.2 c	15.6 c	65.4 b	3.1 c	9.7 c
70-μm sieve	12.9 b	14.4 c	67.8 a	5.9 b	20.9 b
<70μm sieve	24.7 a	24.7 b	59.3 c	19.2 a	35.0 a
Total	47.4	...	...	31.2	70.6

[a]Means with the same letters are not significantly different at $P < 0.05$, n = 3.

TABLE VI
Chemical Composition and Functional Properties of Fiber-Rich and Protein-Rich Products (db)[a]

Grinding Treatment[b]	Starch (%)	Dietary Fiber (%)	Crude Protein (%)	Ash (%)	Lipid (%)	Total (%)	Water Absorption (g of H_2O/ 100 g of sample)	Oil Absorption (g of oil/ 100 g of sample)
Fiber-rich fractions								
Sieved DG	1.6 b	37.3 e	41.1 a	3.7 b	3.5 a	87.2	410 d	296 c
Sonicated DG 5 min	0.9 c	48.8 d	37.6 b	2.1 c	3.5 a	92.9	448 c	286 c
Stone Milled DG 3X	0.7 c	59.4 b	30.1 c	1.9 c	3.7 a	95.8	553 b	426 b
Blade Ground DG 3X	0.0 d	71.7 a	19.9 d	1.4 d	3.3 a	96.3	575 a	470 a
Commercial Wheat Bran	10.4 a	53.4 c	18.2 e	6.6 a	4.0 a	92.6	424 d	294 c
Protein-rich fractions								
Sieved CS	2.3 a	22.6 a	58.1 a	3.5 a	4.7 c	91.2	350 a	217 a
Sonicated CS 5 min	2.6 a	20.0 b	58.5 a	3.9 a	4.6 c	89.6	339 a	202 b
Stone Milled CS 3X	2.8 a	20.8 b	59.5 a	3.4 a	6.3 a	92.8	338 a	126 c
Blade Ground CS 3X	2.1 a	20.6 b	59.2 a	3.3 a	5.8 b	91.0	336 a	111 d

[a]Within fractions, means with the same letters are not significantly different at $P < 0.05$, n = 3.
[b]DG = distillers' grains, CS = centrifuged solids, X = number of grindings.

was more functional than the protein-rich fraction and that the functionality of the former was improved significantly by the blade-grinding process in particular.

CONCLUSIONS

These experiments demonstrate that wet milling of stillage is effective in the separation of protein from fiber, especially with the blade grinder. About 40% of the total dietary fiber in stillage was recovered on a 210-μm sieve, and this fraction exhibited enhanced water- and oil-absorption properties. Centrifugation of the remaining slurry recovered 70% of the stillage protein in the solid fraction, but this fraction had reduced functionality. The fractionation system based on blade grinding did not increase the proportions of soluble protein or fiber in the thin stillage so that effluent disposal costs would not be increased markedly if the thin stillage were recycled through the grinding step. Further studies are needed to determine the potential value of the separated components in human nutrition, prepared foods, and animal feed and the economic feasibility of the process.

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LITERATURE CITED

AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1983. Approved Methods of the AACC. Methods 08-03 and 30-25, approved April 1961, revised October 1981; 44-15A, approved October 1975, revised October 1981; 46-13, approved October 1976, revised October 1986. The Association, St. Paul, MN.

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

LEE, W. J., SOSULSKI, F. W., and SOKHANSANJ, S. 1991. Yield and composition of soluble and insoluble fractions from corn and wheat stillages. Cereal Chem. 68:559.

LIN, M. J. Y., HUMBERT, E. S., and SOSULSKI, F. W. 1974. Certain functional properties of sunflower meal products. J. Food Sci. 39:368.

PROSKY, L., ASP, N. G., FURDA, I., DEVRIES, J. W., SCHWEIZER, T. F., and HARLAND, B. A. 1985. Determination of total dietary fiber in foods and food products. J. Assoc. Off. Anal. Chem. 68:677.

RASCO, B. A., DONG, F. M., HASHISAKA, A. E., GAZZAZ, S. S., DOWNEY, S. E., and SAN BUENAVENTURA, M. L. 1987. Chemical composition of distillers' dried grains with solubles (DDGS) from soft white wheat, hard wheat and corn. J. Food Sci. 52:236.

SAS INSTITUTE 1985. SAS User's Guide: Statistics, Version 5 edition. The Institute: Cary, NC.

SOSULSKI, F. W. 1962. The centrifuge method for determining flour absorption in hard red spring wheats. Cereal Chem. 39:344.

WU, Y. V., SEXSON, K. R., and WALL, J. S. 1981. Protein-rich residue from corn alcohol distillation: Fractionation and characterization. Cereal Chem. 58:343.

WU, Y. V., SEXSON, K. R., and LAGODA, A. A. 1984. Protein-rich residue from wheat alcohol distillation: Fractionation and characterization. Cereal Chem. 61:423.

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