

Quick Fiber Process: Effect of Mash Temperature, Dry Solids, and Residual Germ on Fiber Yield and Purity

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ABSTRACT

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Preliminary calculations showed that recovery of fiber before fermentation in the dry grind ethanol facilities known as the Quick Fiber process increases fermenter capacity and reduces ethanol production cost by as much as 4 ¢/gal. The objective of the current research was to evaluate the effect of mash temperature, dry solids, and residual germ on fiber yield and purity when using the quick fiber process. Fiber was recovered by flotation and skimming, while maintaining a specified temperature, dry solids, and residual germ in the mash. Varying temperature and dry solids

in the mash resulted in a statistically significant effect on the fiber yield, neutral detergent fiber (NDF) content, and weight of NDF/100 g of dry corn. Varying residual germ in the mash resulted in statistically significant differences for NDF through dilution and the weight of NDF/100 g of dry corn. The highest fiber yield was 10.9% at 45°C, 23% dry solids, and 15% residual germ; the highest NDF was 50.9% at 30°C, 21% dry solids, and 0% residual germ. The highest weight of NDF/100 g of dry corn was observed at 45°C, 23% dry solids, and 0% residual germ.

The demand for ethanol is expanding due to environmental concerns and the Clean Air Act (CAA) Amendments of 1990, which requires the use of oxygenated fuel to reduce carbon monoxide pollution. The addition of oxygen to the fuel promotes efficient combustion and reduces tailpipe carbon monoxide emissions. Investigations (Anonymous 1995) have shown that a 10% ethanol blend in fuel can reduce the automobile carbon monoxide emissions as much as 25%. With the high cost of crude oil in the past nine years, more attention is being directed on using ethanol in alternative fuels. Besides being the clean alternative to MTBE in the gasoline market, use of ethanol saves the consumers from 2 to 5¢/gal of gasoline purchased (Renewable Fuels Association [RFA], March 9, 2000, *press release*). Technology improvements since 1970 have led to a 20% decrease in the amount of energy required for ethanol production (Anonymous 1995). New technology focusing on the recovery of new or different coproducts (Singh and Eckhoff 1996) has the potential to reduce ethanol production costs even more.

There are two corn-to-ethanol processes currently used: wet milling (Fig. 1) and dry grind (Fig. 2). The dry grind process has lower capital investment and produces more ethanol (2.6 gal/bu) when compared to the wet milling process (2.5 gal/bu). This lower ethanol production in the wet milling process is due to starch loss in the fiber and gluten fractions. Dry grind suffers from low coproduct value because the coproducts (germ and fiber) are not recovered separately.

Singh and Eckhoff (1996) evaluated recovery of germ in a dry grind ethanol facility by means of conventional wet milling degermination (Fig. 3). Germ recovery in a dry grind ethanol facility, known as the Quick Germ process, resulted in a 10.2 ¢/gal of ethanol production cost reduction.

The recovery of pericarp fiber following the quick germ process in dry grind ethanol facilities further enhances profitability of ethanol production by an additional 4 ¢/gal. The quick fiber process has several advantages: 1) it increases fermenter capacity by 3–4%; 2) concentration of the desired protein in DDGS is increased as fiber is decreased and, thus, the potential for including DDGS in nonruminant livestock is enhanced; 3) high levels of cholesterol-lowering phytosterol components, ferulate phytosterol esters (FPE), free

phytosterol, and phytosterol fatty acyl esters can be extracted from pericarp fiber; 4) adding fiber during the dewatering of gluten slurries increases the flux ≈200% (K. Rausch, *unpublished*).

A recent study (Singh et al 1999) on corn fiber oil investigated several advantages of corn fiber oil compared with other cholesterol-lowering edible oil supplements. It is the only product that contains three different classes of natural cholesterol-lowering compounds (FPE, free phytosterol, and fatty acyl phytosterol esters). Corn fiber oil contains high amount of stanol esters that are more effective in lowering cholesterol than other unsaturated phytosterol. These

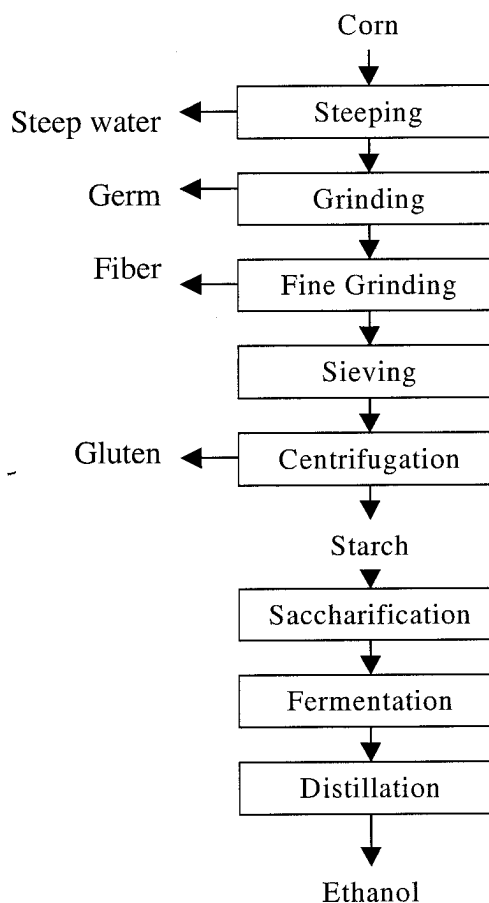


Fig. 1. Wet milling process for ethanol production (Singh and Eckhoff 1996).

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cholesterol-lowering components can be used as nutraceuticals and command a high value in the market (Hicks 1998).

Fiber recovery following the quick germ process was evaluated to optimize the fiber yield in dry grind facilities. Fiber recovery in this study was based on the density difference, which is a function of both temperature and dry solid content. Residual germ corresponded to the solid concentration in the slurry, which affected hydrocyclone performance for scale-up investigation (Bradley 1965). The objective of this study was to determine the effect of mash temperature, mash dry solids, and residual germ in the mash on fiber yield, neutral detergent fiber (NDF) purity in the collected fiber, and weight of NDF/100 g of dry corn as a step toward optimization of the quick fiber process conditions.

MATERIALS AND METHODS

Yellow dent corn of a single hybrid (Pioneer 3394) was stored in a 190 L (55 gal) sealed steel drum at ambient conditions from October 1998 to December 1998 through June 1999. Corn was mixed in the sealed steel drum before sampling, and random samples of ≈1.1 kg were removed and sieved over a 4.8-mm (12/64") round hole screen. After foreign material was removed by hand-picking, 1 kg was weighed, held in a plastic bag, and equilibrated to room temperature before soaking. Three samples of corn ≈30 g each were taken for moisture content determination (AACC 2000).

Preliminary Tests

Ten consecutive tests were conducted by three different operators to determine the most reliable and reproducible blender speed and fiber skimming test procedure. A maximum standard deviation of 0.74 for a mean of 10.8% fiber yield was observed. The difficulty in maintaining constant fiber yield was due to the uncertainty in determining when the fiber skimming was completed. Methods developed represented the best practices for fiber skimming.

TABLE I
Specific Gravity at 30, 45, and 60°C Adjusted to Mash Dry Solids

Mash Dry Solids, %	Specific Gravity (°Be)		
	30°C	45°C	60°C
21	11.5	11	10.5
22	12.5	12	11.5
23	13.5	13	12.5

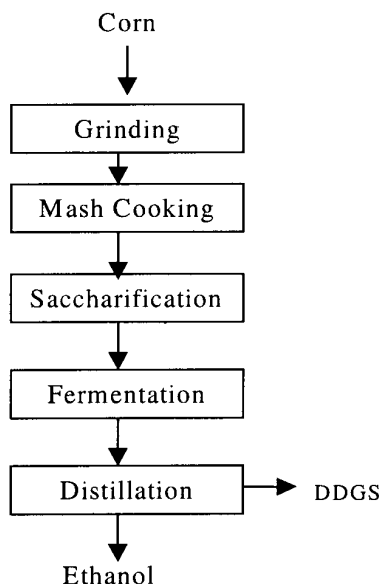


Fig. 2. Dry grind process for ethanol production (Singh and Eckhoff 1996).

Effect of Mash Temperature and Dry Solids in the Mash on Fiber Recovery

Factorial experiments (3 × 3) were performed in triplicate to investigate the effect of temperature and mash (corn slurry after grinding soaked corn with water) dry solids on the floating of fiber. Three levels of mash temperature (30, 45, and 60°C) and three percentages of mash dry solids (21, 22, and 23%) were tested. Dry solids were determined by adjusting the specific gravity of the mash before fiber skimming to 11, 12, or 13 °Be at 45°C as the reference temperature. The mash was dried, and dry matter content was determined by using two-stage oven method (AACC 2000). Because the specific gravity is a function of dry solids and temperature, the specific gravity at 30 and 60°C were adjusted to meet with the corresponding dry solids at 45°C (Table I).

Fiber yield, NDF in the collected fiber, and weight of NDF/100 g of corn (dry basis) were compared statistically across levels of temperature and dry solids in the mash. The weight of NDF/100 g of dry corn was calculated as (fiber yield × NDF) × 100. Corn sample (1 kg) was soaked in 2 L of filtered water at 59°C for 12 hr as outlined by Singh and Eckhoff (1996). Using a variable pump, soak water was pumped into a 2,000-mL graduated cylinder and the volume was recorded. Using the two-stage oven method, three subsamples of soakwater were dried for dry solid content. The

TABLE II
F-Values for Fiber Yield, Neutral Detergent Fiber (NDF), and Weight of NDF/100 g of Dry Corn as a Function of Temperature, % Dry Solids, and Residual Germ for 12-hr Soaked Corn^a

Source	Fiber Yield	NDF Content	Weight of NDF/100 g of Dry Corn
Temperature	0.0001	0.0011	0.0002
Dry solids	0.0001	0.0101	0.0008
Residual germ	0.1885	0.0001	0.0001
Replication	0.086	0.9743	0.2129
Temp. × dry solids	0.0003	0.0001	0.0001
Temp. × residual germ	0.4425	0.6372	0.6567
Temp. × dry solids × residual germ	0.4866	0.0001	0.0532

^a Statistically significant effect ($F \leq 0.05$).

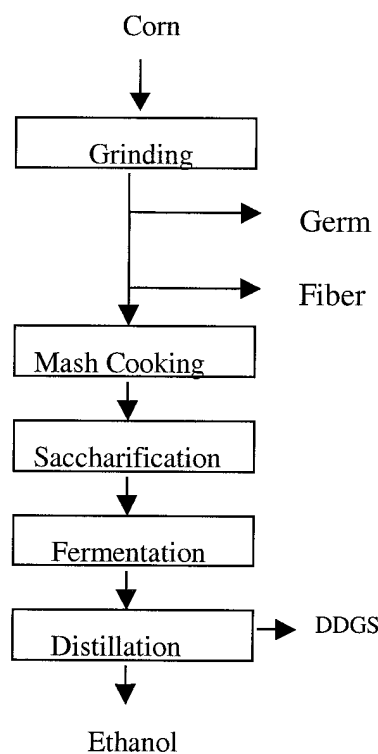


Fig. 3. Dry grind process with quick germ and quick fiber.

soaked corn was ground using a Waring blender at ,500 rpm for 8 min followed by 4,000 rpm for an additional 2 min.

Blender speed was monitored using a programmable tachometer (DART Control, Inc). The rpm was selected based on preliminary tests performed by three operators (10 replicates each). Optimum speed setting for fiber yield was determined based on maximum germ recovery (no whole kernels) and best germ quality (minimum broken germ) or all germ was released and recovered during germ skimming. Fiber yield variabilities were compared among the three operators and found to be similar.

Initial specific gravity of the mash after grinding was measured using a hydrometer, and dry starch was added to adjust mash percent dry solids until good germ flotation was observed. The weight of starch added and the final mash percent dry solids were recorded. Germ were recovered using the wet milling laboratory procedure of Eckhoff et al (1993). Germ moisture determination was conducted in the two-stage oven procedure method.

Mash temperature was adjusted by immersing the bucket containing the mash in a water bath (immersion circulator; Polyscience model 70) until the mash reached the desired temperature of 30,

45, or 60°C. Dry starch was added to increase the percentage of dry solids in the mash to 21, 22, or 23%, and the amounts of starch added and the final specific gravities were recorded. Fiber was recovered by skimming using a flotation procedure similar to the germ recovery procedure (Eckhoff et al 1993). Collected fiber was washed with 3.5 L of filtered water using a 200 mesh vibratory screen, placed in a weighing boat, and dried for moisture content determination. NDF was determined commercially (Silliker Laboratory of Minnetonka, MN).

Effect of Residual Germ in the Mash on Fiber Purity

Factorial experiments (3 × 3) were performed in triplicate to determine the effect of 15% residual germ in the mash during fiber recovery. Preliminary tests were performed to determine the speed setting of the Waring blender that would give ≈15% unreleased or residual germ. The 15% residual germ was determined from the calculated germ yield, where the germ yield was 15% less than the original germ yield.

The 0% residual germ procedure was followed, except that the speed setting of the blender was reduced to 3,500 rpm for 6 min

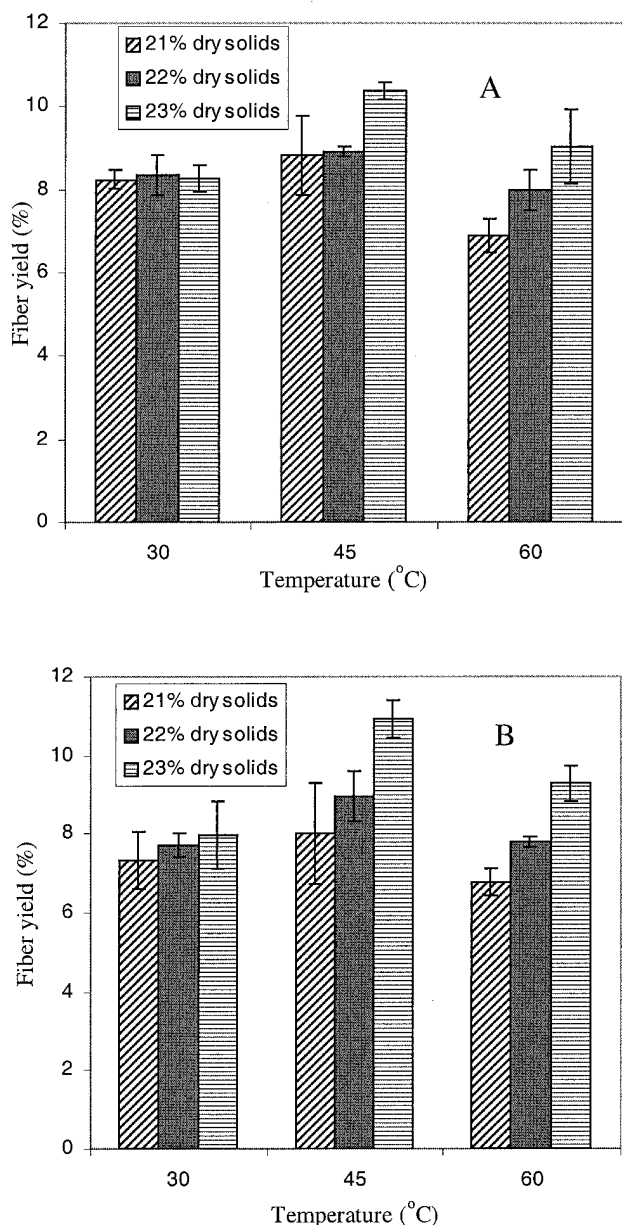


Fig. 4. Fiber yield as a function of mash temperature and dry solids for 12-hr soaked corn: A, 0% residual germ; B, 15% residual germ.

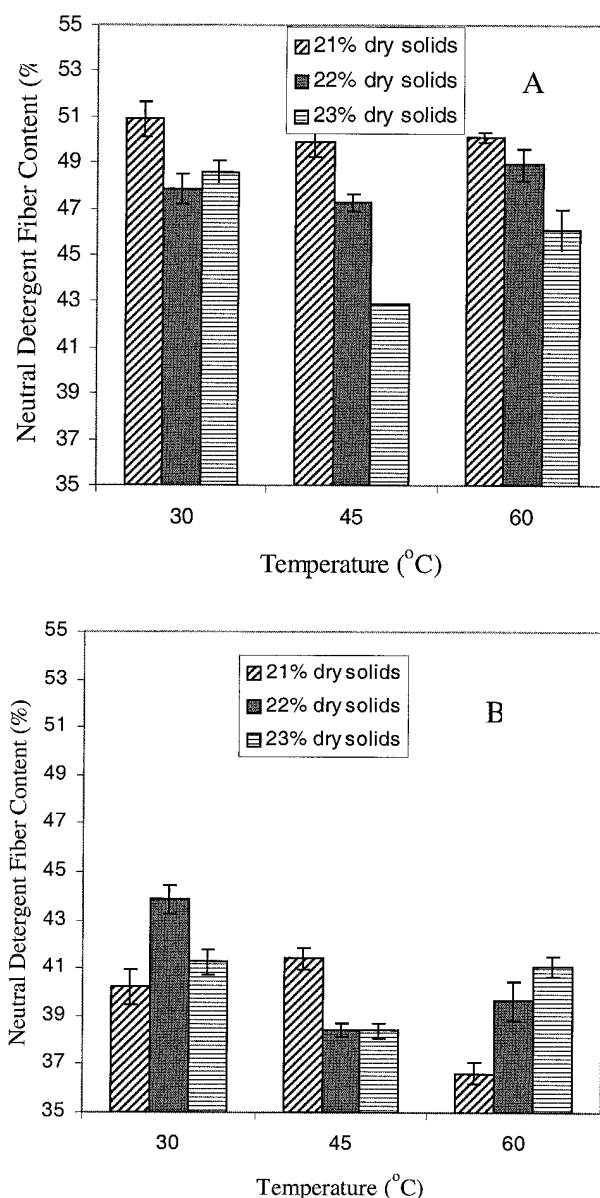


Fig. 5. Neutral detergent fiber (NDF) as a function of mash temperature and dry solids for 12-hr soaked corn: A, 0% residual germ; B, 15% residual germ.

according to the preliminary tests. Fiber yield, NDF, and weight of NDF/100 g of corn (dry basis) were measured, and results were statistically compared.

Statistical Analysis

Analysis of variance (ANOVA) using a general linear model procedure and Duncan's multiple range test were performed (SAS Institute, Cary, NC) at $\alpha = 5\%$ ($P < 0.05$) to determine the effect of temperature, percent dry solids and residual germ in the mash on the fiber yield and purity. An F value of <0.05 was the criteria to determine whether a parameter or interaction had a statistically significant effect on the dependent variable.

RESULTS AND DISCUSSION

Effects on Fiber Yield

Varying temperature and % dry solids in the mash had statistically significant effects on fiber yield, while varying the residual germ in mash did not (Table II). Highest fiber yield (10.9%) was at 45°C, 23% dry solids with 15% residual germ. Lowest fiber yield (6.8%) was at 60°C, 21% dry solids with 15% residual germ.

Figure 4 shows a plot of fiber yield as a function of mash temperature, dry solids, and residual germ. Fiber yield generally increased as the mash dry solids increased at 45 and 60°C (Table III). The increase of mash percent dry solids corresponded to an increase in the slurry density (Table I). The effect of dry solids became greater as temperature was increased. Dry solids had a statistically significant effect on fiber yield (Table II). At 45°C and 0%, 15% residual germ levels, fiber yield at 23% dry solids (highest fiber yield) was significantly different from the 21 and 22% dry solids levels. At 60°C, the fiber yield at 21% (lowest fiber yield) was statistically different from those of other dry solids levels at both 0 and 15% residual germ. However, the effect of dry solids on fiber at 30°C was not statistically significant (Table III)

A temperature of 45°C resulted in the highest fiber yield at all levels of % dry solids in the mash and residual germ (Fig. 4). The fiber yield was higher at 45°C than at 60°C because the % dry solids in the mash raised the baume 0.5 higher than at 60°C with the same dry solid content in the mash. The increase of specific gravity reading due to decrease in temperature from 60 to 45°C (Table I) and with the same dry solids in the mash, enhanced the floating of the fiber. However, the increase of the specific gravity

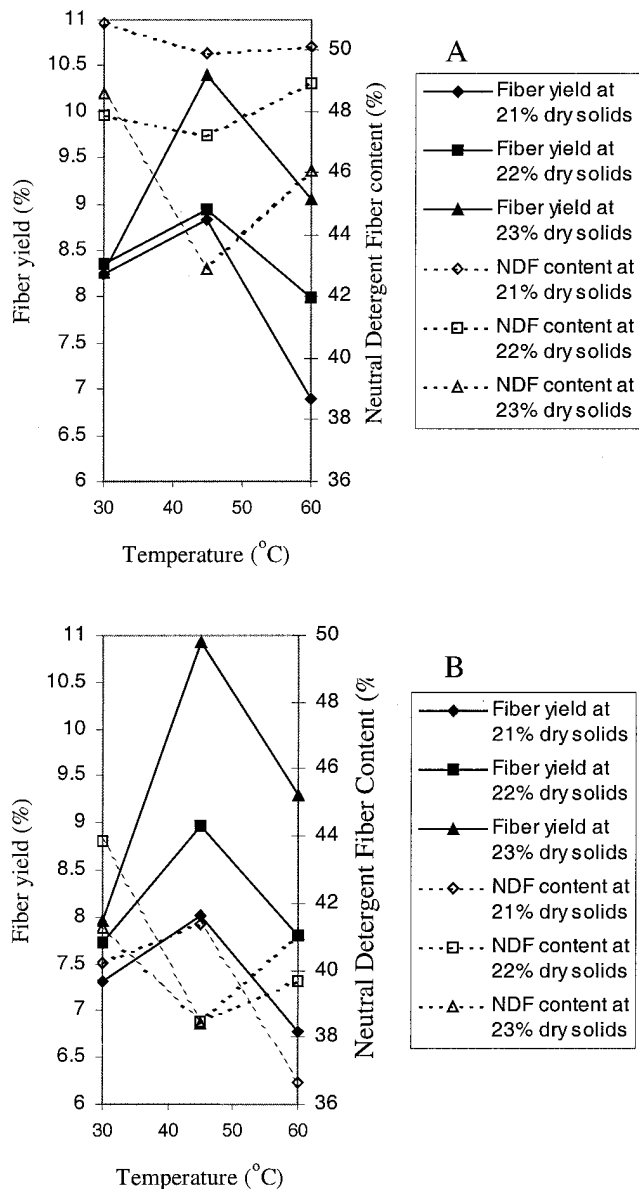


Fig. 6. Fiber yield and neutral detergent fiber (NDF) interaction for 12-hr soaked corn: A, 0% residual germ; B, 15% residual germ.

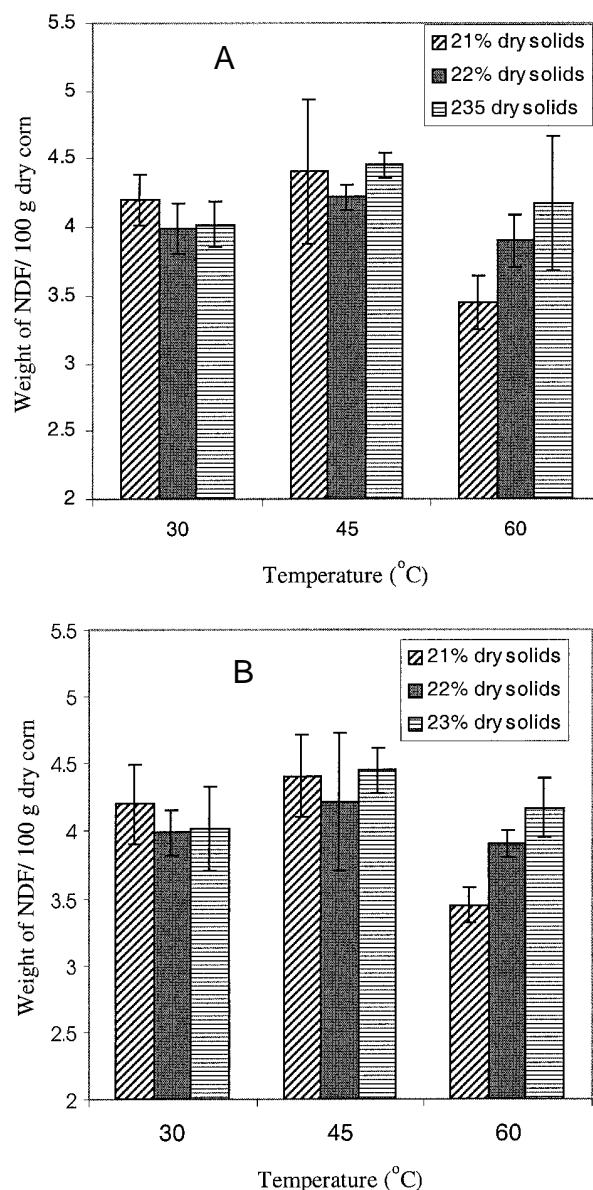


Fig. 7. Weight of neutral detergent fiber (NDF)/100 g of dry corn as a function of mash temperature and dry solids: A, 0% residual germ; B, 15% residual germ.

TABLE III
Fiber Yield (%) as a Function of Temperature, % Dry Solids in Mash, and Residual Germ for 12-hr Soaked Corn^{a,b}

°C	0% Residual Germ			15% Residual Germ		
	21	22	23	21	22	23
30	8.3b-f	8.4c-g	8.3b-f	7.3f-h	7.7e-h	8.0c-g
45	8.8b-e	8.9b-d	10.4a	8.0c-g	9.0b-d	10.9a
60	6.9gh	8.0c-f	9.1bc	6.8h	7.8d-h	9.3b

^a Means of three observations.

^b Values followed by the same letter are not significantly different ($P < 0.05$).

TABLE IV
NDF Content (%) as a Function of Temperature, % Dry Solids in Mash, and Residual Germ for 12-hr Soaked Corn^{a,b}

°C	0% Residual Germ			15% Residual Germ		
	21	22	23	21	22	23
30	50.9a	47.9de	48.6cd	40.2j	43.9g	41.3i
45	49.9b	47.2e	42.9h	41.4i	38.5k	38.4k
60	50.1ab	48.9c	46.1f	36.6l	39.7j	41.1j

^a Mean of three observations.

^b Values followed by the same letter are not significantly different ($P < 0.05$).

TABLE V
Weight of NDF g/100 g of Dry Corn as a Function of Temperature, % Dry Solids in Mash, and Residual Germ for 12-hr Soaked Corn^{a,b}

°C	0% Residual Germ			15% Residual Germ		
	21	22	23	21	22	23
30	4.2a	4.0ab	4.0ab	2.9ef	3.4c-e	3.3de
45	4.4a	4.2a	4.5a	3.7b-d	3.1de	4.2a
60	3.5c-e	3.9a-c	4.2a	2.5f	3.1de	3.8b-d

^a Mean of three observations.

^b Values followed by the same letter are not significantly different ($P < 0.05$).

reading at 30°C did not increase the fiber yield for every level of dry solids content and residual germ (Table III).

The interaction (Table II), temperature × dry solids in the mash, was statistically significant ($P < 0.05$). However, the interactions, temperature × residual germ, and temperature × dry solids in the mash × residual germ, were not statistically significant.

Effects on NDF in Collected Fiber

NDF was used as a measurement of fiber purity because it measures the total concentration of cellulose, hemicellulose, and lignin. Valuable fiber oil can be recovered from these products. Mash temperature, dry solids, and residual germ has statistically significant effects on NDF (Table II). Figure 5 shows the plot of the NDF as a function of mash temperature, dry solids, and residual germ. Highest NDF in the collected fiber (50.9%) was at 30°C, 21% dry solids in the mash, and 0% residual germ; whereas, the lowest (36.6%) was at 60°C, 21% dry solids in the mash, and 15% residual germ. Figure 6 shows the fiber yield and NDF as a function of mash temperature, % dry solids, and residual germ in the mash. NDF in collected fiber was inversely related to fiber yield. NDF decreased as fiber yield increased. The increase in fiber yield corresponded to increases in fiber recovery along with increased impurities, which diluted the NDF in the collected fiber.

The lowest NDF in the collected fiber (42.9%) at 0% residual germ corresponded to the highest fiber yield (10.4%) at 45°C, 23% dry solids, and 0% residual germ. However, the lowest NDF (36.6%) at 15% residual germ did not correspond to the highest fiber yield condition (10.9%) at 45°C, 23% dry solids, and 15% residual germ (Tables III and IV).

Increasing mash residual germ to 15% lowered the NDF in the collected fiber (Fig. 5). Residual germ in the mash corresponded to the unreleased mash germ, which led to whole kernels left with unreleased fiber in the mash. Residual germ was recovered in the fiber fraction instead of the germ fraction, thus more impurities (solids apart from NDF) were collected at 15% residual germ.

Interactions among temperature × dry solids and temperature × residual germ × dry solids in the mash were statistically significant (Table II).

Effects on Weight of NDF/100 g of Corn (dry basis)

Weight of NDF/100 g of dry corn is an important variable because it measures the total weight of NDF recovered from corn, while the fiber yield indicates the % dry matter of the fiber fraction and NDF measures the purity of the collected fiber. Temperature, dry solids, and residual germ had a statistically significant effect on the NDF weight/100 g of dry corn (Table II). Figure 7 illustrates the plot of weight of NDF/100 g of dry corn as a function of mash temperature, dry solids, and residual germ in the mash. A maximum value of 4.5 g of NDF/100 g of dry corn was observed at 45°C, 23% dry solids in the mash, and 0% residual germ; whereas, the lowest value was 2.5 g at 60°C, 21% dry solids in the mash, and 15% residual germ (Table V). The effect of the operating parameters (temperature, dry solids, and residual germ) on the weight of NDF/100 g of dry corn was similar on the fiber yield at 60°C (Figs. 4 and 7). Although fiber yields were inversely related to NDF in the collected fiber (Fig. 6), variations of the fiber yield results had higher magnitudes when compared with those of the % NDF, which resulted in the calculated weight of NDF/100 g of dry corn proportional to fiber yields.

There was a statistically significant interaction of temperature × dry solids. However, the interactions among temperature × residual germ × dry solids were not statistically significant (Table II).

CONCLUSIONS

Varying mash temperature (30, 45, or 60°C), dry solids (21, 22, or 23%) and residual germ in the mash (0 and 15%) resulted in highest fiber dry matter yields at 45°C, 23% dry solids, and 15% residual germ. Fiber yield and % NDF were inversely related. Weight of NDF/100 g of corn (dry basis) was highest at 45°C, 23% dry solids, and 0% residual germ, and lowest at 60°C, 21% dry solids, and 15% residual germ.

Although this study shows operating conditions for highest and lowest fiber recovery, the selection of an optimum condition may depend on other constraints such as recovery bran oil (Moreau et al 1999), which may be affected by fiber purity. Fiber recovery may increase total oil recovery but may decrease ethanol yield. Co-product values such as fiber oil should be a determinant for optimum operating conditions.

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