# Effects of Alkali Debranning, Roller Mill Cracking and Gap Setting, and Alkali Steeping Conditions on Milling Yields from a Dent Corn Hybrid

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## ABSTRACT

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Starch yield was significantly affected by all three main unit operations in alkali wet-milling (debranning, roller milling, and steeping). The conditions for the three unit operations were studied using a single hybrid. Studies on debranning showed that optimal separation between pericarp and corn endosperm was obtained when corn was soaked in a 1.5–2% NaOH solution at 85°C for 5 min. Passing debranned corn through a smooth roller mill once or twice did not affect the product yields, but passing the corn through the roller mill three times decreased the germ yield because of a large amount of broken germ. A 62% higher processing rate could be achieved when passing corn through the mill twice than by passing it through the mill once. The gap should be set at 2.0 mm when passing corn through the mill once, and it should be set at 3.5 mm for the first pass and 2.0 mm for the second pass when passing corn through the mill twice. Starch yield was more sensitive to NaOH concentration and steep temperature than to steep time. The highest starch yield was obtained when steeping corn in 0.5% NaOH for 1 hr at 45°C.

Alkali corn wet-milling has been studied as an alternative to conventional wet-milling (Eckhoff et al 1998). Alkali wet-milling involves removal of the pericarp first to eliminate the diffusion barrier for water and chemicals to reduce the steep time. Cracking the pericarp-free corn in a roller mill is done to further reduce kernel size so that steep time can be reduced. Alkali wet-milling can reduce steep time dramatically; as a result, capital and processing costs can be reduced. A preliminary economic analysis showed that the total investment for building a 2,667 tonne/day (105,000 bu/day) wet-milling plant would be reduced by 23% when using the alkali process instead of the conventional $SO_2$ process (Du 1997).

The alkali wet-milling procedure includes soaking corn kernels in 2% NaOH for 5 min at 85°C, debranning the soaked kernels, cracking the corn kernels in a roller mill, and steeping them in 0.5% NaOH for 1 hr at 45°C with agitation; then germ, fine fiber, and gluten are separated. This procedure gave a practical comparable starch yield of 67.8% at only 1 hr of steeping compared with 69% from the conventional $SO_2$ wet-milling process using a 24-hr steep (Eckhoff et al 1998).

Among all of the steps involved in the alkali wet-milling process, debranning is one of the most critical steps for removing the pericarp. The alkali corn debranning process was developed for the purpose of obtaining bran-free corn that was further processed into edible corn products. Wagner (1940) developed a process to manufacture food products from corn with the removal of pericarp and germ. The process involved steeping corn in 0.25% NaOH at 38°C under 6.8 kg of pressure with agitation to obtain decorticated, decapped, and degerminated corn. A similar process was developed by Hansen (1949) to obtain dehulled and decapped corn by immersing corn in 1% NaOH at 82–100°C for 15 min. Subba Rao et al (1953) developed another process in which corn was boiled in 10% NaOH for 4 min to dehull. However, the high temperature and high alkali concentration caused 35% of the solids to be lost during cooking and washing. Blessin et al (1970) concluded that complete dehulling could be achieved by soaking corn in 15% NaOH for 3–4 min at 71°C. Although the process gave a 93% yield of debranned corn, the temperature and alkali concentration were high enough to damage the germ and endosperm. Mistry and Eckhoff (1992) optimized the process parameters to obtain maximum pericarp yield and concluded that the highest pericarp yield (4.7%) was obtained at 6% NaOH, 9 min, and 57°C. Only one of the above studies investigated the effect of soak conditions on the yield of debranned corn.

The cracking step in alkali corn wet-milling reduces the kernel size, which also reduces the water and chemical diffusion time. A previous study conducted by Watson and Sanders (1961) showed that starch could be released in 2 hr when the pericarp was cut open. Studies conducted by Eckhoff and Tso (1991) and Eckhoff et al (1993) showed that the steep time could be reduced to 4 hr without any significant reduction in starch yield or starch quality when steeping #10 corn grits (2.4–4.0 mm) instead of whole kernels.

Another important process step in alkali wet-milling is agitation steeping because ≈22% of starch can be lost without this step (Eckhoff et al 1998). The effects of soaking conditions on debranning and steep conditions and roller mill settings on wet-milling results have not been studied.

The objectives of this study were to 1) study the effect of soak time, soak temperature, and alkali concentration on corn debranning; 2) study the effect of multiple passes and roller mill gap settings on alkali wet-milling yields; and 3) study the effect of steep temperature, steep time, alkali concentration, and agitation speed on alkali corn wet-milling yields.

## MATERIALS AND METHODS

A single yellow dent corn (hybrid FR1064 × LH59) was harvested during the fall of 1994 and ambient air-dried to ≈15% (wb) moisture content. Dried corn (with test weight of 56 lb/bu) was divided into 100-g subsamples, sealed in plastic bags, and refrigerated until processed. Corn moisture content (15.3%) was measured using the 103°C, 72-hr convection oven method (AACC 1995). The chemical composition of the corn (70.4% starch, 8.2% protein, 2.5% fiber, and 4.5% fat) was analyzed using near-infrared transmittance (GrainSpec, Foss Electric Inc., Brampton, ON, Canada) by the Identity Preserved Grain Laboratory, Champaign, IL. Transmittance readings of 250 g were taken over a wavelength range of 800–1,100 nm.

Sodium hydroxide (NaOH, reagent-grade pellets) was used to prepare a 20% alkali solution by dissolving 80 g of NaOH in 400 mL of water. This solution was diluted with water to the desired concentration for soaking and steeping.

The study was divided into three sets of experiments, each looking at one of the unit operations while the other two unit operations were held constant. This approach provided significant information on each unit operation but it was not possible to study inter-

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actions. A study containing all variables would require too many tests to get the same degree of statistical confidence.

## Alkali Debranning

The independent variables for this experiment were soak time, soak temperature, and alkali concentration. The dependent variables were pericarp and debranned corn yield. The three sets of experiments were conducted with three replicates for each condition. For each set of experiments, 100-g samples of corn were soaked in a 200 mL of NaOH solution. For the first set of experiments, corn was soaked in 2% NaOH for 5 min at 85°C for different periods of time (1, 3, 5, 7, and 9 min). In the second set of experiments, corn was soaked in NaOH with a concentration of 0.5, 1.0, 1.5, 2.0, and 2.5% at 85°C for 5 min. In the third set of experiments, corn was soaked in 1.5% NaOH for 5 min at temperatures of 65, 75, 85, 95, and 105°C. For all of the experiments, the soaked corn was debranned using the method described by Eckhoff et al (1998). The moisture content of the pericarp and debranned corn was then measured using the two-stage convection oven method (AACC 1995).

## Roller Milling

*Effect of multiple passes through a roller mill on alkali corn wet-milling yields.* Corn (100 g) was soaked and debranned using the method described by Eckhoff et al (1998). The debranned corn was cracked by a roller mill once, twice, or three times. The cracked corn was steeped in a 0.5% NaOH solution at 45°C for 1 hr and milled following the finalized procedure described by Eckhoff et al (1998).

The independent variable for this study was the number of times the corn passed through the roller mill after the soaking and debranning steps. Three test conditions with a different number of passes, as shown in Table I, were conducted in duplicate. A single pass through the roller mill with a gap setting of 2 mm was used as a control. Product yields were calculated based on the initial corn dry matter. The oil content in germ was determined by a commercial analytical laboratory using method G-10 (CRA 1984).

*Effect of the roller mill gap settings on alkali corn wet-milling yields.* Corn (100 g) was soaked and debranned following the procedure described by Eckhoff et al (1998) and cracked in a roller mill once or twice with different gap settings, as indicated in Tables II and III. The gap of the roller mill was set at 1.0, 1.5, 2.0, 2.5, 3.0, or 3.5 mm for corn passing through the mill once. When corn passed through the roller mill twice, the gap for the first pass was set at 3.5 mm and the gap for the second pass was set at 1.0, 1.5, 2.0, 2.5, or 3.0 mm. Corn passing through the roller mill once with a gap setting of 2 mm was used as a control. All of the tests were conducted in duplicate and the average yields were reported.

## Alkali Steeping

Corn (100 g) was soaked, debranned, and cracked by passing it once through a roller mill with a gap setting of 2 mm. The effect of steep conditions including steep temperature, steep time, NaOH concentration, and agitation speed during steep on product yields was studied. Five levels of each variable were used, temperature range was 25–65°C, steep time was 15–120 min, NaOH concentration was 0.1–0.9%, and the agitation speed was 100–900 rpm.

**TABLE I**
**Effect of Number of Passes of Alkali-Debranned Corn Through a Smooth Roller Mill on Alkali Wet-Milling Yields (%)[a]**

| Passes | Gap Setting (mm) | Pericarp | Soakwater Solids | Germ Yield | Germ Oil[b] | Fine Fiber | Starch | Gluten | Total |
|---|---|---|---|---|---|---|---|---|---|
| 1 (control) | 2.0 | 3.4 | 1.6 | 6.7a[c] | 36.9a | 5.3c | 68.5a | 14.0a | 99.5 |
| 2 | 3.5, 2.0 | 3.0 | 1.5 | 6.5a | 38.6a | 5.6c | 69.6a | 13.8a | 100.0 |
| 3 | 3.5, 2.0, 1.0 | 3.1 | 1.5 | 3.5b | 37.8a | 6.9a | 68.3a | 13.6a | 96.9 |

[a] Based on initial corn dry matter.
[b] Based on germ dry weight.
[c] $n = 2$. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

**TABLE II**
**Effect of Gap Setting for Passing Alkali-Debranned Corn Once Through a Smooth Roller Mill on Alkali Wet-Milling Yields (%)[a]**

| Gap Setting (mm) | Pericarp | Soakwater Solids | Germ Yield | Germ Oil[b] | Fine Fiber | Starch | Gluten | Total |
|---|---|---|---|---|---|---|---|---|
| 1.0 | 3.2 | 1.4 | 5.9bc[c] | 38.0ab | 5.7cd | 66.6b | 16.3bc | 99.1 |
| 1.5 | 3.1 | 1.3 | 6.7a | 36.0c | 5.1d | 67.1b | 15.7bc | 99.0 |
| 2.0 (control) | 3.3 | 1.6 | 6.7a | 36.9bc | 5.4d | 68.5a | 14.3c | 99.8 |
| 2.5 | 3.0 | 1.6 | 6.4a | 36.9bc | 6.1c | 64.1c | 17.6ab | 98.8 |
| 3.0 | 2.9 | 1.5 | 6.2ab | 37.9ab | 7.7a | 60.7e | 19.3a | 98.3 |
| 3.5 | 3.0 | 1.7 | 5.7c | 39.2a | 7.0b | 61.9d | 16.9ab | 96.2 |

[a] Based on initial corn dry matter.
[b] Based on germ dry weight.
[c] $n = 2$. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

**TABLE III**
**Effect of Smooth Roller Mill Gap Settings for Two Passes on Alkali Corn Wet-Milling Yields (%)[a]**

| Gap Setting (mm) | Pericarp | Soakwater Solids | Germ Yield | Germ Oil[b] | Fine Fiber | Starch | Gluten | Total |
|---|---|---|---|---|---|---|---|---|
| Control (1 pass, 2.0 mm) | 3.3 | 1.6 | 6.7a[c] | 36.9b | 5.4c | 68.5ab | 14.3b | 99.8 |
| 3.5, 1.0 | 3.0 | 1.6 | 2.4b | 38.6ab | 7.2a | 67.8b | 17.3a | 99.3 |
| 3.5, 1.5 | 3.2 | 1.4 | 4.4ab | 39.6a | 6.3b | 68.1ab | 15.0ab | 98.4 |
| 3.5, 2.0 | 3.0 | 1.5 | 6.5a | 38.6ab | 5.7bc | 69.7a | 14.1b | 100.5 |
| 3.5, 2.5 | 3.0 | 1.5 | 6.5a | 38.6ab | 5.7bc | 69.7a | 14.1b | 100.5 |
| 3.5, 3.0 | 2.9 | 1.6 | 6.5a | 34.3c | 6.2b | 67.0b | 15.6ab | 99.8 |

[a] Based on initial corn dry matter.
[b] Based on germ dry weight.
[c] $n = 2$. Values followed by the same letter in the same column are not significantly different ($P < 0.05$).

A modified central composite design was conducted to test the effect of each variable and the interactions, the central point was at 45°C, 0.5% NaOH, and 60 min. Two replicates for each condition were conducted and the average product yields were reported.

Starch samples from all the milling runs were used to test starch properties including distilled water binding capacity, starch gelatinization (differential scanning calorimetry data), pasting properties (Rapid Visco Analyser data), relative gel strength, and freeze-thaw stability. Results are reported separately.

### Statistical Analyses

Statistical analysis was performed using a statistical analysis software package (SAS Institute, Cary, NC). The effects of the soaking conditions on the pericarp and debranned corn yields were tested by analysis of variance (ANOVA) and Duncan's multiple range test. The effects of multiple passes through the roller mill and the gap settings on wet-milling product yields and the oil content in germ, as well as the effect of steeping conditions on wet-milling yields were determined by ANOVA and Duncan's multiple range test using SAS programs. Least significant differences (LSD) of the mean product yields were obtained at a 5% significance level. The effect of steeping conditions on wet-milling yields were determined by ANOVA and response surface methodology using SAS programs.

## RESULTS AND DISCUSSION

### Alkali Debranning

Soak time, soak temperature, and alkali concentration all played significant roles in the soaking and debranning process. The goal for debranning in alkali wet-milling was to obtain an optimum separation between pericarp and endosperm. The debranned corn yield decreased while the pericarp yield increased with an increase in the soak time (Fig. 1). The yield of both debranned corn and pericarp was constant after 5 min of soaking. When increasing the NaOH concentration, the debranned corn yield decreased and the pericarp yield increased and then leveled off when the NaOH concentration was >1.5% (Fig. 2). When the corn kernels were exposed to alkali for a short period of time (<5 min) or to low NaOH concentrations (<1.5%), the action of alkali on the bonds that hold the pericarp to endosperm was not completed and resulted in a low pericarp yield and high debranned corn yield (Figs. 1 and 2).

The debranned corn yield decreased with an increase in soak temperature and the pericarp yield was maximized at 85°C (Fig. 3). When the temperature was <85°C, the pentosan and hemicellulose were less susceptible to alkali attack, leaving most of the pericarp attached to the kernel, resulting in a low pericarp yield and high debranned corn yield. When the temperature was >85°C, the pericarp began to dissolve and gave a low pericarp yield and a low debranned corn yield.

### Roller Milling

*Effect of multiple passes through the roller mill on alkali corn wet-milling yields.* There was no significant effect on the product yields when passing debranned corn once through a roller mill with a gap setting of 2 mm, or twice with a first pass gap setting of 3.5 mm and a second pass gap setting of 2.0 mm (Table I). However, the germ yield decreased by 48% and fine fiber yield increased by 30% when passing debranned corn through the mill three times. This reduction was caused by the small gap (1.0 mm for the third pass) that further broke the germ which then could not be recovered by skimming. The damaged germ ended up in the fiber fraction and gave a high fine fiber yield. Multiple passes through the roller mill did not affect the oil content in germ, but the potential oil recovery based on initial corn dry matter was much lower (1.1%) when passing corn through the roller mill three times than that achieved (2.1%) when passing corn through the roller mill once or twice.

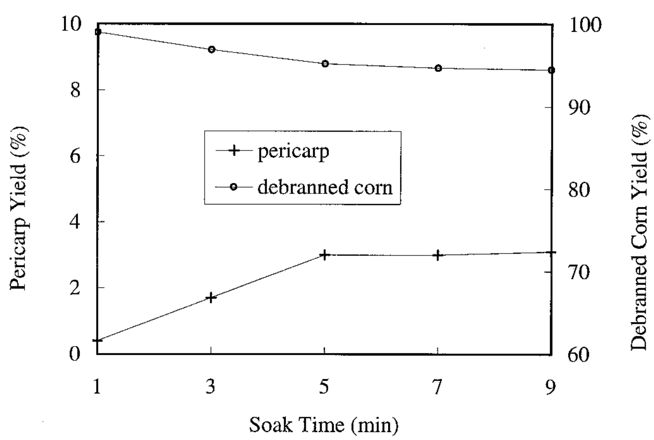


**Fig. 1.** Effect of soak time on pericarp and debranned corn yields. Corn was soaked in 2% NaOH at 85°C.

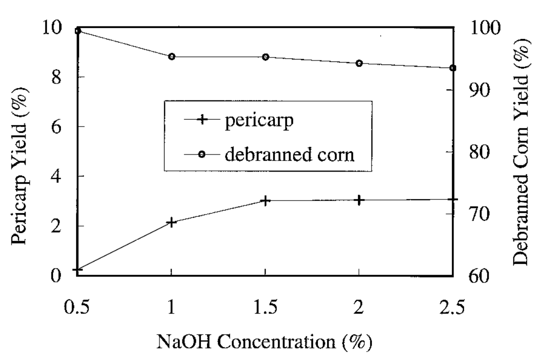


**Fig. 2.** Effect of soak time on pericarp and debranned corn yields. Corn was soaked at 85°C for 5 min.

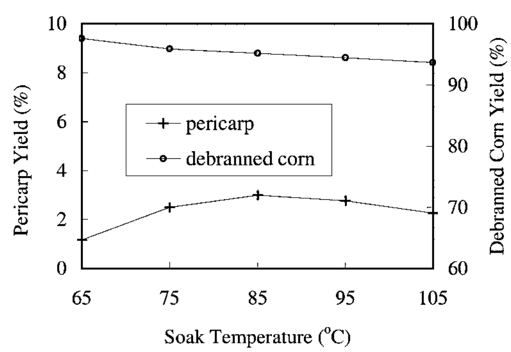


**Fig. 3.** Effect of soak time on pericarp and debranned corn yields. Corn was soaked in 2% NaOH for 5 min.

**TABLE IV**
***F*-Value for Effect of Steep Conditions on Product Yields for Alkali Corn Wet-Milling[a]**

| | Germ | Fiber | Starch | Gluten |
|---|---|---|---|---|
| Concentration (C) | 38.5*** | 88.8*** | 17.7*** | 106.4*** |
| Time (TM) | 31.0*** | 22.5*** | 0.4 | 6.1 |
| Temperature (TP) | 5.0 | 23.0*** | 12.7*** | 35.0*** |
| Speed (S) | 2.3 | 1.6 | 0.0 | 0.6 |
| C × TM | 5.9 | 4.2 | 0.9 | 1.8 |
| C × TP | 7.2 | 1.7 | 9.8*** | 14.5*** |
| TM × TP | 0.01 | 8.1 | 1.3 | 0.1 |
| C × TM × TP | 0.2 | 0.1 | 0.0 | 0.1 |
| Lack of fit | 3.2 | 1.0 | 1.2 | 2.1 |
| $R^2$ | 0.65 | 0.79 | 0.53 | 0.89 |

[a] *** = Significant at $P < 0.05$.

A higher roller mill capacity was achieved with two passes of corn through the roller mill than with one pass. For a 15-cm long smooth roll roller mill, it took 275 sec to pass 100 g of corn through the roller mill once when the gap setting was 2.0 mm, while it took a total of 105 sec to pass 100 g of corn through the roller mill twice when the gap setting was 3.5 mm for the first pass and 2.0 mm for the second pass.

*Effect of the roller mill gap settings on alkali corn wet-milling yields.* The effect of gap setting was conducted for both one and two passes through the mill. When passing corn through the mill once, the highest germ and starch yields and the lowest fiber and gluten yields were obtained when the gap was 2 mm (Table II). The total oil recovery (germ weight × oil content in germ) was highest for the control sample.

For corn passing through the roller mill twice, the gap setting for the first pass was 3.5 mm. When the gap was set at 2.0 or 2.5 mm for the second pass, the highest starch yields were obtained and germ yield was among the highest (Table III). When the gap setting for the second pass was increased to 3.0 mm, starch yield decreased by 3.9% and gluten yield increased by 10.6%. Gap settings at 3.5 and 2.0 or 2.5 mm for the first and second pass, respectively, gave the highest starch and germ yields and the lowest fiber and gluten yields. Gap settings for two passes through the roller mill did not significantly affect the oil content in germ except when the gap was set at 3.5 and 3.0 mm for the first and second pass, respectively.

## Alkali Steeping

Statistical analysis showed that germ yield was significantly affected by the steep time and alkali concentration ($R^2$ = 0.65). Fiber yield was significantly affected by alkali concentration, steep time and steep temperature ($R^2$ = 0.79). Starch and gluten yields were significantly affected by alkali concentration and steep temperature ($R^2$ = 0.53 for starch and $R^2$ = 0.89 for gluten). Agitation speed during steeping did not significantly affect the yield of any product. Starch and gluten yield was also significantly affected by the product of alkali concentration and steep temperature (Table IV). Starch yield was significantly low when

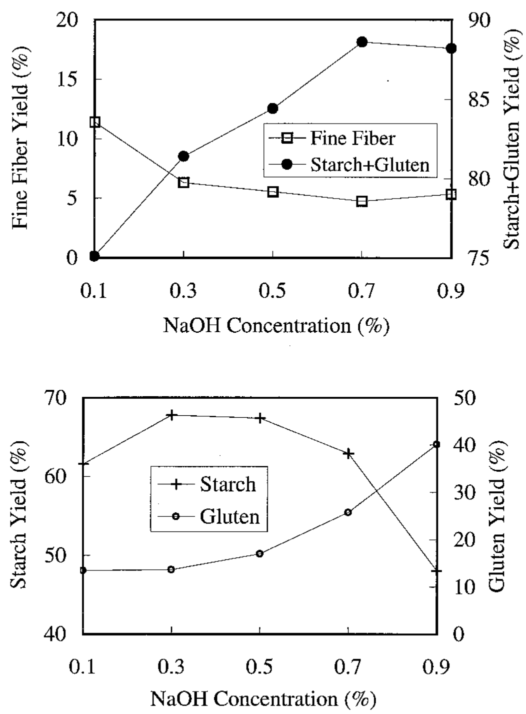


**Fig. 4.** Effect of NaOH concentration on corn starch plus gluten yield, starch yield, gluten yield, and fine fiber yield. Debranned corn was steeped at 45°C for 1 hr.

**TABLE V**
**Critical Value for Each Factor and Predicted Product Yield**

| Factor | Germ | Fiber | Starch | Gluten |
|---|---|---|---|---|
| Concentration (%) | 0.16 | 0.69 | 0.47 | 0.14 |
| Time (min) | 6 | 88 | · · · | · · · |
| Temperature (°C) | · · · | 46 | 40 | 56 |
| Predicted yield (%) | 8.4 | 5.0 | 69.1 | 12.8 |

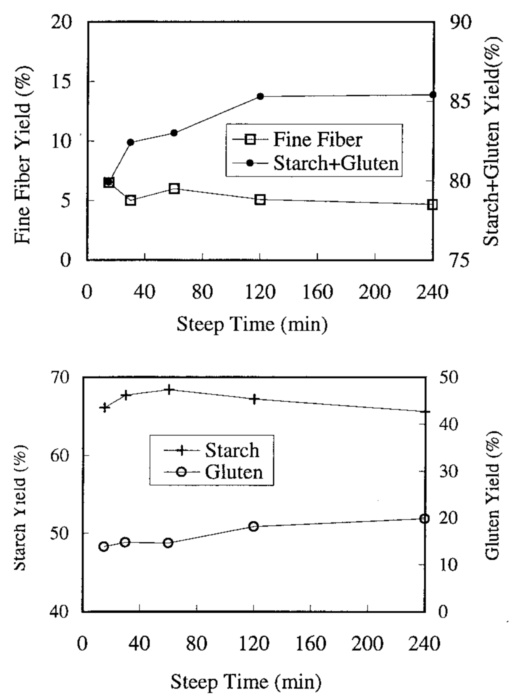


**Fig. 5.** Effect of steep time on corn starch plus gluten yield, starch yield, gluten yield, and fine fiber yield. Debranned corn was steeped in 0.5% NaOH at 45°C.

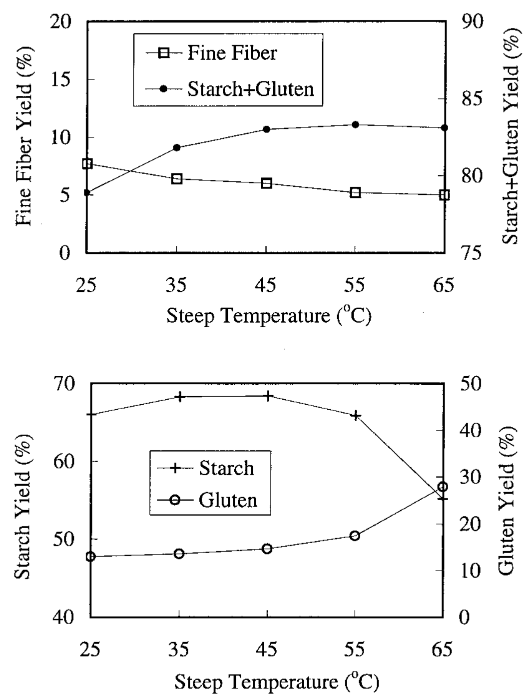


**Fig. 6.** Effect of steep temperature on corn starch plus gluten yield, starch yield, gluten yield, and fine fiber yield. Debranned corn was steeped in 0.5% NaOH for 1 hr.

NaOH concentration was low (0.1–0.3%) and steep temperature was low (35–45°C). A low starch and high gluten yield was also obtained at medium NaOH concentration (0.5%) with a high temperature (65°C) or a high NaOH concentration (0.7–0.9%) with a temperature of 45–55°C. The lowest starch and highest gluten yield were obtained at the highest level of NaOH or the combination of medium to high NaOH level and high temperature. This was due to the swelling of starch granules under those conditions and resulted in difficulty in separation of starch and gluten (Fellers et al 1969). A low starch yield was also obtained at low levels of NaOH (0.1–0.3%), low steep temperature (30–45°C), and short steep time (30–60 min) because of the low solubility and low dispersibility of glutelin under those conditions (Dimler et al 1944). This was also confirmed by the low gluten yield under the same conditions.

The highest starch yields were obtained when steeping cracked corn in 0.3 or 0.5% NaOH for 60 or 120 min at 35 or 45°C. These results indicate that at low NaOH concentration (0.3%) at least twice the steep time is needed and at medium NaOH concentration (0.5%), a temperature of 35 or 45°C can be used. When using a medium concentration of NaOH (0.5%), it would be more energy efficient to use 35°C. When the NaOH concentration and steep time were held constant, increasing the steep temperature caused the starch yield to significantly increase or decrease depending on the relative levels of these factors. The combination of high temperature (>45°C), high level of NaOH (>0.5%), and long steep time (>60 min) increased starch viscosity and resulted in poor separation of starch and gluten.

Increasing steep time significantly increased starch yield and decreased fiber yield at low levels of NaOH. At high levels of NaOH and low temperature, both starch and gluten yield increased with increased steep time while fiber yield decreased. At high levels of NaOH and high temperature, increasing steep time did not significantly affect starch yield. Based on the response surface analysis results, the highest germ yield could be obtained when steeping the cracked corn in 0.16% NaOH for 6 min, lowest fiber yield could be obtained when steeping the cracked corn in 0.69% NaOH at 46°C for 88 min. The highest starch yield and the lowest gluten yield could be obtained when steeping the cracked in 0.47% NaOH at 40°C and 0.14% NaOH at 56°C, respectively (Table V).

When steeping the cracked corn for 1 hr at 45°C, the fine fiber yield decreased and the yield of starch plus gluten increased with an increase in NaOH concentration (Fig. 4). This result indicated that clean fine fiber was obtained. However, this did not result in a high starch yield but rather an increased gluten yield due to the difficulty of the starch and gluten separation. Steeping the cracked corn in 0.5% NaOH at 45°C resulted in the fine fiber yield decreasing and the yield of starch plus gluten increasing with an increase of steep time (Fig. 5). The clean fine fiber again did not result in a higher starch yield but rather a higher gluten yield. When steeping the cracked corn in 0.5% NaOH for 1 hr, increasing the steep temperature also reduced the fine fiber yield but increased the yield of starch plus gluten (Fig. 6). The starch yield reached its peak at ≈35 and 45°C. The results indicate that the separation between starch and gluten was more sensitive to NaOH concentration and steep temperature than to steep time (Figs. 4–6).

In summary, the results indicate NaOH concentration, steep temperature, and steep time significantly affect alkali wet-milling results. Cracked corn could be steeped in 0.5% NaOH for 60 min at 45°C. The optimum conditions for other hybrids may be different and the effect of hybrid on alkali processing parameters needs to be studied.

## CONCLUSIONS

Optimal separation between pericarp and the rest of the corn kernel used in this study occurred when soaking corn in 1.5–2% NaOH at 85°C for at least 5 min. Passing the corn through a roller mill once or twice did not affect the starch yield; however, passing corn through the roller mill twice did increase the flow rate by 62%. Debranned corn should not be passed through a roller mill three times because it caused germ damage and reduced the germ yield by 48%. The gap for passing corn through the mill once should be set at 2 mm; for passing corn through twice, the gap should be set at 3.5 mm for the first pass and 2.0 mm for the second pass. Starch yield was more sensitive to NaOH concentration and steep temperature than to steep time. The highest starch yield was obtained when steeping corn in 0.5% NaOH for 1 hr at 45°C.

## ACKNOWLEDGMENTS

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