

# Intermittent Milling and Dynamic Steeping Process for Corn Starch Recovery

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## ABSTRACT

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A procedure that reduces diffusional limitations by periodically milling the corn to reduce particle size and stirring the ground mash in the presence of sulfur dioxide (SO<sub>2</sub>) and lactic acid was developed. The process, called intermittent milling and dynamic steeping (IMDS), includes three main stages: initial soaking (a short-time immersion in water) of whole kernels, initial cracking of the partially hydrated kernels, and dynamic steeping with interspersed milling. This study evaluated the three stages of the process separately, evaluating the effect of variables on each stage of the process. Corn fractions yield (germ, fiber, gluten, starch) were used to decide the best conditions for the soaking and steeping stages, and germ damage was used to determine the best kernel cracking method. Starch, gluten, and germ yields were not affected by soak temperatures (52–68°C) or soak time (1–3 hr). A temperature of 60°C was

chosen for soaking because it increased the rate of kernel hydration without gelatinizing starch, which happens at higher temperatures. A 2-hr soak time was preferred because there was less fiber in the germ fraction and less germ damage was observed. Although there were no advantage to using SO<sub>2</sub> or lactic acid in the soak water, the presence of these compounds during dynamic steeping enhanced starch yield. The starch yield for 3 hr of dynamic steeping was not statistically different from the starch yield for a 7.5-hr dynamic steep. The Bauer mill was preferred over the use of a roller mill or a commercial grade Waring blender for kernel cracking. The IMDS process produced, on an average, 1 percentage point more starch than the conventional 36-hr steeping process. Total steep or kernel preparation time was reduced from 24–40 hr for conventional wet-milling to 5 hr for the IMDS process.

In corn wet-milling, kernels are countercurrently steeped for 24–40 hr in 50–52°C process water initially containing 0.10–0.20% SO<sub>2</sub>. These steep conditions permit the growth of *Lactobacillus* sp., in the fresh corn, which convert sugars and soluble starch leached from the corn kernels into lactic acid. The lactic acid is beneficial for corn steeping, due to kernel softening and other activity that enhance starch recovery (Roushdi et al 1981, Eckhoff 1989, Eckhoff and Tso 1991b). Du et al (1996) showed that other organic and inorganic acids have an effect on yield similar to that of lactic acid.

In steeping, absorbed SO<sub>2</sub> acts on the disulfide bonds in the endosperm protein matrix that encapsulates the starch granules (Watson 1984). The result is a dispersion of endosperm protein and an enhancement of starch release (Watson and Sanders 1961). Conventional steeping is a diffusion-limited process. Watson and Sanders (1961) showed that when all diffusional barriers are removed, 1–2 hr is enough time for SO<sub>2</sub> to react with the endosperm protein, resulting in the release of the starch granules.

In an attempt to decrease steep time, researchers have tried to enhance the diffusional rate of the process by dry-milling the corn before steeping to get smaller particles and thus decrease the diffusional path length (Chwalek 1980, Powell and McGeorge 1975, Eckhoff et al 1993, Eckhoff and Tso 1991a). Processes that break open the dry kernel before wet-milling produce excessive germ damage. Broken germs are hard to recover in hydrocyclones, and the oil released by broken germs affects starch quality and puts an oil film on process equipment. Other objections to combined wet- and dry-milling processes are that they do not promote lactic acid production and that they cause excessive loss of solubles into the steepwater.

The intermittent milling and dynamic steeping process (IMDS) is a process which, after an initial short soak period, the kernels

are broken up to reduce the diffusional path length for steep solution components (Fig. 1). Unlike conventional steeping, the kernel soak is performed in short time (1–3 hr) with hydration of the germ being the main objective. Breaking open the kernel after this initial hydration phase reduces damage to the germ and retains the ease and purity of germ recovery associated with wet-milling. Kernel cracking, to open up the kernel, and dynamic steeping (steeping with stirring) with intermittent milling, done to enhance germ separation and reduce particle size, are the two other major steps of the process. Upon completion of the IMDS process, the steeped material is sent to first-stage germ recovery without any additional milling. Subsequent wet-milling steps are the same as in conventional wet-milling.

The objectives of this research were to: 1) evaluate the effect of time, temperature, SO<sub>2</sub> concentration, and lactic acid concentration during soaking and dynamic steeping on corn fraction yields, and 2) study the effect of different kernel cracking methods on germ damage and recovery.

## MATERIALS AND METHODS

### Sample Preparation

A yellow dent hybrid (FR600 × Mo17) was used for all parts of the study except for determining the effect of different grinding methods, during which a blend of unknown local hybrids was used. For each test run ≈1,500 g of corn was sieved over a 4.8-mm (12/64") round-hole sieve. Three replicate subsamples were used for moisture determination using the 103°C, 72-hr forced-air oven procedure (AACC 1995). The mean moisture content of the corn was 14.25% wb.

### Effect of SO<sub>2</sub> and Lactic Acid Concentrations During Soaking

A duplicated 2×2 factorial design was used with SO<sub>2</sub> levels of 0 and 0.15% and lactic acid levels of 0 and 0.55%. The purpose of the test was to determine whether SO<sub>2</sub> and lactic acid in the soak water increased starch yield or had an effect on germ recovery.

Cleaned corn (1,000 g) was immersed in 1,867 mL of solution for 2 hr at 60°C using each of the four treatment conditions. The apparatus used for soaking was the same as in conventional batch-laboratory steeping (Eckhoff et al 1993). At completion of the soak period, the soak water was drained and solids determination was performed by the two-stage air oven method (AACC 1995).

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Soaked corn was then cracked by passing it through a 20.3-cm (8") diameter Bauer mill (model 148-2) with 0.5-cm gap between the plates (one stationary and the other rotating at 1,500 rpm). Dynamic steeping, with a total time of 6 hr, was accomplished at 52°C in 1,600 mL of a solution of 0.2% SO<sub>2</sub> and 0.55% lactic acid. The cracked corn and steep solution was stirred inside a plastic bottle equipped with a variable speed agitator. During the dynamic steeping, intermittent milling was performed by placing all the material in a Waring blender (model 34BL22) for 2 min at 60% of full power at 2 and 4 hr from the beginning of dynamic steeping. After the last 2 hr of dynamic steeping, the mash was skimmed for germ. All other process steps followed the laboratory procedure of Eckhoff et al (1993). The steepwater from dynamic steeping was used as process water in subsequent milling steps.

### Effect of Soak Time and Soak Temperature

A 3×3 duplicated factorial experimental design was used at soak times of 1, 2, or 3 hr and temperatures of 52, 60, or 68°C. No SO<sub>2</sub> nor lactic acid was used in the soak water (a condition determined from the previous test). All milling procedures were the same as previous described.

### Evaluation of Kernel Cracking Methods

Three methods for kernel cracking were compared to determine which one opened up the kernel with a minimum of germ damage. A 10-cm diameter × 15-cm wide smooth roll roller mill (model FD 2305), a commercial grade Waring blender (model 34BL22), and a 20.3-cm (8") diameter Bauer mill (model 148-2) were the three cracking methods compared.

A double-pass method was used for the roller mill with a gap setting of 3.99 mm (0.16") for the first pass and 3.06 mm (0.12") for the second pass. The double-pass system was needed due to the size variability of the corn kernels. The gap settings were selected based upon some preliminary tests. Soaked corn was dropped into the nip of the cylinders which were rotating in opposite directions at 150 rpm. The cracked kernels were collected at the bottom of the rolls, and 250 mL of water was used to rinse the cylinders after cracking to increase total mass recovery.

The Bauer mill was tested at 1,500 rpm using three different plate configurations: C-E Bauer plates 8504, 8118, and 8818, manufactured by C-E Bauer Combustion Engineering. The plate set 8504 gave the best results in preliminary tests, while the other two produced an excessive amount of broken germ and a large amount of fines as measured by visual inspection. Only the 8504 set of plates was tested in the cracking method experiment. Soaked kernels were hand-fed into the mill at an approximate rate of 3,000 g/min. After grinding, the mill was opened and rinsed with 250 mL of water to ensure total mass recovery.

The Waring blender had its blades dulled to a radius edge to prevent slicing of the germ, as described by Eckhoff et al (1993), and was controlled by a variable transformer (model 116, Superior Electric Co., Bristol, IA). Combinations of blender time (1–3 min) and power (30–70% of full blender power) were tested in preliminary tests. The combination giving the most complete cracking of the kernels with minimum germ damage was 2 min at 45% of full power, which is the condition used in the comparison. Soaked corn was divided, before cracking, into three equal groups of ≈600 mL volume. Each batch of corn was mixed with 500 mL of water and then cracked in the blender separately. The three batches were recombined for steeping.

For all three methods, the germs were recovered after dynamic steeping by skimming using 18- and 14-mesh pieces of stainless steel screen, following the procedure of Eckhoff et al (1993). During germ skimming, the amount of time required for germ recovery was kept between 20 and 30 min to provide for a fair comparison of germ yield. Recovered germ was rinsed with 1 L of water over a 0.4-mm round-hole sieve.

After washing, the germ was dried at 49°C for 24 hr, sieved for 4 min through a stack of four U.S. Standard sieves of 6, 8, 10, and 16 mesh on a RoTap shaker (W.S. Tyler Co., Cleveland, OH), and the fractions on each sieve weighed. For all germ samples tested, no whole germ passed through the 10-mesh sieve. Particles on the 16-mesh sieve and in the pan were considered to be pieces of broken germ. Percentage of broken germ was determined by adding the weight of the germ pieces in the 16-mesh sieve and in the pan and dividing by the total dried germ weight.

The germ samples recovered by each of the different grinding methods were compared to germ recovered from conventionally steeped corn. Oil content of the germ fractions was measured by hexane extraction (AOAC 1984). The procedure of Eckhoff et al (1993) was used with a steep time of 36 hr, SO<sub>2</sub> concentration of 0.15%, and 0.55% lactic acid.

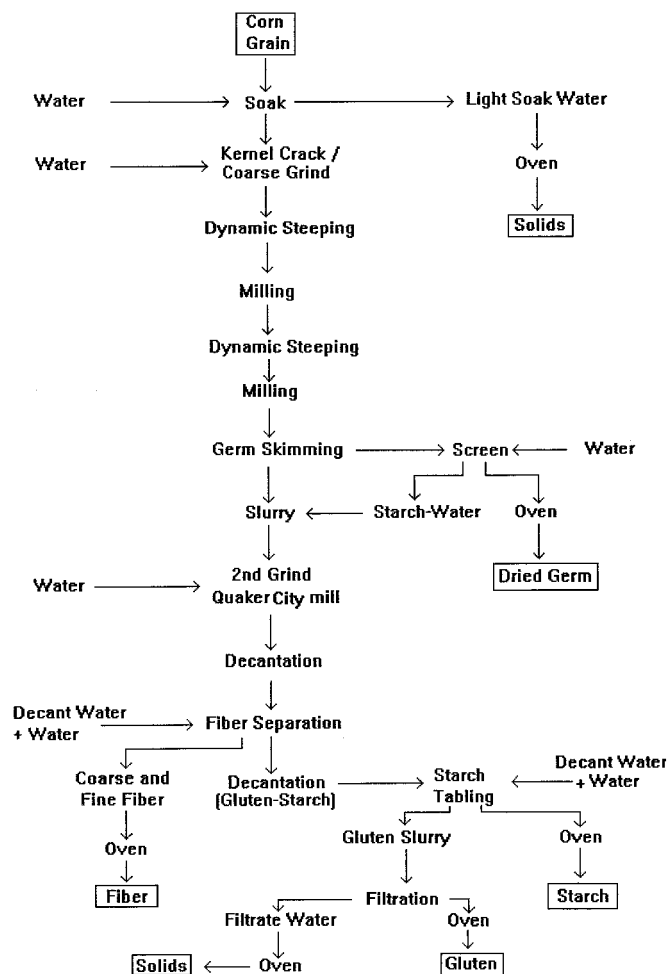


Fig. 1. The intermittent milling and dynamic steeping process (IMDS). After an initial short soak period, the kernels are broken up to reduce the diffusional path length for steep solution components.

TABLE I  
Product Yields from Intermittent Milling and Dynamic Steeping (IMDS) of Wet-Milled Corn Soaked in Solutions Containing Different Concentrations of SO<sub>2</sub> and Lactic Acid at 60°C<sup>a</sup>

Lactic Acid (%)	SO <sub>2</sub> (%)	Soak Solids	Germ	Fiber	Starch	Gluten	Filtrate Solids
0.0	0.0	0.1a	7.4ab	10.6ab	66.5a	8.9a	5.8a
	0.15	0.4b	7.1c	10.3b	65.9a	10.1a	5.5a
0.55	0.0	0.7c	7.5a	12.4a	64.3a	10.1a	4.8a
	0.15	0.4b	7.2bc	11.1ab	66.4a	9.0a	5.5a

<sup>a</sup> Each value represents the mean of two replicates. Mean comparisons followed by the same letter are not significantly different ( $P > 0.05$ ) within same column.

### Effect of Lactic Acid in Dynamic Steeping

To determine the effect of lactic acid during dynamic steeping in the IMDS process, four replicates of the procedure with and without lactic acid in the steepwater were performed. Cleaned corn (1,000 g) for each replicate was soaked in water and cracked with the Bauer mill set at a 0.5-cm gap and operated at 1,500 rpm. The cracked kernels were steeped in 0.15% SO<sub>2</sub> steepwater with or without 0.55% lactic acid. Yields of starch, germ, fiber, gluten, and solids from the soak water and filtrate were measured and reported on a dry solids basis following the procedures of Eckhoff et al (1993). Starch yield was used as the deciding factor regarding whether or not to use lactic acid during steeping in the IMDS process.

### Effect of SO<sub>2</sub> Level and Steep Time

A 4×3 full factorial experiment with duplicate determinations was performed to study the effect of time and SO<sub>2</sub> concentration during dynamic steeping. The temperature was held constant at 52 ± 1°C since preliminary runs using higher temperatures (≥55 ± 1°C) made stirring the material difficult due to the formation of grumes in the mash. Steep times of 3.0, 4.5, 6.0, and 7.5 hr, and SO<sub>2</sub> concentrations of 0.05, 0.1, and 0.15% were used. The steep time was divided into three equal time periods for all steep times with intermittent milling after the first two steeping periods. For example, a 3-hr steep time had intermittent milling after 1 and 2 hr.

Along with starch yield, the protein content of starch and oil content of the germ fraction were used as quality factors. Protein in starch was determined by Kjeldahl method (AACC 1995) and oil content of the germ was measured by hexane extraction (AOAC 1984).

### Comparison of the IMDS Process to 36-hr Batch Steeping

For comparison, two 1,000-g batches were steeped for 36 hr at 52°C in a 0.2% SO<sub>2</sub> and 0.55% lactic acid solution using the procedure of Eckhoff et al (1993). For the IMDS process, 1,000-g samples of cleaned corn were used as previously described. After 2 hr of soaking and cracking the kernels, the corn was dynamic steeped in a 0.15% SO<sub>2</sub> solution with 0.55% lactic acid for a total of 3 hr (1-hr steep followed by milling). Comparison between the product yields were made.

### Statistical Analysis

The yields of starch, germ, fiber, gluten, soak water solids, and filtrate solids were submitted to analysis of variance (ANOVA), and least significant difference (LSD) values were calculated at a 5% level for fraction yield means comparison tests. The analysis was done using an SAS computer program (SAS Institute, Cary NC).

## RESULTS AND DISCUSSION

### Effect of SO<sub>2</sub> and Lactic Acid Concentration During Soaking

Visual observation of cracked corn showed that all soaking treatments were homogeneous and similar one to the other. The

germ floated well and recovery took ≈30 min, which was approximately the same time required for the conventionally steeped sample. No differences were observed in the starch, gluten, or filtrate solids yields due to the addition of lactic acid or SO<sub>2</sub> in the soak water (Table I). The fiber, germ, and soak solids fractions were statistically dependent upon the addition of lactic acid or SO<sub>2</sub> in the soak water.

Higher germ recovery was observed when SO<sub>2</sub> was not present. Fan et al (1965) showed that SO<sub>2</sub> retards water absorption rate at the beginning of hydration but increases water absorption rate later. The 2-hr soak period used in the IMDS process would correspond to the early stages of hydration. Thus, the absence of SO<sub>2</sub> might have improved the germ hydration, which consequently facilitated germ release from the kernel. Measurement of germ oil content shows no difference between the various treatments and the control.

Although soak solids and fiber yields showed differences due to the addition of lactic acid and SO<sub>2</sub>, the values of fiber yields are in the range normally found in the conventional wet-milling procedure: 10.8% (Yapenco 1996) and 12.5% (Anderson 1982). Values for soak solids are insignificant when compared to the other fractions yields.

The protein content in starch was similar for all treatments, ranging from 0.45% for the use of both lactic acid and SO<sub>2</sub> to 0.55% for lactic acid alone. This difference is within the range of the standard error of protein measurement previously observed (Wang 1994). The amount of protein in starch was higher than for the 36-hr conventional steeped samples (0.45 vs. 0.31%). For the production of ethanol, this difference would not be important, although for other starch products, such as specialty starches and fructose, more protein in the starch would be a disadvantage because it would require more starch washing hydrocyclone stages to achieve a comparable level of protein.

The oil content of the germ was similar for the control and all treatments, varying from 40% for lactic acid alone to 43.7% when only SO<sub>2</sub> was used (control was 42.2%). These values are comparable to the 41.3% oil content reported by Vojnovich et al (1975), but are lower than reported industrial values of 45–50% (May 1991).

### Effect of Soak Time and Soak Temperature

Soak time and temperature had little effect on the starch, gluten, and germ yields (Table II). Significant differences were observed for fiber and gluten filtrate solids. Soak solids varied from 0.1 to 0.3%, but there was no discernible pattern to the results. Gluten filtrate solids varied from 5.0 to 6.2%. The two extremes occurred for samples with 3 hr of soaking. Filtrate solids decreased at all soak times with increasing soak temperature. The soak water and gluten filtrate solids are the most difficult fractions to measure accurately, due to the low concentration in the solution and error associated with subsampling.

TABLE II  
Product Yields (%) from Intermittent Milling and Dynamic Steeping (IMDS) of Wet-Milled Corn Soaked at Three Different Time and Temperature Combinations<sup>a</sup>

Time (hr)	(°C)	Product Yields (%)					
		Soak Solids	Germ	Fiber	Starch	Gluten	Filtrate Solids
1	52	0.1ab	7.2a	11.1bc	66.1a	9.2a	6.1ab
	60	0.3a	7.5a	10.8b	65.5a	9.0a	5.6a-c
	68	0.1b	7.5a	11.2bc	66.0a	9.2a	5.5a-c
2	52	0.1b	7.4a	11.1bc	66.4a	8.2a	6.1ab
	60	0.2ab	7.4a	11.0bc	65.4a	9.8a	5.8ab
	68	0.1ab	7.2a	12.0a	65.4a	9.9a	5.4bc
3	52	0.2ab	7.1a	10.7c	66.6a	8.9a	6.2a
	60	0.2ab	7.4a	11.9a	65.8a	8.9a	5.5a-c
	68	0.2ab	7.5a	11.5ab	65.7a	9.1a	5.0c

<sup>a</sup> Each value represents the mean of two replicates; mean comparisons followed by the same letter are not significantly ( $P>0.05$ ) different within same column.

**TABLE III**  
Percent Distribution of Germ Particles on Sieves, Germ Damage, and Germ Yields After Cracking the Kernels<sup>a</sup>

Cracking Method	Germ Damage	Germ Yield	Sieve No.				
			6	8	10	16	Pan
Blender	9.4a	7.3a	20.0	62.5	8.3	6.6	2.8
Roller mill	8.4ab	6.3b	16.8	66.9	8.3	5.9	2.5
Bauer mill	6.5bc	7.2a	16.7	68.2	8.5	4.5	2.0
Control	6.1c	6.9ab	7.5	78.7	8.1	4.5	1.6

<sup>a</sup> Except for the control (two replicates) each value represents the mean of three replicates. Means comparison followed by the same letter within the same column were not different ( $P > 0.05$ ).

The most important observations regarding the effect of time and temperature was that the 1-hr soak time samples exhibited more difficulty in germ skimming. There was more pericarp floating with the germ and the germs did not float well. Singh (1995) found that the ease of germ separation increased with increasing soak time. He also found that gelatinized starch resulted in difficult germ recovery after soaking at 75°C. For practical purposes, the 68°C soak temperature is too high to be controlled without increasing risk of gelatinization of starch. Soak times of 3 hr at 60 and 68°C resulted in an increase in broken germs (based on visual observation). A 2-hr soak time is recommended based on the difficulties encountered in germ recovery at 1 hr. The temperature recommended is 60°C, which promotes faster germ hydration than 52°C and presents less risk of gelatinization of starch than 68°C.

Protein content of the starch was similar for all combinations of soak time and temperature. Values ranged from 0.44 (1 hr/68°C) to 0.51% (1 hr/52°C). All combinations were higher than the protein in starch for the 36-hr batch-steeped control (0.31%).

The oil content of the germ for 36-hr batch-steeped control was 49.4% compared to 41.1% (2 and 3 hr/68°C) to 42.9% (1 hr/68°C and 3 hr/60°C) for the combinations of soak time and temperatures tested. Either the solubles were not removed completely from the IMDS germ, or there was no additional fiber or endosperm attached to the conventionally milled germ. Due to the short soak time (2 hr) used in the IMDS process compared to conventional steeping (24–40 hr), it is likely that less solubles were removed from the germ. To achieve a 7% increase in oil content, total solubles would need to increase by 0.5 percentage points on the total kernel basis. The normal steep solids plus filtrate solids for conventional batch steeping is 7.0% (Eckhoff et al 1993), while the average soak solids and gluten filtrate solids for the IMDS was 5.7%.

### Kernel Cracking Method

The Bauer mill and conventional 36-hr batch-steeping produced the least amount of germ damage, while the blender and roller mill produced the highest amount of damage (Table III). The percent of germ damage increased from 6.1% (control) to 9.4% (blender). The average germ-particle distribution for all methods resulted in a majority of the germ staying on the No. 8 sieve. The control sample had 78.7% of the germ samples on the No. 8 sieve. However, the other three cracking methods had a larger percentage retained on the No. 6 sieve. One reason for this could have been due to the attachment of pericarp to the germ. Visual observations of the samples indicated that the samples were equally clean. Another potential reason for the higher percentage of germ retained on the No. 6 sieve is that less solubles were removed from the germ during the 2-hr soak than would occur during the 36 hr of conventional steeping. If less solubles are removed, the germ will shrink less and more germ will be retained on a larger sieve.

The highest germ yield resulted from the blender at 7.3%, but it was not statistically different from the Bauer mill or the control (Table III). The roller mill was statistically different from the

**TABLE IV**  
Product Yields (%) from Intermittent Milling and Dynamic Steeping (IMDS) of Wet-Milled Corn Steeped in 0.15% SO<sub>2</sub> With and Without Lactic Acid<sup>a</sup>

Corn Fractions	Lactic Acid	
	0%	0.55%
Soak solids	0.1a	0.1a
Germ	7.8a	7.3a
Fiber	15.2a	11.3b
Starch	61.8a	65.8b
Gluten	9.0a	9.0a
Filtrate	4.2a	5.4b

<sup>a</sup> Mean comparisons followed by the same letter within the same row are not significantly ( $P > 0.05$ ) different.

blender and the Bauer mill with only 6.3% recovered germ. The blender was deemed an unacceptable method of cracking, not just because of the higher amounts of germ damage, but because it left some kernels uncracked. The roller mill was similar in germ damage to the blender and was not deemed acceptable because of the need to have at least two passes to prevent excessive damage to the germ. The Bauer mill method is the recommended method for kernel cracking.

### Effect of Lactic Acid in Dynamic Steeping

Starch yield and filtrate solids increased and the fiber fraction decreased when lactic acid was used (Table IV). The average starch yield difference between steeping with lactic acid and without lactic acid was 4.0%. This is about the same percentage point increase in starch yield reported by Eckhoff and Tso (1991b) and Steinke et al (1991). These authors reported the increase in starch yield was related to a decrease in fiber which contains a large fraction of starch. Similar increases in fiber yield were observed in this study.

Wang (1994) reported that as the concentration of lactic acid increased, larger amounts of solids were released. This may help to explain the higher solids in the gluten filtrate found in the present study. In the IMDS process, the steep solution remains as a component of the process water throughout the whole milling procedure. Soluble solids released in steeping would result in an increase in gluten filtrate solids.

Lactic acid also helps increase starch released from the endosperm after the first grind. Steeping in the presence of lactic acid produced a slurry before germ skimming with a higher density (10–12 Bé) than when steeping without lactic acid (7–9 Bé). The higher density improved the ease of germ skimming due to better germ flotation.

### Effect of SO<sub>2</sub> Level and Steep Time

There were differences in the yield of starch, germ, and fiber due to both time and SO<sub>2</sub> concentration. No treatment effects were found for gluten filtrate or soak water solids. For gluten filtrate solids, there was a difference between 4.8% (3 hr/0.05% SO<sub>2</sub>) and 5.7% (6 hr/0.10% SO<sub>2</sub>). There were no time by SO<sub>2</sub> interaction effects for all fraction yields (Table V).

Three-hour total steeping time had similar starch yield to the 6- and 7.5-hr steep times (Table V). When 0.15% SO<sub>2</sub> was used, starch yields were higher than for the other two SO<sub>2</sub> levels (i.e., 0.05% SO<sub>2</sub> and 0.1% SO<sub>2</sub>). The average starch yield increased with increasing SO<sub>2</sub> concentration from 0.05 to 0.15% for each steep time by 1.7, 2.3, 1.8, and 2.0 percentage points for 3, 4.5, 6, and 7.5 hr of steeping, respectively.

The highest germ yield was 7.4% for 6 hr of steeping, which was not statistically different from the 7.0% obtained for 7.5 hr or the 7.1% obtained for 3 hr. Within SO<sub>2</sub> levels, 0.15% SO<sub>2</sub> had the best germ recovery (7.4%), while 0.05% SO<sub>2</sub> resulted in low germ recovery (6.7%). Germ yields obtained by this work are comparable

**TABLE V**  
**Product Yields (%) from Intermittent Milling and Dynamic Steeping (IMDS) of Wet-Milled Corn Steeped for Different Times and SO<sub>2</sub> Concentration Combinations with 0.55% Lactic Acid<sup>a</sup>**

Time(hr)	SO <sub>2</sub> (%)	Soak Solids	Germ	Fiber	Starch	Gluten	Filtrate Solids
3	0.05	0.2a	6.7a	13.9ab	63.8de	9.0a	4.8b
	0.10	0.2a	6.9ab	12.9b-e	64.7b-d	9.1a	5.1ab
	0.15	0.1a	7.1bc	11.7de	65.4ab	9.2a	5.5ab
4.5	0.05	0.2a	7.0b	14.7a	62.9e	8.4a	5.0ab
	0.10	0.2a	6.7ab	12.4c-e	64.6b-d	8.8a	5.6ab
	0.15	0.2a	7.0bc	12.5b-e	65.8a-c	8.6a	5.6ab
6	0.05	0.2a	6.9ab	13.3a-c	64.4b-d	8.8a	5.5ab
	0.10	0.2a	7.1bc	11.8de	65.1a-c	9.2a	5.7a
	0.15	0.2a	7.4c	11.6e	66.2a	8.7a	5.5ab
7.5	0.05	0.2a	7.0bc	13.2a-d	64.1c-e	9.2a	5.1ab
	0.10	0.1a	6.9ab	12.5b-e	65.6a-c	8.7a	5.5ab
	0.15	0.2a	7.0ab	11.5e	66.1a	8.2a	5.4ab

<sup>a</sup> Each value represents the mean of two replicates. Mean comparisons followed by the same letter are not significantly different ( $P > 0.05$ ) within same column.

**TABLE VI**  
**Protein Content (%) of Starch and Oil Content of Germ Steeped Under Different Conditions**

Time(hr)	Starch			Oil		
	% SO <sub>2</sub> Treatment					
	0.05	0.10	0.15	0.05	0.10	0.15
3	0.30	0.32	0.32	42.7	43.0	43.4
4.5	0.34	0.32	0.34	41.8	41.8	42.0
6	0.33	0.35	0.32	44.0	42.1	42.3
7.5	0.32	0.34	0.31	43.7	42.9	43.2
Control	...	...	0.31	...	...	47.2

with results from previous studies: 7.3% (Knight 1969), 7.0% (Eckhoff et al 1993), and 6.2% (Anderson and Watson 1982).

Fiber yields ranged from 11.6% (7.5 hr/0.15% SO<sub>2</sub>) to 14.7% (4.5 hr/0.05% SO<sub>2</sub>). However, no difference was found between 3 and 7.5 hr of steeping with 0.15% SO<sub>2</sub>. Despite different steep times, the lowest fiber yields were obtained when 0.15% SO<sub>2</sub> was used. A reasonable explanation of this phenomenon is that the SO<sub>2</sub> was more available to break down the structural protein network at higher SO<sub>2</sub> levels (0.10 and 0.15%) than at the level of 0.05%. Normally, an increase in starch yields is related to a decrease in fiber fraction. This is because more starch is released from the fiber which also indicates a good disruption of the protein matrix.

Total solids soluble (soak water solids plus gluten filtrate solids) ranged from 5.0% for 3 hr of steeping with 0.05% SO<sub>2</sub> to 5.7% for 6 hr with 0.10% SO<sub>2</sub>. These values are lower than total solids released when standard procedure of 36 hr of steeping was used: 7.5% (Anderson and Watson 1982), 6.8% (Knight 1969), 7.0% (Eckhoff et al 1993). A smaller amount of solubles released is desirable because they end up in the low-valued gluten feed fraction.

#### Starch and Germ Quality at Different Steeping Conditions

The protein content of starch ranged from 0.31% for the control to 0.35% for 6 hr of steeping in 0.1% SO<sub>2</sub> solution (Table VI). The percentages found in this study were lower than the protein in starch in some reports: 0.56% (Steinke et al 1991), 0.63% (Rubens 1990), and 0.54% (Anderson 1963); but they were similar to those found by others: 0.32% (Eckhoff and Tso 1991a), 0.30% (Watson 1967), and 0.36% (Watson et al 1951). No relationship was observed between protein content and steep time or concentration level of SO<sub>2</sub>. Overall, shorter steep times than those of the standard procedure (36 hr/52°C) or low SO<sub>2</sub> levels (0.05 or 0.1%) did not have a detrimental effect on the quality of the starch as measured by residual protein.

The oil content of the germ for all treatments ranged from 41.8% for the 4.5-hr steep with 0.05 and 0.1% SO<sub>2</sub> to 44% for the 6-hr steep with 0.05% SO<sub>2</sub> (Table VI). There was a higher oil content in the germ for the control (47.2%) than for the treatments (≤44%). The largest difference was obtained between the 4.5-hr

**TABLE VII**  
**Percentage of Product Yields From Wet-Milled Corn Using Intermittent Milling and Dynamic Steeping (IMDS) and Standard Procedures<sup>a</sup>**

Fractions	Method	
	IMDS	Standard
Soak and steep solids	0.1a	4.0b
Germ	7.4a	7.1a
Fiber	11.6a	12.0a
Starch	66.2a	65.1b
Gluten	8.7a	9.2a
Filt. solids	5.5a	1.7b
Total	99.3	99.2

<sup>a</sup> Each value represents the mean of two measurements. Mean comparisons followed by the same letter within same row are not significantly different ( $P > 0.05$ ).

steep with 0.05 and 0.10% SO<sub>2</sub> and the control, which accounted for 5.4 percentage points difference. Increased retention of solubles in the germ is probably the reason for lower oil content, but there is also less total oil recovered in the germ fraction. Based upon the data comparing conventional steeping to the IMDS process, the IMDS process will yield 1.2 kg less oil per metric ton of corn processed.

#### Comparison of the IMDS Process to 36-hr Batch Steeping

The only statistical difference observed was for starch yield (Table VII). Starch yield was 0.9% higher for the IMDS process than for conventional steeping. Differences in gluten filtrate and soak water solids also were observed. However, when the two are added together to form total solubles, the results are 5.6% for the IMDS process and 5.7% for the conventional steeping. Because of the difficulty in accurately measuring solubles, the IMDS process probably has a lower total soluble recovery than a conventional process, even though such results were not observed in this set of data.

The advantage of the IMDS process over conventional steeping, is that the IMDS process requires a total combined soak and steeping of 5 hr compared to 24–40 hr for conventional steeping. There was a slight increase in starch yield for the IMDS, and protein content in starch was comparable to that of conventional steeping. The only disadvantage of the IMDS process is that the germ oil content is lower (42 vs. 47%) due to the retention of solubles in the germ and total extractable oil in the germ is less by ≈1.2 kg of oil per metric ton of corn processed.

## CONCLUSIONS

Although the use of lactic acid and SO<sub>2</sub> during soaking did not increase starch yield, the use of lactic acid during dynamic steeping increased starch yield and decreased the fiber fraction by 4 percentage points. Increasing SO<sub>2</sub> levels from 0.05 to 0.15% in the steep solution increased starch yield by an average of 2 percentage points. Starch yield, protein content in starch, gluten yield,

and germ yield were not affected by soak temperature (52–68°C) or soak time (1–3 hr), but the germ had less damage and was more easily recovered at a 2-hr soak time at 60°C. There was no difference in starch yield between 3 and 7.5 hr of dynamic steeping. Based upon germ damage and particle size of the resulting mash, the Bauer mill is the best method for kernel cracking. It produced the same amount of germ damage as a conventional 36-hr batch steeping. Due to the lower amount of solubles removed, germ oil content from the IMDS is  $\approx$ 7 percentage points lower than germ from conventionally steeped corn. Protein in starch levels obtained by the IMDS process (average 0.32%) were similar to the protein in starch recovered by the conventional process (0.31%). The IMDS process produced  $\approx$ 1 percentage point more starch than the conventional 36-hr batch-steeping process.

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