REVIEW

MILLING

Wet Milling of Corn—A Review of Laboratory-Scale and Pilot Plant-Scale Procedures

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ABSTRACT

New corn hybrids and various handling and processing conditions can be evaluated using laboratory- or pilot plant-scale procedures. Review of laboratory- and pilot plant-scale wet-milling procedures used in past research indicates that while there are significant differences in some of the procedures, most of the procedures can result in starch yield and other component yields comparable to industrial values. The major variable affecting accuracy and precision appears to be the attention to detail expended by the researcher. Development of a standardized wet-milling procedure may enhance the ability to compare data between researchers.

In the past 50 years, various methods have been developed to evaluate the milling characteristics (millability) of corn for wet milling. Wet milling is the industrial process for extracting starch from cereal grains and has been presented in detail (Anderson 1970, Berkhout 1976, Watson 1984, Simms 1985, May 1987, Johnson 1991, Blanchard 1992).

Corn millability can be estimated using small samples of corn by determining the quantity and the quality of the recoverable components as well as the relative difficulty encountered in component separation. Millability studies have been done to evaluate differences in wet-milling characteristics of corn varieties and hybrids (Anderson and Pfeifer 1959; Anderson et al 1960, 1961a; Anderson and Griffin 1962; Anderson 1962, 1965; Dimler 1966; Watson and Yahl 1967; Zehr et al 1995); and to study the effects of growth location (Singh 1994), harvesting conditions (Brown et al 1979; Weller 1987; Weller et al 1988, 1989), drying conditions (MacMasters et al 1954, 1959; Watson and Hirata 1962; Lasseran 1973; Vojnovich et al 1975; Brown et al 1979; Le Bras 1982; Weller 1987; Weller et al 1988, 1989; Mistry et al 1993), storage time (Lasseran 1973), use of different steeping procedures (Anderson et al 1961b; Roushdi et al 1979, 1981a–c; Krochta et al 1981; Hassanean and Abdel-Wahed 1986; Caransa et al 1988; Steinke and Johnson 1991; Steinke et al 1991; Fox and Eckhoff 1993; Shandera et al 1995; Biss and Cogan 1996), and alternative processing techniques on product yields (Yahl et al 1971; Ling and Jackson 1991; Wang and Johnson 1992a,b; Neryng and Reilly 1984; Eckhoff and Tso 1991a,b; Rausch et al 1993, Eckhoff et al 1993a). Millability studies have also been conducted to study the relationships of grain proximate composition and physical properties to wet milling (Fox et al 1992) and to predict starch yield using spectroscopy techniques (Wehling et al 1993).

The corn wet-milling industry has, over the years, expended considerable resources to improve the mechanical efficiency of their processes by focusing on improved process control and more efficient process equipment. However, the process has not achieved the level of efficiency of many other processing industries due to variability in the milling quality (millability) of incoming corn. Recent advances in biotechnology and genetic engineering, as applied to corn hybrid development, have increased the diversity within commercial hybrids. This has focused industrial attention on finding ways to decrease the variability in incoming corn and reduce production costs (Eckhoff 1995).

Millability of corn samples can be estimated by using either laboratory- or pilot plant-scale wet-milling procedures. The difference between laboratory- and pilot plant-scale wet-milling is more than just sample size, although sample size can make a major difference. Laboratory procedures generally mill 50 g to 2 kg of corn, while pilot plant milling can be in quantities ranging from 10 kg to 70 MT. Pilot plant-scale milling will often use small-scale industrial equipment to make the fractionations and separations; whereas, laboratory-scale milling uses much smaller equipment, mostly different in design from industrial equipment. Pilot plant-scale studies tend to be more expensive, require larger quantities of a particular hybrid than laboratory milling, and is generally justified only when a relatively large amount of starch is required for subsequent testing of starch properties, or when new processing technologies are being scaled up. This article reviews the various laboratory- and pilot plant-scale wet-milling procedures used in assessing millability and compares component yields to those obtained in industry.

SAMPLE PREPARATION

Commercial corn is received at the milling facility in the shelled form (i.e., kernels already removed from the cob) having been inspected for U.S. Grade factors and undesirable mycotoxins (Freeman 1973). The corn is cleaned using reciprocating screens to remove some foreign material and broken kernels before steeping.

Samples for laboratory- or pilot plant-scale milling can be hand-picked (Wehling et al 1993), hand-sieved (Shandera et al 1995), or mechanically cleaned (Steinke and Johnson 1991, Eckhoff et al 1993, 1996) for foreign material, mold, heat damage, and broken kernels before analysis. The choice of cleaning method depends on the objective of the milling test. If the objective is to compare the millability of various hybrids, it would be prudent to assure that all samples have comparable levels of damaged kernels and foreign material. Such corn should be well cleaned and free from foreign material and kernel defects. For testing commercial elevator or bin samples, the sample should be representative of the corn as it might be milled and cleaned to some degree with a mechanical cleaner.

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Mechanical cleaning can be performed by using a variety of equipment. Steinke and Johnson (1991) screened corn using a Carter dockage tester, while Eckhoff et al (1993b) used a Gamet reciprocating shaker and a 4.76-mm round-hole sieve. Mostly, other researchers have failed to report the method of cleaning used. Choice of cleaning equipment should yield samples representative of the corn to be tested.

The size of sample to be milled also determines whether and to what degree the sample should be cleaned. The smaller the sample size, the less foreign material should be present in the sample due to difficulty in assuring uniform distribution of the foreign material, particularly if the foreign material includes large particles of corn cobs, stalks, or broken kernels.

The size of sample used in previous research has varied from 50 g to 1,500 g (Table I). Selection of the sample size primarily depends upon the accuracy of the test procedure and the amount of starch required for subsequent testing. The smaller the sample size, the more care the researcher or miller may need to take to ensure the reproducibility of the results. A 1-g loss of solids while milling 100 g of corn reduces total solids recovery by 1%; whereas, solids recovery is reduced only 0.1% when 1 g is lost while milling 1 kg of corn. About 60–65 g of starch can be obtained by laboratory wet-milling 100 g of corn, while pilot plant-scale milling of 25 kg of corn can yield over 15 kg of starch.

STEEPING

Steeping softens the corn kernels for grinding, facilitates disintegration of the protein matrix that encapsulates the starch granules in the endosperm, and removes solubles, mainly from the germ, to increase germ recovery (Cox et al 1944). Sulfurous acid, used to disrupt the protein matrix, also acts to limit undesirable fermentation during steeping. Steeping, which is considered to be the heart of the wet-milling process, has been conducted in laboratory- and pilot plant-scale milling primarily in three ways: 1) static batch, 2) recirculated batch, and 3) countercurrent steeping (Table I).

In static-batch steeping, the corn sample and the steeping solution are placed in a beaker or flask with no stirring, agitation or mixing of the solution (Steinke and Johnson 1991, Fox et al 1992, Eckhoff et al 1996). Steep temperature is controlled by placing the container in a waterbath or other temperature-controlling device.

Recirculated-batch steeping involves continuous pumping of the steep solution through a temperature-controlled heater or waterbath to maintain proper steep temperature (Anderson 1957, 1963; Roushdi et al 1979, Watson 1984, Krochta et al 1991, Eckhoff and Tso 1991a, Eckhoff et al 1993b, Singh and Eckhoff 1995a). Steepwater may also be recirculated through a make-up tank where the composition of the steepwater may be changed to simulate industrial steeping practice (Rubens 1990). The steepwater recycling rate should be sufficiently high to ensure that there are no external mass transfer limitations. Eckhoff and Tso (1991a) recirculated steepwater at a rate of ≈380 ml/min for steeping 1,500-g samples. Krochta et al (1981) reported that a recirculation rate of 150–200 ml/min was sufficient to ensure uniform concentration in the steep solution. In a 100-g procedure, Eckhoff et al (1996) found no difference in starch yield due to the use of static batch steeping. Shandera et al (1995), while steeping 300-g corn samples, recirculated steepwater at 150 ml/min during the first hour; thereafter, the steepwater was recirculated for 15 min every hour during the 40-hr steeping period.

The steeping solution in static-batch and recirculated-batch steeping systems have generally contained 0.10–0.20% sulfur dioxide (Table I) which is comparable to industrial practice (Blanchard 1992). Eckhoff and Tso (1991a) showed that the starch yield increased from 64.9 to 67.3% when sulfur dioxide was increased from 0.1 to 0.2%. Krochta et al (1981), who used mill starch (starch and protein fractions combined) as an index of millability, reported mill starch yields of 66.6, 70.3, 71.3, and 72.6% for the SO_2 concentrations of 0.1, 0.2, 0.3, and 0.4% in the steep solution, respectively.

Lactic acid may also be added to the steeping solution to better simulate the industrial steeping process (Roushdi et al 1981c; Ling and Jackson 1991; Eckhoff et al 1993b, 1996; Singh and Eckhoff 1995a). Du et al (1996) found that starch yield increased from 59.1 to 63.8% when 0.55% lactic acid was added to the steeping solution. Similar results were obtained by Eckhoff and Tso (1991a), who reported that the starch yield increased from 64.9 to 69.1% with the addition of 0.55% lactic acid to the steeping solution. The work of Steinke et al (1991) compared the starch yield from a countercurrent steep apparatus, which produced lactic acid, to a batch-steeping system with no added lactic acid, also showed ≈6% increase in starch yield due to the production of lactic acid. Roushdi et al (1981c) reported starch yield of 63.8% when samples were steeped in solution containing 100 ppm SO_2 and 0.55% lactic acid compared to a starch yield of 58.5% when samples were steeped in solution containing 300 ppm SO_2 and no lactic acid. Du et al (1996) found that other organic and inorganic acids could be substituted for lactic acid and give a similar yield increase.

The amount of steepwater used to steep the corn in batch steeping is also an important factor because the steepwater carries the SO_2, and total absorbable SO_2 increases with increased ratios of steepwater to corn. Most researchers have used steepwater-to-corn ratios varying from 1.8:1 to 2:1 (Table I). Krochta et al (1981) reported that mill starch yield decreased from 70.3 to 67.5% when the steepwater-to-corn ratio was reduced from 2:1 to 1:1 at the same SO_2 concentration in the steepwater.

TABLE I
Steeping Variables Used in Various Laboratory-Scale Procedures

	Sample Size (g)	Steeping Solution (ml)	SO_2 Concentration (%)	Lactic Acid Concentration (%)	Temperature (°C)	Time (hr)
Static batch steeping						
Pelshenke and Lindemann (1954)	50	100	0.2	0	50	48
Steinke and Johnson (1991)	300	600	0.2	0	50	48
Eckhoff et al (1996)	100	180	0.2	0.55	52	24
Recirculated batch steeping						
Anderson (1963)	1,500	2,800	na[a]	na	na	48
Watson (1984)	na	na	0.1	na	52	48
Eckhoff and Tso (1991a)	1,500	2,800	0.2	0	50	48
Eckhoff et al (1993b)	1,000	1,867	0.2	0.55	52	36
Singh and Eckhoff (1995a)	1,000	2,000	0.15	0.55	50	24
Countercurrent Steeping						
Steinke et al (1991)	300	1,050	varies	varies	50 ± 2	48
Yaptenco (1993)	1,000	na	varies	varies	45 ± 0.2	36

[a] Not available.

Current commercial practice is to countercurrently steep corn in large stainless-steel tanks ranging in size from 3,500 to 15,000 bushels each. Laboratory countercurrent steeping apparatuses have been built to emulate the commercial system and to provide for greater control over steep variables (Watson et al 1951, Steinke et al 1991, Yaptenco 1993), but they require constant monitoring and generally a minimum of three days for the countercurrent system to achieve stability. Steinke et al (1991) considered the system to be at steady-state after ≈96 hr when the pH of the steeps did not change. Yaptenco (1993) considered steady-state to occur when the steep profiles for SO_2 and pH stabilized. Neryng and Reilly (1984) also attempted to simulate countercurrent steeping by using three interconnected steep tanks with a 160 ml/hr steepwater flow rate, when steeping ensiled corn over a 36-hr period. Laboratory countercurrent steeping allows for the study of steep parameters and process conditions not possible with batch steeping. However, because of its simplicity, batch steeping is preferred whenever the parameters being studied are not compromised by batch steep.

In countercurrent steeping, fresh corn comes in contact with low concentrations of SO_2, and as the steep progresses, the SO_2 concentration increases. The reverse is the case for batch steeping, i.e., the steeping solution is initially high in SO_2 concentration, and the concentration decreases as steeping progresses. However, Anderson (1963) found comparable milling yields between corn commercially countercurrently steeped and laboratory recirculated-batch steeped. Anderson (1963) did not report on the degree of lactic acid fermentation in the countercurrent commercial system. Roushdi et al (1979) reported that the steeping of corn by using the countercurrent system is more efficient for leaching protein from kernels than the recirculated-batch system, resulting in lower protein and higher solubles in countercurrent steeped corn. However, starch yields were similar for the two steeping systems, when lactic acid was added to the recirculated batch system.

Watson et al (1955) in an effort to approach commercial conditions developed a two-step procedure that offered advantages over batch steeping, but without the constant monitoring required by countercurrent steeping. In the first step, corn was steeped with a solution composed of 1.5% (by weight) lactic acid and 500 ppm SO_2 concentration, adjusted to pH 3.7 using potassium hydroxide. After 40 hr, the initial steeping medium was discarded and replaced with a second solution containing 0.5% lactic acid and 1,000 ppm SO_2 adjusted to pH 4.0. The corn was steeped in the second solution at 52–54°C for 8 hr with continuous circulation. Although the two-step steeping procedure was an improvement over batch steeping (one-step), it still does not emulate the dynamics of a commercial steep system.

Industrial steep times range from 24–40 hr with an industry average of 28–30 hr. Most researchers have used a steeping time of 48 hr in laboratory-scale wet milling (Table II). Eckhoff at al (1993b), using a 36-hr steeping time, reported wet-milling yields comparable to the yields of industry and other laboratory milling procedures. However, a 36-hr steeping time is inconvenient for the researcher and miller and is impractical for maintaining a daily milling regime when milling a large number of samples. Eckhoff et al (1996) obtained wet-milling yields comparable to other laboratory procedures by steeping corn for 24 hr in a static-batch mode using a 100-g sample size. However, Wang and Johnson (1992a) reported that the starch yield increased from 58.4 to 60.2% when the steeping time was increased from 24 to 48 hr. Singh and Eckhoff (1995a), who used a 24-hr steeping time by steeping 1 kg of corn in 2-L steeping solution using a recirculated-batch mode, reported lower solubles and a higher protein fraction as compared to yield data of industry and other laboratory- and pilot plant-scale procedures.

A 24-hr steep time helps maintaining a regular daily milling regime, provides a degree of stress to help identify easier-milling corn samples, and is convenient for the miller and researcher, but

TABLE II
Comparison of Industry Yield Data with Data from Laboratory-Scale and Pilot Plant-Scale Studies

	Milling Fractions (%)							
	Solubles[a]	Germ	Fiber	Starch	Protein[b]	Recovery[c]	Protein in Starch (%)	Steeping Time (hr)
Industry yield data								
Knight (1969)	6.8	8.0	9.7	68.5	6.0	99.0	0.30	36–50
Bier et al (1974)	6.5	7.8	11.2	68.0	6.5	100.0	na[d]	na
Anderson and Watson (1982)	7.5	7.5	11.5	67.5	5.8	99.8	0.35	na
May (1987)	7.0	7.9	13.0	66.0	5.7	99.6	0.3–0.35	22–50
Blanchard (1992)	6.5	7.5	12.0	68.0	5.6	99.6	0.3–0.35	30–50
Laboratory yield data: static batch								
Steinke and Johnson (1991)	7.2	6.6	19.2	58.4	8.9	100.3	0.56	48
Eckhoff et al (1996)	6.8	5.2	10.2	67.3	8.8	98.3	na	24
Laboratory yield data: static batch with intermittent recirculation								
Shandera et al (1995)	5.2	7.4	10.9	63.4	13.0	99.9	0.33	40
Laboratory yield data: recirculated batch								
Anderson (1963)	7.1	na	18.7[e]	65.4	8.1	99.3	0.54	48
Watson (1984)	7.6	7.3	9.5	63.7	11.3	99.4	0.30	48
Eckhoff and Tso (1991a)	6.2	6.0	8.8	67.3	9.8	98.1	0.32	48
Eckhoff et al (1993b)	7.0	7.0	9.9	64.8	9.9	98.6	0.32	36
Singh and Eckhoff (1995a)	3.4	6.6	11.2	62.6	15.4	99.2	0.64	24
Laboratory yield data: countercurrent								
Watson et al (1951)	7.5	7.2	8.4	62.8	11.5	97.4	0.36	na
Steinke et al (1991)	7.7	6.7	10.7	64.9	10.0	100.0	0.42	48
Yaptenco (1993)	7.0	5.8	9.1	65.9	9.5	97.3	na	36
Pilot plant-scale yield data								
Rubens (1990)	5.1	10.5	21.8	58.8	7.6	103.8	0.63	na

[a] Sum of steepwater, gluten filtrate, and other process water fractions.
[b] Sum of protein fraction, "squeegee" starch, and process water containing protein; where applicable.
[c] Sum of all milling fractions.
[d] Not available.
[e] Includes the germ fraction.

may be insufficient time for optimizing starch yields from some hybrids and for some processing conditions. Selection of a steep time can affect the ability of the procedure to discriminate between hybrids or test conditions. Longer steep times have a tendency to mask differences because the longer steep times will often compensate for poorer processing characteristics.

FIRST GRIND

The objective of first grind is to detach the germ from other corn kernel components (degermination) without damaging the germ. If the germ is damaged, it becomes more difficult to recover and may release oil into the slurry. Oil liberated during the process is absorbed by corn protein, not by starch as is often assumed, and the starch-protein separation is not affected (Watson 1964). However, the release of oil during degermination will coat process equipment and ultimately increase maintenance costs and lower yields. Simultaneous with germ release, about one-half of the starch (prime starch) is also released (Watson 1988), but the precise amount of starch released depends upon the extent of steeping and the hardness of the endosperm.

In laboratory milling, corn can be ground for degermination using a Quaker City laboratory mill, a Labconco mill, a coffee mill, or a Waring blender (Watson 1964). The most convenient method for degermination is in a Waring blender with blunt blades and operated at reduced speed with equal volumes of steeped corn and water (Watson et al 1951, Steinke and Johnson 1991, Eckhoff et al 1993b). Watson et al (1951) and Steinke and Johnson (1991) degerminated using a single blender speed (constant power setting); whereas, Eckhoff et al (1993b) degerminated using a two-step procedure where the speed of the blender (power input) was increased halfway through the first grind. The two-step degermination is believed to decrease germ damage by using less power initially (lower rpm) to break open the kernels, but because of differences in endosperm hardness, the two-step procedure may not be any better than using a single speed (constant power input). Because of differences between blenders of even the same brands, each blender used for degermination should be adjusted for speed (input power or rpm) to achieve a uniform germ recovery. The blender may be equipped with a tachometer to monitor the rpm of the blades, which can be controlled by using a variable transformer. Eckhoff et al (1996) maintained 7,500–7,600 rpm for 3 min.

Anderson (1963) and Eckhoff and Tso (1991a) ground the steeped corn in a Quaker City mill to free the germ and the hull from the rest of the kernel. With the Quaker City mill, it is difficult to adequately adjust the gap setting between the rotating plates to achieve for reproducible germ yield results. Researchers have suggested modifying the screw-gap adjustment to be more precise, but because the head is cast iron, such modification is difficult. In pilot plant-scale wet-milling of corn, Anderson (1957) and Rubens (1990) closely simulated the industrial practice by using a Bauer mill and a Foos-type mill, respectively, for the first grind. Both the Bauer mill and the Foos-type mill are attrition mills with one rotating and one stationary plate, each equipped with spiked tooth blades.

Industrial practice involves two grinds for degermination, with the second grind set at a closer mill gap spacing. The germ is removed after each grind, which reduces the damage to the germ, causing less oil to be liberated. A two-grind procedure can be easily emulated at a laboratory scale, but the manual removal of germ is very time consuming. Hence, one grind is often used in laboratory- and pilot plant-scale milling.

GERM SEPARATION

After the first grind, germ is separated from the rest of the slurry based on density difference, with the lighter germ floating on the top of the ground mash. A specific gravity range of 7.5–9° Baume of the slurry at ≈27°C is most suitable for germ separation (Anderson 1963). The floating germ can be skimmed by hand using wire screens (Watson et al 1951, Anderson 1957, Steinke and Johnson 1991, Eckhoff et al 1993b). Since the germ floats only when the slurry is in suspension, the slurry can be manually stirred intermittently (Steinke and Johnson 1991, Eckhoff et al 1993b), or a mechanical arrangement, such as a rotating paddle (Watson et al 1951) or a rotating perforated plastic disc (Shandera et al 1995), can be used at the bottom of the slurry container.

Anderson (1963) added about 200 g of starch to facilitate floatation of the germs because more water is used in the degermination step when using a Quaker City mill. Any starch added to aid germ flotation should preferably be obtained from the same hybrid. This addition complicates the process because preliminary milling runs are required. The amount of dry starch added should be accurately measured and must be subtracted from the amount of starch obtained after wet milling, to ensure correct yield mass balances. By controlling water addition in degermination, the need for added starch can be eliminated in all but cases where unique hybrid characteristics or processing parameters prevent germ flotation.

Eckhoff et al (1996) employed a shaking U.S. No. 7 sieve (2.80 mm) in the bottom of a bucket to remove germ and coarse fiber. Ling and Jackson (1991) also collected coarse fiber and germ together as overs on a U.S. No. 30 sieve (600 μm). After drying the germ and the coarse fiber fraction, the coarse fiber can be aspirated off the sieve so that amount of germ can be determined (Eckhoff et al 1996). Neryng and Reilly (1984) collected fiber and germ as the overs on a U.S. No. 40 sieve (420 μm). The overs, together with additional water, were finely ground with a mortar and pestle taking care that the germ was not damaged and to ensure that no endosperm pieces were collected in the fiber and germ fraction. The mortar and pestle grinding was in lieu of the second grind, and the ground material was again sieved through the U.S. No. 40 sieve (420 μm).

For small sample sizes, germ can be removed manually by dissecting steeped kernels. Pelshenke and Lindemann (1954), who used a sample size of 50 g, manually removed the germs from the steeped corn with a small knife. This procedure is very time consuming and should be limited to small sample sizes. It also does not yield any information regarding the potential ease of germ removal in a commercial system.

The corn wet-milling industry uses hydrocyclones, commonly referred to as germ-clones, for separating germ from the rest of the slurry. Germ-clones are commonly 152 mm in diameter and 1–1.2 m in length (Singh and Eckhoff 1995b). Rubens (1990), in an effort to closely simulate the industrial milling operation, used an industrial model, i.e., 76.2-mm (3-in.) diameter hydrocyclone (germclone), for recovering germ. The slurry was adjusted to a density of 8–9° Baume (sp. gr. 1.059–1.066). Rubens (1990) reported higher germ yields compared to industry and other laboratory- and pilot plant-scale studies (Table II). In laboratory- and pilot plant-scale milling studies, inadequate washing of the germ may leave fiber or starch attached to the germ, thus resulting in higher germ fraction.

FINAL GRIND

After the germ is recovered, the degerminated slurry is finely ground or impacted to free the remaining starch from the endosperm cellular structure and the softened protein matrix. Two types of mills can be used for grinding the slurry: "refiners" or "impact mills" (Blanchard 1992). A refiner is a modern version of the stone mill and consists of two vertical steel discs, rotating in opposite directions, with each being independently driven. Slurry is fed into the center of the disks where it is milled as it passes between the two rotating disks. An impact mill has one disc, which may be horizontal or vertical, and is fitted with a row of

rotating pins and a row of stationary pins. The slurry is accelerated by the rotating disk and is milled by impacting stationary pins and rotating pins.

On the laboratory scale, a Quaker City mill has generally been used to free starch from the gritty or unbroken particles (Watson et al 1951, Anderson 1963, Eckhoff et al 1993b). The fineness of the grind can be adjusted by increasing the plate-to-plate pressure. The operation of a tightly mated Quaker City mill is analogous to grinding with a Buhr-stone or refiner mill.

According to Eckhoff et al (1996), preparation of the disk plates in the Quaker City mill is very important. Inadequate preparation of new plates results in lower starch yields because there is insufficient contact area between the plates. New plates should be ground in place by running the mill with only cooling water passing through for ≈105 hr to ensure an adequate contact surface. It is important to periodically tighten the plates (every 8–10 hr) during the 105 hr to maintain adequate friction to wear-in the plates. The wear-in time is approximate, and comparison of yields from control corn before and after wearing-in the plates is the ultimate indicator of adequate wear-in. To ensure that sufficient wear-in of the plates has been achieved, corn samples for which yield data has been estimated using old plates, should be milled using the new plates. Since each mill grinds the plates differently due to slight variations in the shaft location, the mill used to wear-in a set of plates should be used during the plate's service life. It is recommended that a laboratory should have two mills to ensure continuous operation. About 1,000 samples can be milled with one set of plates. Plates used past their service life develop too much contact area and may slightly increase starch yields. The major disadvantage of using plates past their service life is that, as the surface area increases, the time to pass a sample through the mill increases. Eckhoff et al (1996) used a sample size of 100 g while determining the service life of the plates. The use of larger sample sizes may reduce the service life of the plates appreciably <1,000 samples.

Steinke and Johnson (1991) used a Waring blender at full speed for fine grinding and reported a high fiber fraction compared to the industrial yields and yields from other laboratory- or pilot scale procedures (Table II). However, their later study (Steinke et al 1991) used the same procedure and resulted in fiber yields comparable to the industrial yields. The degermed slurry should be finely ground before fiber removal. Inadequate grinding may leave starch or protein particles with the fiber fraction, thus resulting in higher fiber fraction yields. A blender simulates cutting action, but if a blender is used with blunt blades, it may simulate shearing or impact action. Shandera et al (1995) reversed the original blades of the Waring blender to fine grind the degermed slurry.

In pilot plant-scale wet-milling of corn, the slurry can be finely ground by passing it through a stone-mill or an impact mill, such as a Buhr-stone mill (Anderson 1957), a Rietz disintegrator (Anderson 1957), or a Foos-type mill having bar plates, in place of spiked tooth blades to increase the surface area (Rubens 1990).

FIBER SEPARATION

In industrial wet-milling facilities, fibrous material derived from the pericarp and endosperm cell walls is removed by a series of pressure-fed screens arranged to provide countercurrent washing. Before the introduction of pressure-fed screens into commercial corn wet-milling, fiber was removed by using shaker screens and reels and a split-fiber system, where fine fiber (cell walls) and coarse fiber (pericarp) were removed separately (Bier 1983). In laboratory wet-milling, the coarse fiber and the fine fiber can be separated from the slurry by screening the slurry over sieves of appropriately sized openings (Table III) (Anderson 1957, 1963; Ling and Jackson 1991; Steinke and Johnson 1991; Shandera et al 1995).

The current commercial fiber-washing system using pressure fed screens is a combined coarse and fine fiber removal system. This process of fiber separation can be simulated in the laboratory by using nylon bolting cloth or stainless-steel screens (Table III). Separation is performed much more conveniently if the cloth or the screen is attached to a shaking mechanism (Watson et al 1951, Rubens 1990, Eckhoff et al 1993b). Rubens (1990) and Eckhoff et al (1993b) used a Sweco screening device and a Kason Vibrascreen shaker, respectively, both of which are commercially available shaking machines. The use of a shaking mechanism removes

TABLE III
Sizes of Sieves, Cloth, and Screens Used for Fiber Separation in Various Laboratory-Scale and Pilot Plant-Scale Procedures

	Sieve Retaining Coarse Fiber Only	Sieve Retaining Fine Fiber Only	Screen or Cloth Retaining Both Coarse and Fine Fiber
Separate removal of coarse and fine fiber			
Anderson (1957)	26-mesh (650 μm)	200-mesh (74 μm)	...
Anderson (1963)	1 mm (1,000 μm)	200-mesh (74 μm)	...
Ling and Jackson (1991)	No. 30 (600 μm)	230-mesh (63 μm)	...
Steinke and Johnson (1991)	No. 40 (425 μm)	200-mesh (74 μm)	...
Shandera et al (1995)	No. 40 (425 μm)	230-mesh (63 μm)	...
Eckhoff at al (1996)	No. 7 (2,830 μm)	200-mesh (74 μm)	...
Combined removal of coarse and fine fiber			
Watson et al (1951)	...	...	61 μm × 71 μm
Rubens (1990)	...	...	230-mesh (63 μm)
Eckhoff et al (1993a)	...	...	200-mesh (74 μm)
Eckhoff et al (1993b)	...	...	325-mesh (44 μm)

TABLE IV
Operating Conditions for Starch-Protein Separation Using Tabling

	Table Dimensions (cm)				
	Length (cm)	Width (cm)	Pitch (cm/cm)	Specific Gravity of Mill Starch	Flow Rate to Table (ml/min)
Watson et al (1951)	579.12	5.08–10.16	0.0083	1.0585 (8° Be)	na[a]
Anderson (1963)	609.60	10.16	0.0094	1.0433 (6° Be)	300
Eckhoff et al (1993b)	610.00	10.60	0.0093	1.04–1.045 (5.5-6.2° Be)	300
Shandera et al (1995)	305.00	15.00	0.0075	1.0585 (8° Be)	150
Eckhoff et al (1996)	244.00	5.08	0.0104	1.04–1.045 (5.5–6.2° Be)	50–52

[a] Not available.

some of the subjectivity involved in hand washing of the fiber. The shaking also provides for mechanical washing of the fiber across the surface of the screen. This washing action simulates the fiber washing across the pressure-fed screen. Hand working of the fiber is a very time-consuming process. Eckhoff et al (1993a) spent ≈4 hr on fiber separation for each sample, hand working the fiber over a 200-mesh (74 μm) stainless-steel screen. Hand working of the fiber is also more subjective than mechanical sieving. Factors such as fatigue and hand pressure on the sieve surface are difficult to quantify, but they affect results.

In commercial practice, a starch-protein slurry is obtained as the underflow of the "grit screen" and the first pressure-fed screen, both these screens are 50 μm in hole-opening size. In laboratory procedures, however, the use of 50 μm screens increases the time required for separation. Eckhoff et al (1993) used 325 mesh (44 μm) screens, while most other researchers used 200 mesh (74 μm) screens.

Anderson (1963) reported a high amount of fiber fraction because the germ and fiber fractions were combined (Table II). Rubens (1990) and Steinke and Johnson (1991) reported a higher amount of fiber fraction and a lower amount of starch fraction compared to the other procedures and industrial data.

STARCH-PROTEIN SEPARATION

Mill starch consists primarily of starch and corn protein (gluten) particles that can be separated by particle density differences. In commercial practice, centrifuges are used for primary starch-protein separation and the recovered starch fraction is washed countercurrently by using a battery of hydrocyclones. Before 1950, starch tables were used to separate starch and protein (Kerr 1950, Zipf 1951, Brautlecht 1953). In laboratory-scale milling, the starch table is the most extensively used starch-protein separation method (Table IV). The heavy starch fraction settles on the table and the lighter protein fraction remains suspended in the water and flows off the end. The tables may be made of aluminum, iron (properly painted to avoid rust), or stainless steel. The appropriate width, length, and pitch of the table depends upon the concentration and flow rate of the mill starch slurry that is fed onto the table.

Singh and Eckhoff (1996) determined that a table slope of 0.0104 cm/cm and a pumping rate of 50 ml/min gave best starch yields when 1 L of mill starch slurry (1.04 specific gravity) was separated using a 8.3- × 6.1-cm aluminum table. Both starch yield and the protein content in the starch decreased with increasing table slope and pumping rate, and the rates of starch yield loss with increasing table slope and pumping rate were approximately linear.

Starch and protein can also be separated by batch centrifugation because of the greater average density of starch granules (1.5 g/cm^3) compared to the density of the protein particles (1.1 g/cm^3) (Gausman et al 1952, Biss and Cogan 1988, and Steinke and Johnson 1991). After centrifugation, the liquid on the top of the separated starch and protein layers is decanted, and the protein layer, which lays above the starch, is scraped off. More water is added to the partially cleaned starch, and the centrifuging, decanting, and scraping cycle is repeated until starch of desirable quality is obtained. Pelshenke and Lindemann (1954) used both centrifugation and tabling to remove protein from the starch. The mill starch slurry was centrifuged three times with water as described above, suspended again in water, and passed over a table to make a final separation.

Starch-protein separation can also be achieved by using 10-mm diameter hydrocyclones, which are commercially used for starch washing process (Rubens 1990, Singh and Eckhoff 1995a). However, the starch obtained by using hydrocyclones has higher levels of residual protein content when compared to the tabling method (Table V). The use of hydrocyclones at the laboratory scale for starch-protein separation needs optimization to achieve the same level of starch recovery and purity as obtained with tabling.

Centrifuges and hydrocyclones have advantages that in certain situations may make their usage more attractive than tabling. The use of a hydrocyclone, as reported by Singh and Eckhoff (1995a), increases the speed of separation because tabling requires adjusting the specific gravity of mill starch slurry and flow rate of mill starch slurry on to the table, and ambient drying before the starch can be removed from the table. Both centrifuges and hydrocyclones use less total floor space in the laboratory.

Neryng and Reilly (1984) separated starch and protein fraction using U.S. No. 200 (74 μm) and No. 270 (53 μm) standard sieves. Protein, which was recovered as the overs of both the sieves, was washed to completely separate starch. The starch was recovered as the underflow of the 270-mesh sieve. Starch particles are of the order of 10–30 μm in diameter; whereas, protein (gluten) particles are typically of the order of 5–10 μm in diameter (Singh 1994). The successful separation of starch and protein with 270-mesh sieve can not be easily explained since both starch and protein particles should pass through the 53-μm sieve. Reported starch yield values were comparable to other methods of separating starch and protein (Table V). Even though the protein content in the starch was high (1.29%), there was a reduction of the protein content from the original 8.5–9.5% protein content in the mill starch. One explanation is that the filtering of the mill starch was done statically. If the time for filtration was sufficiently long, separation between the starch and protein could occur in the Buchner funnel with the protein floating to the top, as would occur on a starch table. The starch would pass through the sieve while the stickier protein would coat the sieve screen and bind

TABLE V
Comparison of Starch Yield Data (%) of Laboratory-Scale and Pilot Plant-Scale Studies Based on the Method of Starch-Protein Separation

	Starch Yield	Protein Yield[a]	Protein in Starch
Tabling method			
Watson et al (1951)	62.8	11.5	0.36
Anderson (1963)	65.4	8.1	0.54
Watson (1984)	63.7	11.3	0.30
Eckhoff and Tso (1991a)	67.3	9.8	0.32
Eckhoff et al (1993b)	64.8	9.9	0.32
Centrifugation method			
Steinke and Johnson (1991)	58.4	8.9	0.56
Steinke et al (1991)	64.9	10.0	0.42
Centrifuge washing followed by tabling method			
Pelshenke and Lindemann (1954)	68.5	6.5	0.42
Hydrocyclone method			
Rubens (1990)	58.8	7.6	0.63
Singh and Eckhoff (1995a)	62.6	15.4	0.64
Sieving Method			
Neryng and Reilly (1984)	54.7	14.7	1.29

[a] Sum of protein fraction, "squeegee" starch, and process water containing protein; where applicable.

TABLE VI
Standard Deviation of Starch Yield Measurement Reported for Various Laboratory-Scale Studies

Study	Standard Deviation (%)
Anderson (1963)	0.50
Neryng and Reilly (1984)	2.00
Steinke and Johnson (1991)	0.70
Steinke et al (1991)	0.40
Eckhoff et al (1993)	0.97[a]
Wehling et al (1993)	2.52
Singh and Eckhoff (1995a)	0.55
Shandera et al (1995)	1.02[a]
Eckhoff et al (1996)	0.40

[a] Estimation based on reported coefficient of variation and mean starch yield.

with fiber that had not been adequately separated. Corn gluten is also known to agglomerate under certain conditions. This also would account for the protein being retained by the sieve.

The laboratory-milling studies that used a starch table for starch-protein separation (Watson et al 1951, Anderson 1963, Watson 1984, Eckhoff and Tso 1991a, Eckhoff et al 1993b) reported 0.30–0.54% residual protein content in starch (Table V). Steinke and Johnson (1991) and Steinke et al (1991), using sedimentation and centrifugation for starch-protein separation reported 0.56 and 0.42% residual protein content in starch, respectively. Rubens (1990) and Singh and Eckhoff (1995a), who used hydrocyclones for starch-protein separation to emulate the industrial process, reported 0.63–0.64% residual protein content in starch. Rubens (1990) used four stages of hydrocyclones for starch-protein separation, and Singh and Eckhoff (1995a) used a five-pass washing system; whereas, the industry uses 8–14 stages of hydrocylones. However, there has not been the same level of effort expended in developing the centrifuge or hydrocyclone methods for laboratory use.

MASS BALANCE AND RECOVERY

A recovery of 98% is generally achievable (Table II), regardless of the method used, if the samples are carefully milled. Low recovery of total solids presents a problem particularly if the total recovery varies between test conditions. It is then difficult to ensure that the differences observed in the experiment are due to test conditions and not due to lost solids. Adjustments in milling technique will usually correct mass balance variability. Recoveries greatly over 100% imply that either the samples were not dried sufficiently or the milling equipment was not adequately cleaned. If the variation in total mass recovery is small, the test data should be acceptable even if results are over 100%, since it implies a systematic error in the methods used or in the calculations of the mass. Such systematic errors should be investigated and corrected.

REPRODUCIBILITY AND ACCURACY

A low coefficient of variation or standard deviation of the milled fraction yields of replicated runs indicates that the procedure is reproducible. Starch yield, being the most important fraction, should have as low a standard deviation as possible for the replicated runs. A standard deviation of ≈0.5% is required to delineate starch yields differing by 1% (Eckhoff et al 1996). Reported standard deviation of starch yield varies from 0.40 to 2.52% (Table VI). A procedure is accurate if the milled fraction yields are comparable to the yields from industry and other laboratory- or pilot plant-scale studies. The procedure should respond to changes in hybrids or corn quality or processing factors in a manner similar to that of industrial facilities.

CONCLUSIONS

Various methods have been developed for laboratory- and pilot plant-scale wet milling that give starch yield and protein content in starch similar to those of industrial practice. Selection of a procedure and the scale of milling depends upon the availability of equipment, test objectives, and the amounts of products required for subsequent evaluation. The data available suggests that any of the laboratory milling procedures previously used can achieve accurate and reproducible yields if sufficient care is taken by the researcher while milling. Details that need attention and should be reported for any milling procedure (as applicable) include: make and model of all equipment, the amount of water used in each step of the milling process including clean-up, steep temperature, lactic acid and SO_2 concentration in steepwater, ratio of corn to steepwater, steepwater recirculation rate, specific gravity or Baume at germ skimming, screen size used in germ skimming, grinding mill operational parameters (gap, rpm, time of operation), screen size used for fiber washing, specific gravity or Baume of mill starch slurry, flow rate on to starch table, length and width of starch table, slope of starch table, centrifuge rpm, hydrocyclone operational pressure and flow rate, number of centrifugations or hydrocyclone passes, protein (gluten) filtration paper pore size, and vacuum pressure. Subjective procedures such as the techniques for germ skimming, fiber-washing method, and hand-washing of the starch on the starch table should be recorded on video tape or described in sufficient written detail for easy reproduction by other researchers.

A suitable laboratory procedure will use batch steeping with recirculation of steepwater, a speed-controlled blender with dulled blades for first grind, hand-skimming of the germ, fine grinding using a disk mill, fiber separation using a reciprocating screen, and starch table for starch-protein separation. Use of a hydrocyclone or centrifuge rather than tabling will require further optimization to achieve both high starch yield and low residual protein content in starch. Pilot plant-scale procedures have been less studied than the laboratory-scale methods, with only two procedures reported. A pilot plant-scale procedure should include batch steeping, disk milling for all three grinds, germ recovery using germ-clones, fiber washing using pressure-fed screens, and starch-protein separation using hydrocyclones. Use of a small centrifuge for starch-protein separation is also possible.

Further efforts need to be expended for the refinement of the use of centrifuges and hydrocyclones for starch-protein separation and the use of countercurrent steeping procedures. Development of a standardized method for laboratory- and pilot plant-scale wet-milling of corn may be beneficial and is highly recommended.

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