A Laboratory Countercurrent Steep Battery for Corn Wet-Milling

K. F. YAPTENCO,[1] E. J. FOX,[2] and S. R. ECKHOFF[3]

ABSTRACT

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A laboratory-scale steep battery was designed to serve as a research tool for studying continuous countercurrent steeping as performed in corn wet-milling plants. The steep battery consisted of 16 tanks (12 active; 4 for loading and unloading) each of approximately 1 gallon capacity designed to hold 1 kg of corn and associated steepwater. Sequencing of water flow through the 12 active tanks was accomplished via computer control and differed from previous laboratory steep batteries in that it had the capability to recycle steepwater within individual tanks. The system was designed to accommodate a wide range of steep times, steep temperatures, evaporator draw rates, and individual tank steepwater recycle rates. Steepwater profiles for total solids, pH, sulfur dioxide, and total acidity were measured and found to be comparable to industrial profiles reported in the scientific literature, if adjusted to compensate for incoming solids with industrial steepwater. Product yields for corn steeped in the laboratory steep battery were statistically similar to batch-steeped corn fractionated using the same milling procedure. The product yields were also comparable to values found by other researchers using laboratory countercurrent steeping. Operation of the steep battery required continuous monitoring due to potential for breakdown (sticking of floats, pump tubing failure, and electrical failure). When preventative maintenance was used, the system could be operated with few disruptions. The laboratory steep battery can potentially be used to study many of the design parameters of conventional steep systems, as well as to explore alternative water routing schemes. It can also be used to study the effect of steeping chemicals, enzyme adjuncts, or corn quality.

Wet milling is the largest nonfeed use of corn, consuming about 13% of the 1991 U.S. corn crop. Corn wet-milling fractionates kernels into four basic components: starch, germ, fiber, and protein. The primary product, starch, is recovered in purified form with a yield of 67–69% of the initial dry corn matter and a recovery efficiency of 93–96% of the total starch content of corn (Watson 1986, 1977). Close to one-fourth of the corn starch produced is sold as modified or unmodified starch products for the food or paper industries, with the remainder being processed into hydrolyzed products including corn syrup, fructose, and dextrose, or fermented to ethanol or other fermentation chemicals.

Steeping is the first step in corn wet-milling where the corn is soaked 20–40 hr in a process water containing sulfur dioxide. Steeping induces chemical and biochemical reactions inside the kernel leading to partial dissolution of the endosperm protein matrix and ultimately to release of the starch granules. During milling, process, water containing 0.5–1.5% soluble solids (resulting from the downstream washing of the starch, fiber, and protein fractions) is dosed with 0.1–0.2% SO_2 and continuously pumped into the steep tanks in a countercurrent manner, allowing the corn to steep for 20–35 hr or more at 48–52°C (Watson et al 1955, Watson 1977, Blanchard 1992). During steeping, SO_2 disrupts the protein matrix that encapsulates starch granules by breaking inter- and intramolecular disulfide bonds, thus making the physical separation of starch and protein easier. The SO_2 also activates endogenous protease activity in the endosperm, which helps solubilize the protein matrix (Wahl 1969). Fermentation producing lactic acid also occurs in countercurrent steeping, which enhances separation. Fresh corn entering the steep battery is exposed to low (20–200 ppm) levels of SO_2. The naturally occurring *Lactobacillus* spp. can propagate at these levels of SO_2 and will consume solubles that leach into the steepwater directly from the steeped corn or enter with the recycling of the process water (Watson 1984, Blanchard 1992).

In current industrial practice, a steep battery consists of a series of 8–16 stainless steel tanks (although this number may reach 50) connected to each other in series (Blanchard 1992). Each tank may have a capacity of 76–382 tons of corn (3,000 to 15,000 bu) and is fitted with piping and a pump to move steepwater from tank to tank, pass steepwater through a heat exchanger, and drain steepwater from the system.

Monitoring of the steepwater solids content, SO_2 concentration, pH, and total acidity is performed in industry by withdrawing samples from each tank. Each parameter is plotted with respect to the tank number (relative location of tank in process) to produce a characteristic profile. Ranges of these characteristics that give the best milling results are based on the experience of the operator (Watson et al 1951) and upon production constraints (Blanchard 1992). Results of the analyses are used to control the draw rate (the rate at which steepwater is drawn from the steep battery), incoming SO_2 levels, and temperature so that steepwater drained at the end of the steep battery (light steepwater drawn off the tank with the newest corn) is kept at pH 3.9–4.1, total acidity corresponding to 1.0–1.5% lactic acid, and 0.01–0.02% SO_2 (Watson et al 1951). Soluble solids levels in the steepwater at the last tank (tank with the newest corn) may reach 5–7% (Watson 1977).

Most studies on steeping used batch-steeping methods that are easy to use but do no simulate the continuous countercurrent nature of an actual battery. Experiments in wet mills or large-scale pilot plants are difficult to perform because variations in corn properties and mill house water composition render results unreliable (Watson et al 1951). These problems have led some researchers to design laboratory-scale models of steeping batteries that can perform continuous countercurrent steeping (Watson et al 1951, Steinke et al 1991). However, these models did not fully achieve industry conditions due to design limitations.

Watson et al (1951) described a countercurrent laboratory-scale steeping battery made of 12 glass jars each holding about 400 g. Water entered each jar at the bottom and overflowed into the next steep by intermittent application of a vacuum at the draw end (end of steep battery where steepwater is being drawn off) to create a batch advance of steepwater. A water bath was used to control steep temperature. The disadvantage of this experimental

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[1] Former graduate assistant, Department of Agricultural Engineering, University of Illinois. Current address: Department of Agricultural Engineering, University of Philippines, Los Banos, Philippines.

[2] Former graduate fellow, Department of Agricultural Engineering, University of Illinois. Current address: Cargill Corn Milling Division, 3201 Needmore Rd., Dayton, OH 45414.

[3] Professor, Department of Agricultural Engineering, University of Illinois. Corresponding author. Fax: 217/244-4022.

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design was its static batch advanced method of moving water, rather than the continuous system favored by most of the industry (Steinke et al 1991). The steeping solution was not recirculated within the jars which limited mixing.

Steinke et al (1991) designed another system using a battery of nine 1,200-ml jacketed glass vessels; six vessels at a time were used for steeping. Steeping solution entered the system at the top of the first flask and moved down through the grain until the water overflowed into the next flask. Steepwater was moved through the system by pressure from two peristaltic pumps supplying fresh steepwater and draining old steepwater. Temperature was maintained at 50 ± 2°C by pumping hot water through the vessel jackets and a hot water bath. A disadvantage of the Steinke steeping system is the lack of ability to recycle the steepwater with in a steep tank. This problem is the same as with Watson et al (1951) in that the interstitial velocity (velocity of the fluid between kernels) in the tanks is controlled by the draw rate (the rate of steepwater leaving the steep battery) rather than by a combination of the draw rate, and the recirculation rate as occurs in industry. The interstitial velocity controls both the external mass transfer and external heat transfer rates, which may limit the rate of steeping. When there is no recirculation, the interstitial velocity is decreased, which may impede completion of steeping. The contribution of the recirculation rate to the mass transfer rate depends upon the magnitude of the draw rate, the design of the steep tanks, and the magnitude of the recirculation rate. In industrial practice, the recirculation rate may be several hundred times the draw rate, which means that interstitial velocities could be several hundred times larger in systems with recirculation than in systems without recirculation.

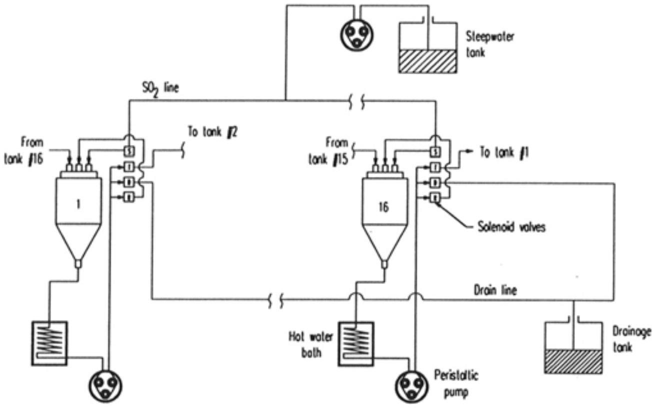

Fig. 1. Schematic diagram of the steep battery.

The objective of this study was to describe the design and basic operation of an improved laboratory-scale steep battery. Representative steep profiles were generated to demonstrate the similarity of the laboratory-scale steep battery to industrial steeping. Test analysis involved determining steepwater characteristics, determining milling yields of steeped corn, and evaluating tank temperature and pump flow rate stability.

MATERIALS AND METHODS

System Description

The steep battery consisted of 16 plastic tanks connected in series (Fig. 1 and 2) assembled from 4-L plastic bottles and 140-mm dia. plastic funnels (Fig. 3). These tanks are the same as constructed and used for batch steeping by Eckhoff et al. (1993). Twelve tanks were active (steepwater flowing through them) at any given time. The remaining four tanks were used for purposes of allowing a buffer so that the system could be left unattended for up to four times the time required to sequence through one tank. For a 36-hr steep time, every 3 hr a steep tank containing dry corn would be added to the active steep battery and a steep tank containing fully steeped corn would be dropped from the active steep battery. The four nonactive tanks could be filled with 1 kg of dry corn each, and the computer would sequence them into the active cycle while dropping out the appropriate tank containing fully steeped corn, allowing for 12 hr of unattended operation.

The tanks were mounted on a steel frame constructed of 25- × 25- × 0.5-mm (1- × 1- × 1/8-in.) angle iron 25- × 25- × 0.5-mm square tubing. The total length of the steep battery was 2- × 0.8- × 1.4-m. The frame was covered at two levels with 12.5-mm (1/2 in.) plywood. Each steep tank was connected to the frame by use of a steel collar mounted on vertically placed square tubing and equipped with four solenoid valves (Fig. 4). One normally closed valve is used for introducing fresh steepwater (S) in the steep tank, one is used for draining (D) the steep tank, and one is used for transferring (T) steepwater to the next tank. The fourth solenoid valve is a normally open valve and is used for controlling the recirculation of steepwater. Each valve was powered with 115 VAC and required ≈10 W each to activate.

Four 4-channel variable speed Cole-Parmer peristaltic cartridge pumps were used as recirculation and transfer pumps, with each pump handling pumping for four adjacent steep tanks. Pump speed could be varied between 6 and 600 rpm. A single-channel single speed (60 rpm) Cole-Parmer peristaltic pump was used for

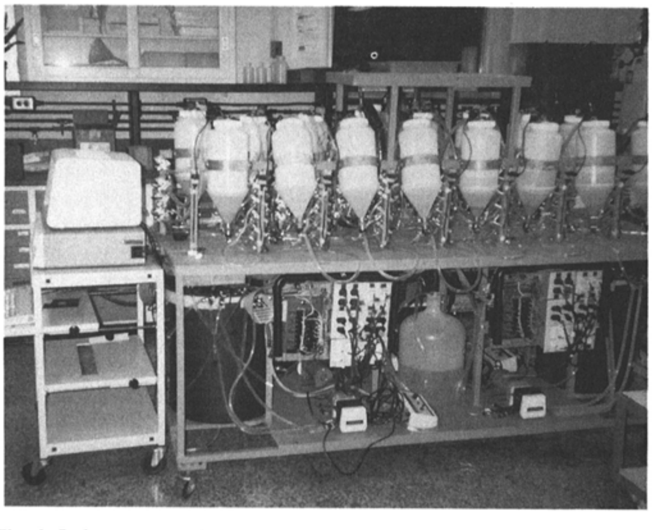

Fig. 2. Laboratory-scale steep battery.

Fig. 3. Assembled steep tanks.

pumping in fresh steepwater based upon demand from a 50-L storage tank into a common Tygon tube line connected to all the S-valves of the battery. Old steepwater was removed from the system through another common line connecting all the D-valves. All pumps operated on 115 VAC.

Water levels in each tank were maintained using an interfaced personal computer (IBM XT) that monitored an upper limit switch mounted on the top of each tank and activated the proper combination of valves to keep corn covered with steepwater. A homemade float valve, fashioned from a plastic funnel and a plastic Petri dish, was placed in each tank. The float was made airtight and would float on the surface of the steepwater in the tank. A glass rod attached to the plastic funnel rose with rising water in the tank and triggered the upper limit switch. The upper limit switch was a phototransistor sensor which consisted of a light-emitting diode on one side of the sensor and a light-sensitive transistor on the other side. When the water level was high, the float switch would block the light and cause a signal line to carry a logic "high" signal to the computer for processing. When the water level was low, the float switch would sink and the light would hit the transistor sending a logic "low" signal to the computer for processing.

Temperature was maintained in each tank by recirculating steep water through Tygon tube coils submerged inside a water bath. Each steep tank required ≈4.3 m of tubing to ensure adequate heat transfer surface area. Two water baths, fashioned from 120-L plastic tanks and heated with a thermostatically controlled 115 VAC 1.5 kW electric immersion heater, were used with each waterbath containing the recirculation coils for eight steep tanks. The temperature in each steep tank was measured, and the amount of tubing in the tank was adjusted, based upon preliminary testing, to provide for uniform tank temperatures.

The steep tank was connected to the computer by a parallel digital input/output (I/O) card (Keithly Metrabyte Corp., Taunton, MA). The card consisted of four industry-standard 8255 programmable peripheral interface chips. Each chip had three ports that could be programmed individually for data input, output, or a combination of both. The computer program to operate the steep tank was programmed in the BASIC language. Details of the program can be found in Yaptenco (1993).

The steeping sequence followed the "pull" system, which is currently being used in most commercial wet mills, where the draw to the steepwater evaporators is set at a constant rate and water is pulled into the steeps. In the pull system, the amount of steepwater being evaporated is always maximized, with water pulled from one steep tank into the next by lower limit switches in the tanks that attempt to keep the corn covered with steepwater at all times. At a fixed evaporator capacity, the fresh steepwater flow into the steeps varies according to the equilibrium moisture content of the steeped corn. The steep battery can also be operated as a "push" system, where water is pushed into the steep battery at a constant rate, and limit switches in each tank are used to push the steepwater toward the evaporator by some minor hard wire changes. The pull system is preferred in industry because an inoperative limit switch results in no water being transferred, while the push system with an inoperative limit switch results in tank overflow.

Each steep tank operates in one of five modes. When no steepwater is being added into or taken out of the steep tank, the system is in the recirculation mode. In the recirculation mode, the steepwater is pumped through the waterbath tubing and back into the same steep tank.

The second mode of operation is when the next steep tank (the one with the next newest corn) is low on water. In the transfer mode, the T-valve opens and allows the water that had been recirculating to be transferred to the next steep tank after it is passed through the waterbath. This transfer of water continues until the target tank water level reaches the upper limit switch. When this occurs, the original steep tank returns to the recirculation mode by closing the T-valve. When sufficient water has been transferred from the original steep tank and the float valve registers a low water level, then the T-valve is opened on the previous tank (the one with the next oldest corn) and steepwater is pumped from that tank into the original steep tank. The percent time the steep is in the recirculation mode depends upon the pumping rate of the recirculation pumps and the draw rate.

The third and fourth modes of operation are used when the original steep tank is on either end of the steep battery. When the steep tank has just been filled with dry corn, it is brought into the steeping system by wholesale transferring of water from the previous tank. Once the original tank is full, the D-valve opens and steepwater is drawn from the tank to the evaporator (the draw). In the laboratory steep battery, water is drawn out at specific intervals (four times per hour) rather than on a continuous basis. This is to avoid having an additional draw pump with each steep tank, as the transfer pump also serves as the draw pump. The fourth mode occurs when the original steep tank contains the oldest corn in the steep battery. This corn is to be exposed to the fresh steepwater solution. In this case, when the float switch indicates a low level in the original tank, the S-valve opens and fresh steepwater is pumped from the 50-L storage tank. The fifth mode of operation is when the tank is to be taken out the active steep battery. During this period, the steepwater is drained out of the tank without any fresh steepwater being added. The fifth period occurs at approximately the same time that a new tank is being added into the steep battery. Wholesale continuous movement of water occurs from one tank to the next. Once the tank is empty, the corn can be removed for milling or discarded. Fresh dry corn is added into the steep while it awaits being cycled back into the active steep battery.

System Evaluation

Three separate runs were used for the evaluation. The first run was used for generating profiles of steepwater characteristics, determining milling yields, and observing water levels. The second and third runs were used for observing the stability of pump flow rates and tank temperatures, respectively. Stability of the system was measured by comparing sequential profiles over a period of time.

Corn of a blend of unknown commercial hybrids from a local feed mill was used in the first run. The corn was cleaned using a Carter-Day dockage tester with a 6.0-mm (15/64 in.) round hole screen to remove fine material and broken kernels. Moisture content was determined by using a standard oven method (AACC

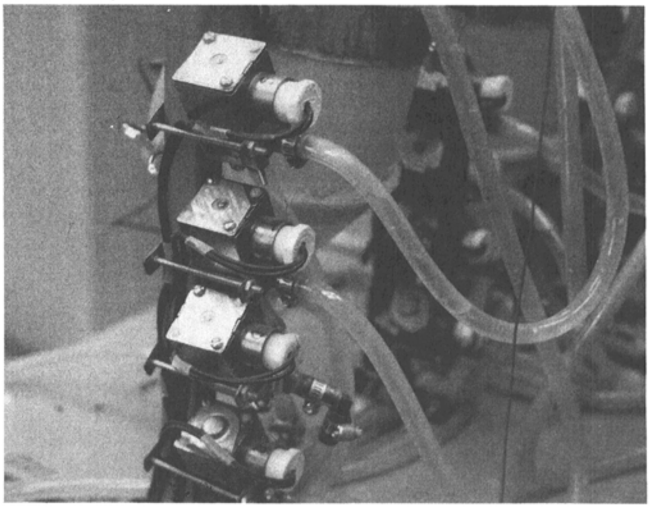

Fig. 4. Solenoid valves.

1983). Test weight was determined using a Dickey-john grain analysis computer. Average moisture content was 14.4% wet basis, while average test weight was 26.2 kg (57.7 lb.) per bushel after cleaning. For the second and third runs, a blend of unknown commercial hybrids, different from that used in the first test, was used with a moisture content of 14.0%.

In all three tests, 19 L of steep water taken from the end tank (newest corn) of a local wet mill was used to initially inoculate the battery with *Lactobacilli* bacteria. The light steepwater was put into all 12 of the active tanks during start-up. Fresh SO_2 solution was then drawn into the steeps on a demand basis depending upon the draw rate and the rate of water absorption by the corn. This ensured the maximum time for the establishment of a good fermentation in the steep system. A preliminary test showed that starting the system with fresh water did not result in an appropriate fermentation. The operating conditions used for the first run were: 0.15% incoming SO_2 level; 36-hr steep time; 12 tanks; 677.3 ml/kg draw rate; 1,000 ± 0.2 g (wb) of corn per tank; 52°C tank temperature; 300 ml/min recirculation rate.

The same operating conditions were used for runs two and three, except that the draw rate was set at 400 ml/kg and steep temperature was set at 46°C. The second and third runs were part of another series of experiments designed to study the effect of lower steep temperature and are reported as part of this study because they provided for more comprehensive testing of steep temperature and flow rate stability than were measured in run one.

Steepwater Characteristics

Steepwater profiles for SO_2 concentration, total solids content, pH, and total acidity were used as the basis for determining when the system reached steady state. Steady state occurred when the profiles for two consecutive days were the same. Samples (10 ml) for each variable were gathered every two days. Only one sample per variable per day was gathered to avoid excessively disturbing the system; 24 hr was allowed to elapse between each sampling for the same reason.

Samples for the SO_2 profile were analyzed by iodine titration with results expressed in parts per million (ppm) (Eckhoff et al 1993). Samples for pH were stirred and analyzed with a digital pH meter; total acidity was determined by titrating the sample with 0.1*N* NaOH to pH 8.3 (Corn Refiners Association 1991). Results were expressed as percent lactic acid using a correlation developed by a wet-milling company that multiplies the volume of NaOH used (in ml) by 0.9 (calibration based on high-performance liquid chromatography analysis of steepwater) and dividing the product by the sample weight (in g). Lactic acid concentration of steepwater was calculated by multiplying the total steepwater solids level by percent lactic acid. Samples for solids content were placed in preweighed aluminum cups and dried in a 49°C forced-air oven for 24 hr, with a final drying step at 103°C for 5 hr (Eckhoff et al 1993).

A profile of each variable was generated using data starting from the first day each profile stabilized. Confidence intervals for the mean at the 95% level of significance were generated for each data point for comparison to industrial wet-mill profiles to provide a rough picture of the system's performance. Differences in corn and steep battery characteristics exist, but the comparisons provide a basis for evaluating the performance of the steep battery.

System Stability

For the second run, the pump flow rates were adjusted to 5 ml/sec based upon previous calibrations. Actual flow rates were then monitored by pumping water into a 250-ml graduated cylinder and determining the time to reach a certain volume with a stopwatch. Two readings per tank were taken and the average time and flow rate calculated. The overall mean and a 95% confidence interval for all 16 tanks was calculated.

The third run, used for observing tank temperatures, had a nominal temperature set at 46°C and monitored at hourly intervals for three days. The temperature was set at 46°C. A mercury-in-glass thermometer was used for monitoring temperature. The overall mean and a 95% confidence interval for all 16 tanks was calculated.

Changes in water levels were monitored by connecting a vertical plastic tube to the outlet of a tank. Water levels were marked off on a strip of tape placed next to the tube. Three tanks were monitored randomly for several days with results expressed as the difference between the highest and lowest levels recorded.

Mass Balance Analysis

The draw rate was monitored during the first run by collecting water from the drain valve of whatever tank was draining at the time. The volume was recorded after each transfer; three readings were performed. Mass balance calculations were used for determining inflow rate of fresh steepwater and water absorption rate of corn during the first run. Uncertainty analysis was performed to generate confidence intervals at the 95% level of significance.

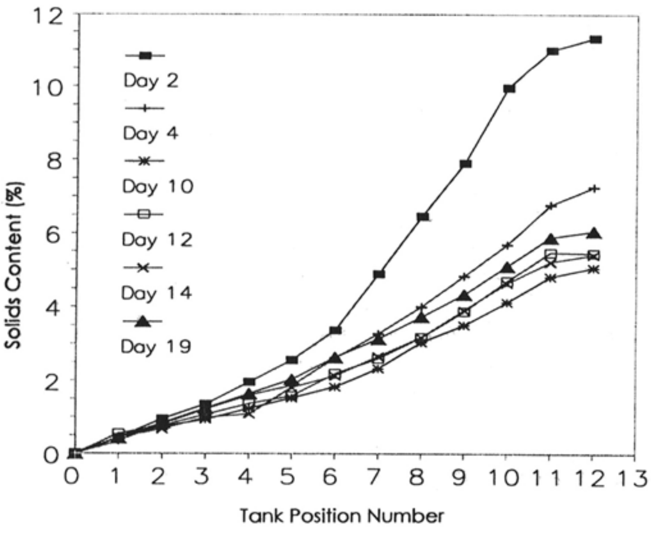

Fig. 5. Solids profile during start-up.

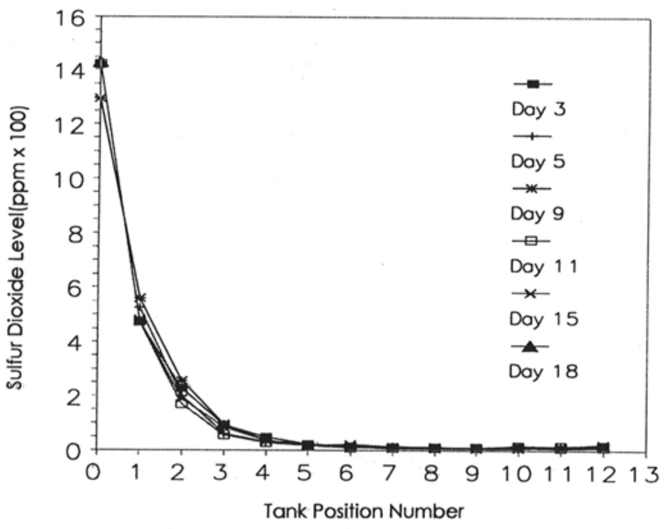

Fig. 6. SO_2 profile during start-up.

Wet Milling Yields

Countercurrent steeped corn from the first run was milled using a standard laboratory procedure (Eckhoff et al 1993). Three replicates were conducted for this steeping method. For use as a control, two 1-kg samples of the same corn were batch-steeped at 52°C and 36 hr and milled for comparison. SO_2 concentration was set at 0.15% and lactic acid concentration at 0.55%.

RESULTS AND DISCUSSION

Steepwater Characteristics

Solids and acidity profiles stabilized after the ninth day of operation, while pH and SO_2 needed only two days to stabilize (Figs. 5–8). Time to reach steady state was observed in subsequent tests to be three to four days, but the steep battery experienced some leaks and malfunctions for the first three days of steeping, which slowed time to achieve steady state. In Figure 5, data from day 2 represents some residual effects from the use of the light steepwater from the wet-milling plant. With time, the additional solubles brought into the steep battery by the light steepwater are leached out and only solubles created during steeping are in the system. The same is true for the high initial lactic acid content in Figure 8.

The solids profile at steady state (Fig. 9) showed consistently lower levels of dissolved solids when compared to those of the commercial plant observed by Watson (1984) due to the absence of soluble and suspended solids in the fresh steeping solution used in the study. Watson's data is the only published data on a steepwater profile of its type found in the scientific literature. The data is reported as a function of the tank location number. Because each tank is continuously changing its relative position in the steeping process, the tank location number represents the position of a tank in the system rather than the actual tank number. Tank position number 0 represents the incoming steepwater and tank position number 12 represents the tank with the newest corn. Process water for the wet-milled samples of Watson (1984) were introduced with a solids content at 1.5% and exited the system slightly below 6.5%. In comparison, steepwater entered the laboratory-scale battery with no dissolved solids and exited with 5.5% solids content.

Replotting the battery solids profile with an offset value of 1.5% (Fig. 9) to each data point to simulate the use of process water showed the profile of the wet-milling falling within the confidence intervals from tanks 2 to 6; tank 1, however, fell outside the interval. The use of the confidence intervals on the laboratory steep battery profile is not for statistical comparison to Watson's data but to illustrate the level of variability in the steep battery data. From tanks 7 to 10, the wet-milling profile started to differ markedly from that of the laboratory battery, but still remained inside the confidence intervals. This indicated that the use of distilled water appeared to allow for adequate growth of lactic acid bacteria. The smell of the steep tanks also indicated that an appropriate fermentation was occurring. The maximum difference between the two profiles without the offset value was 1.82% solids. With the offset, this decreased to 0.32% solids. There are many factors that could affect the level of steepwater solids, including corn hybrid, growing environment, drying conditions, and the total water usage (L/kg of corn) and similarity between the steep battery data and Watson's data does not validate the laboratory steep battery, but it indicates that the results achieved by the laboratory steep battery are reasonable and within the range of values expected for a commercial system.

The shape of the SO_2 profile for the commercial plant (Fig. 10) showed levels gradually decreasing as water moved through the system, with incoming concentration at around 1,300 ppm and final concentration at the drain tank down to about 80 ppm. In comparison, the battery profile (Fig. 10) at steady state showed incoming steepwater at 1,382 ppm, which dropped down rapidly

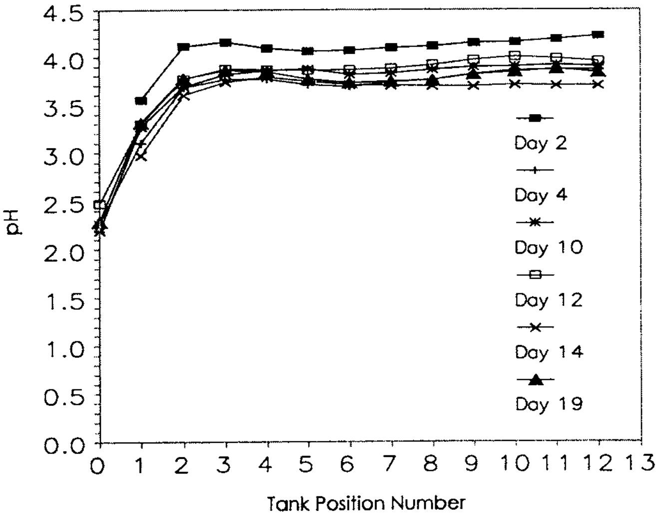

Fig. 7. Profile of pH during start-up.

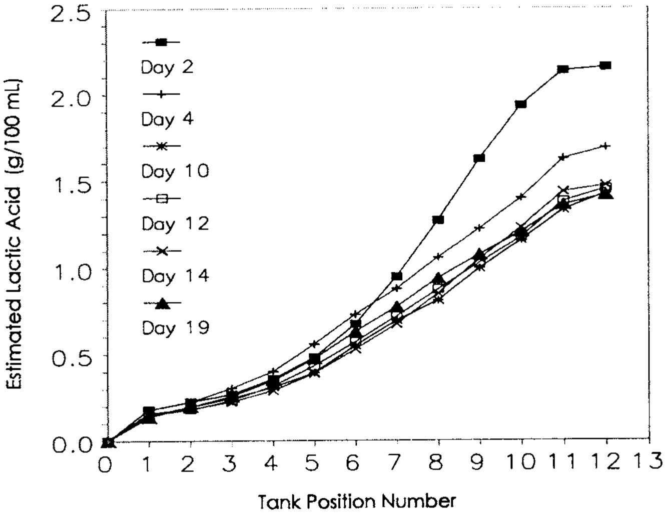

Fig. 8. Lactic acid profile during start-up.

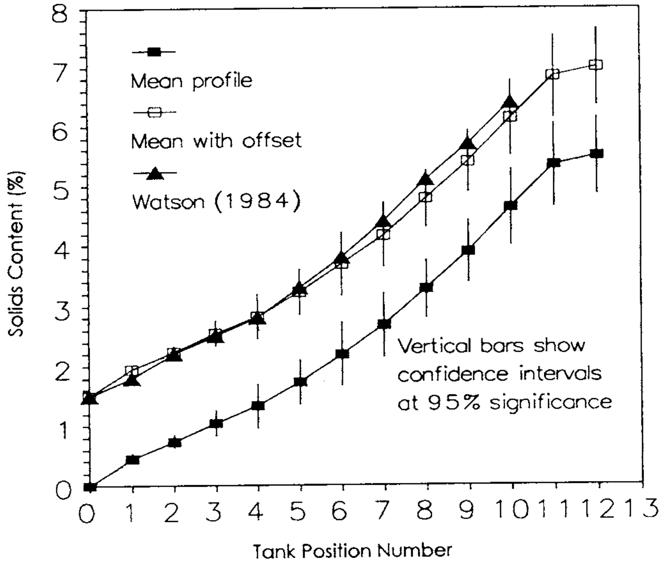

Fig. 9. Solids profile at steady-state.

to about 77 ppm after the first three tanks, yet before slowing its rate of decrease over the remaining tanks. Concentrations in the remaining tanks were all less than 50 ppm. The rapid drop in SO_2 levels from tank to tank could be due to the time samples were taken, as sampling was performed toward the end of a recirculation cycle. This allowed SO_2 to diffuse into the corn kernels and altered the shape of the profile. Experience in our laboratory showed that, in batch steeping, the final SO_2 level can range from 200 to 900 ppm, depending upon the corn hybrid studied.

The pH profiles of the milling plant and the battery were similar (Fig. 11). For the plant, pH of incoming steepwater was ≈3.5; after the first tank of the battery, pH rose and stabilized between 4.0 and 4.1. For the laboratory steep battery, pH started at 2.3 and stabilized after the first tank at 3.8 (mean of pH values from tank positions 2 to 12 during steady state). The wet-milling profile did not fall inside any of the confidence intervals of the laboratory steep battery. The maximum difference between the two profiles (considering tank positions 2 to 10) was 0.34. The process water used for steepwater in an industrial facility contains 1–2% solubles and may buffer the steepwater to raise the pH.

Steepwater in the commercial plant (Fig. 12) started with about 0.6 g of lactic acid per 100 ml of steepwater and left at 1.6 g/100 ml. Battery water, on the other hand, entered with no lactic acid and exited at 1.6 g/100 ml (Fig. 12). Watson (1984) showed a profile with two distinct linear portions. The first portion, spanning tank positions 1 to 4 and corresponding to a stage of steeping characterized by SO_2 diffusion and protein breakdown (Watson et al 1951), had a slight upward slope. This slope increased in the second portion spanned by tank positions 5 to 10, where SO_2 concentration had dropped below 400 ppm. The second larger slope indicated increasing bacterial activity due to the lower levels of SO_2 in the steepwater. The estimated lactic acid profiles of the laboratory steep battery also showed a similar shape. Linear regression analysis showed a fairly linear curve spanning tanks 1 to 4 with a slope of 0.05 g/100 ml between tanks ($r^2 = 0.98$). Tanks 5 to 11 were spanned by a steeper linear curve with a slope of 0.15 g/100 ml ($r^2 = 0.99$). Acidity between tank 11 and 12 did not increase as much as previously observed (slope = 0.06 g/100 ml) and may indicate the limit of activity that can be sustained by the bacteria. Increasing the battery length could possibly result in a profile with acidity leveling off after 10 or more tanks.

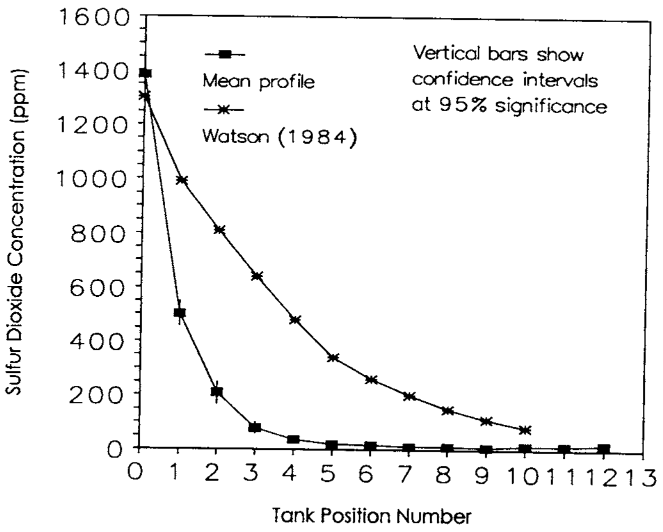

Fig. 10. SO_2 profile at steady-state.

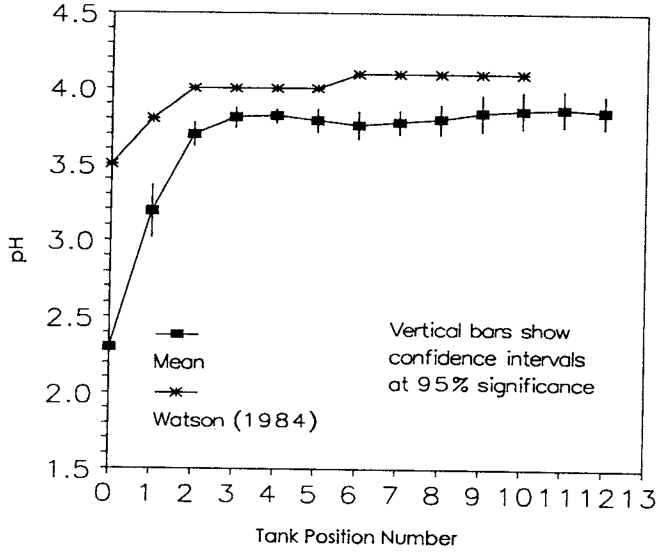

Fig. 11. Profile of pH at steady-state

Replotting the battery acidity profile with an offset value of 0.6 g/100 ml (Fig. 12) showed the wet-milling profile falling outside all confidence intervals, except for tanks 5 to 7. However, the offset greatly improved the comparison between the two sets of data. The maximum difference in profiles without the offset was 0.6 g/100 ml. With the offset, this decreased to 0.2 g/100 ml. Watson (1984) used total acidity to estimate lactic acid, as did the current study, although the relationship between total acidity and lactic acid Watson used is not known.

System Stability

Mean pump flow rate for the battery during the second run was 4.5 ml/sec with a standard deviation of 0.5 ml/sec. Variation in the pump rate changes the interstitial velocity and, thus, the external mass transfer rate between the steepwater and the corn surface. Generally the mass transfer rate (diffusion) in steeping is limited by the diffusivity of the kernel, so moderate variation in steepwater velocity may not be important. Individual pump rates are important when they fall below the draw rate because corn in steep tanks may become uncovered. Variation in pump flow rates could be due to accidental resetting of speed controllers, new pump tubes, changed gap settings in pump cartridges, accumulation of solids in the tubing, or air pockets in the heat exchanger coils in the hot waterbath.

Mean tank temperature for the battery during the third run was 45.0 ± 0.2°C (95% confidence level). Standard deviation was 1.8°C. Instances when tank temperature differed significantly

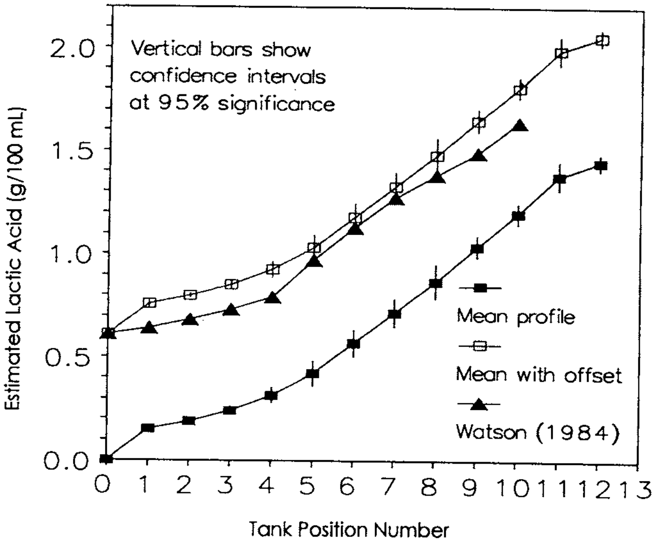

Fig. 12. Lactic acid profile at steady-state.

TABLE I
Steepwater Flow Rates During Steeping

	Steep Tank Value[a]	Reported Value[b]
Draw rate, L/min	677 ± 76	596 - 894
Corn absorption rate, L/min	511 ± 40	500
Fresh steepwater inflow rate, L/min	1,149 ± 84	1,200 - 1400

[a] Confidence interval at 95% level of significance.
[b] Watson 1984 and May 1987.

from the preset temperature occurred during the first half hour of each 3-hr recirculation cycle, when dry corn or fresh steepwater at room temperature were still heating up. Temperature of the SO_2 tank also experienced periodic drops in temperature as fresh steepwater was introduced. The range of values of the actual draw rate, the water absorption rate of corn, and the inflow rate of fresh steepwater of the battery fell within, or were close to, ranges given in literature (Table I).

Milling Yields

In general, milling yields of batch-steeped corn from previous studies fell inside the confidence intervals of the fraction yields for results from the batch-steeped corn from this study (Table II), even though different studies used different steep times and SO_2 levels. Statistical comparison of fraction yields between batch and countercurrent steeped corn from this study (Table III) showed they are statistically similar, indicating that the countercurrent method and the batch method are comparable in terms of product yield. Differences in product yield between the batch steeped corn and the laboratory countercurrent steeped corn were 0.3, 1.7, 0.6, and 0.3%, respectively, for germ, fiber, gluten (protein), and starch.

Countercurrent steeped corn had the highest starch yield at 65.9%, compared to that of previous studies using laboratory steep batteries (Table III). Germ yield, however, was lowest among these studies at 5.8%. Only fiber yield in Watson et al (1951) fell inside the confidence intervals of the fraction yields of this steep battery. However, Steinke et al (1991) separated out an fraction that was mostly starch, but which had a high level of protein that could not be removed using laboratory procedures. This fraction was called "inseparables" and constituted 4.4% of the dry weight. In the current study, this inseparables fraction is recovered with the gluten fraction. Adding the inseparables to Steinke's gluten fraction, the total yield of gluten would be 10%, which is comparable to the 9.5% recovered in the current study. Direct comparison of yields are difficult because each study used different corn hybrids, which may have been exposed to different growing conditions as well as different drying and storage practices. The exact cause of these differences in yield is difficult to determine.

Overall Operation

The system was difficult to maintain in operation on a daily basis due to occasional breakdowns and malfunctions of water level sensors and pump components, in addition to the daily requirements of providing fresh corn and steeping solution and routine checks for leaks and worn-out pump tubing. During the evaluation, it was necessary to maintain a 24-hr watch to minimize the effects of these breakdowns and ensure speedy solutions to any problems. It was determined that preventive replacement of tubing and routine cleaning of steep tank components, particularly the floats and limit switches, resulted in smoother operation. The design objective of having a buffer in the process to allow for unattended operation was not achieved due to the need for consistent maintenance and monitoring for leaks. With 24-hr monitoring, the system was able to be in continuous operation for over five weeks.

TABLE II
Wet Milling Yields of Batch-Steeped Corn[a]

Corn Fractions	Study 1[b]	Study 2[c]	Current Study[d]
Germ	6.2	7	6.1 ± 0.1
Fiber	12.5	11	10.8 ± 1.4
Gluten	8.1	12	8.9 ± 2.0
Starch	65.4	65	65.6 ± 0.9

[a] % of original corn dry substance.
[b] Anderson and Watson (1982).
[c] Eckhoff et al (1991).
[d] Confidence interval at 95% level of significance.

TABLE III
Wet Milling Yields of Countercurrent Steeped Corn[a]

Corn Fraction	Study 1[b]	Study 2[c]	Current Study[d]	Industry Study[e]
Germ	7.2	6.7	5.8 ± 0.6	7.5
Fiber	8.4	10.7	9.1 ± 0.9	11.5
Protein	11.5	5.6	9.5 ± 1.0	5.8
Starch	62.8	64.9	65.9 ± 0.9	67.5
Inseparables	···	4.4	···	···

[a] % of original corn dry substance.
[b] Watson et al 1951.
[c] Steinke et al 1991.
[d] Confidence interval at 95% level of significance.
[e] Anderson and Watson 1982.

Three main design changes would enhance the operation of the system. The first is to replace the float and optical limit switches with nonmechanical upper and lower limit switches. The second is to design an inline heater and control system to eliminate most of the Tygon tubing needed for heating in the waterbaths. The third recommended design change is to use a separate draw tank and pump to allow for continuous rather than intermittent draw from the system.

CONCLUSIONS

A laboratory steep battery was designed with controls that allowed for steepwater routing similar to the methods currently employed by the corn wet-milling industry. Steepwater profiles for total solids, pH, SO_2, and total acidity were comparable to profiles observed in conventional corn wet-milling facilities. Product yield (starch, protein [gluten], fiber, germ, and steepwater solids) for corn steeped in the laboratory steep battery was statistically similar to batch-steeped corn fractionated using the same milling procedure. The product yield was also comparable to values found by other researchers using laboratory countercurrent steeping. When steepwater from an operating facility was used to inoculate the tanks at the start, the use of fresh water with no additional solids gave an adequate fermentation.

Operation of the steep battery required continuous monitoring because of potential for breakdown (sticking of floats, pump tubing failure, and electrical failure). With preventative maintenance, the system could be operated with few disruptions. Suggested design changes included the use of nonmechanical level sensors, individual inline heaters for each tank, and a separate light steepwater tank from which to regulate the draw from the steep battery.

The laboratory steep battery could potentially be used to study many of the design parameters of conventional steep systems, as well as to explore alternative water routing schemes. It can also be used to study the effect of steeping chemicals, enzyme adjuncts, or corn quality.

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