

Laboratory Wet Milling of Ensiled Corn Kernels

A. NERYNG¹ and P. J. REILLY,² Department of Chemical Engineering, Iowa State University, Ames 50011

ABSTRACT

Cereal Chem. 61(1):8-14

Dried and ensiled corn were each steeped with aqueous SO₂ or water at initial pHs of 4–5; both were also steeped with aqueous SO₂ at pH 7. Initial steepwater composition or pH had less effect on yields of individual fractions and recoveries of various components than did differences between dried or ensiled corn. Recovery of starch after milling was

appreciably higher with ensiled corn. Protein recovery in gluten decreased, but this was partially counterbalanced by increased protein content of the steepwater from ensiled corn. Results using batchwise steeping or a simulated countercurrent procedure were similar, though the latter gave higher starch recoveries.

A significant portion of the corn produced in the United States is wet milled into its major fractions—starch, gluten, oil, and fiber. This process is both capital- and energy-intensive. Corn kernels are first steeped at roughly 50°C for approximately 48 hr in a weak SO₂ solution. This occurs at the same time that steepwater flows from tanks holding the most thoroughly steeped corn towards those containing corn just entering the process. Then, after rough and eventual fine grinding, the fractions are separated from each other in varying degrees of purity by several centrifugal steps.

Several aspects of wet milling have been studied. These include the mechanism of SO₂ action, the role of the lactic acid produced by lactobacilli during steeping, and the effects of variations in SO₂ concentration, steeping time, corn strain, and artificial drying on kernel degradation and fraction yield and purity. Two major reviews (Watson 1967, Wahl 1969) have dealt with the chemical side of wet-milling, as opposed to the mechanical. Because of the time that has elapsed since they appeared, and because so much of the previous work in the area impinges on the methods employed and results obtained in this paper, a quick synopsis of that work is given here.

Basically, soaking in aqueous solutions causes mature corn kernels to imbibe water and other solution ingredients, to swell greatly, and to release soluble portions of the kernel to its surroundings. Moisture in the kernel increases towards its equilibrium value (roughly 45% of total weight) at a rate inversely proportional to the square root of elapsed time (Fan et al 1965).

Although soaking in water alone will lead to significant softening and substantial separation of the components of the kernel upon further processing, long experience has shown that best results are obtained when SO₂ is part of the solution. The primary action of SO₂ appears to be its attack on protein disulfide bonds, which leads to degradation of walls and protein fibers throughout the cells of the kernel (Cox et al 1944, Wagoner 1948, Boundy et al 1967, Wahl 1970a). This process continues after water uptake is essentially complete, and in general starch yield and recovery improves

throughout the whole steeping process (Watson et al 1955a, Anderson et al 1961). Increasing initial SO₂ concentration up to 0.25% or somewhat higher leads to increased starch yields and purities, but to lower viscosity of the paste after gelatinization (Cox et al 1944, Dostal 1953, Lindemann 1954, Watson and Sanders 1961, Krochta et al 1981).

The influence of lactic acid on the steeping process and on final milling results has been more difficult to determine. It is produced by bacteria that are found naturally on the kernels and that enter the system with process water; fermentation during the steeping process is adversely affected by high SO₂ concentrations (Dostal 1953, Watson et al 1955b, Roushdi et al 1981a). It does appear that lactic acid increases kernel softening and may participate in protein breakdown (Cox et al 1944, Wahl 1970a, Maslennikova 1970); high concentrations may also increase fraction yields, though this effect is obscured by the inverse relationship between SO₂ and lactic acid concentrations (Roushdi et al 1981a,b).

The kernel contains hydrolases, proteases, and lipases that can act on its other components (Wahl 1970b). The acid conditions during steeping are not favorable for lipase activity but are ideal for proteolysis of the amylases present (Wahl 1971a,b), and therefore starch and oil at least are not materially altered by enzyme attack during steeping.

Two other factors—corn variety and severity of artificial drying—also have a strong effect on milling results. Very intensive research has consistently shown that drying temperatures above approximately 60°C decrease removal of extractable solubles and starch yield and purity (MacMasters et al 1954, 1959, Watson and Sanders 1961, Watson and Hirata 1962, Wahl 1966), whereas the effect of initial and final water content is negligible (MacMasters et al 1959, Kempf 1961). However, it should be noted that perhaps the strongest effect of all on yields, purities, and ease of milling is that exerted by the variety of corn employed, and that yield of starch is not necessarily correlated with initial starch content (Pelshenke and Lindemann 1954, Watson et al 1955a, Kempf and Tegge 1961, Wight 1981).

The preceding results have all been gathered at essentially laboratory scale, under the assumption that they fairly closely duplicate what would be found under the same conditions at industrial scale. This appears to be valid for both countercurrent (Watson et al 1951) and batch (Anderson 1963) steeping and

¹ Permanent address: Institute of Human Nutrition, Warsaw Agricultural University, Poland.

² Visiting scientist and professor, respectively.

subsequent laboratory-scale milling.

Given this background, the question arises whether corn that has been ensiled rather than artificially dried can be wet milled to obtain satisfactory fraction yields and purities. The prospect is appealing, as ensiled corn has already undergone a natural lactic acid fermentation. Therefore a high lactic acid concentration would always be present during steeping of ensiled corn, allowing any benefit provided by lactic acid to be gained, even at high initial SO₂ levels. In addition, ensiling is more energy-efficient than artificial drying. To counter this, ensiled corn, although stable in the absence of air, rapidly decomposes in its presence, so transportation to the milling plant and subsequent storage can be a problem.

There appears to be no previous literature on the wet milling of ensiled corn. To fill this gap, the project described here was undertaken to investigate the steeping and further wet milling of

ensiled corn under laboratory conditions. Although it is clear from the foregoing literature survey that added SO₂ is beneficial for increased yields and ease of milling of dried corn, and although low initial pHs are used to discourage growth of microbes other than lactobacilli during steeping, it is not clear that the same factors hold for ensiled corn. Therefore, both dried and ensiled corn were steeped at two different initial pHs, with and without SO₂, and yields of various fractions and recoveries of various components were determined after milling. In addition, steepwater compositions and corn moisture levels were measured at various steeping times for both ensiled and dried corn.

MATERIALS AND METHODS

Corn Samples

For steeping at initial pHs between 4 and 5, in either water or an

TABLE I
Composition and Origin^a of Corn Samples Steeped at Initial pHs of 4-5

	Type of Corn					
	Ensiled			Dried		
	A	B	C	A	B	C
Corn hybrid	Pioneer 3541 ^b	Pioneer 3183, 3388,3541	Pioneer 3183	Pioneer 3541, other Pioneer	Pioneer 3183, 3388,3541	Pioneer 3388
Moisture (%)	20.3	27.8	21.9	12.6	10.5	16.4
Starch (% d.b.)	67.3	71.9	63.6	72.2	70.4	69.6
Protein (% d.b.)	10.49	8.75	9.34	9.22	9.60	9.55
Oil (% d.b.)	3.92	4.34	4.90	4.81	4.39	4.90
Total (% d.b.)	81.7	85.0	77.8	86.2	84.4	84.1
Drying temperature (°C)	60-71	38-49	38-49

^aAll farms, identified by capital letters, were located in central Iowa.

^bPioneer Hi-Bred International Inc., Des Moines, IA.

TABLE II
Composition and Origin of Ensiled Corn Samples Steeped at Initial pHs near 7

	Corn Hybrid							
	Pioneer 3183, 3388, 3541	Pioneer 3183	Crow's 12 and 15 ^a	Great Lakes ^b	Stauffer 5602 ^c	Jacques 97 ^d	Tracy 205 ^e	
	B Central Iowa	C Central Iowa	D Northeastern Iowa	E Southwestern Wisconsin	F Southwestern Wisconsin	G Southeastern Wisconsin	H Southwestern Wisconsin	
Approximate composition								
Moisture (%)	24.5	17.4	23.7	27.4	30.4	24.1	22.0	
Starch (% d.b.)	71.8	63.3	66.1	67.5	64.9	73.1	67.1	
Protein (% d.b.)	9.49	9.03	9.08	10.29	10.10	10.59	9.58	
Oil (% d.b.)	3.65	4.75	4.42	3.73	4.22	3.98	4.19	
Total (% d.b.)	84.9	77.1	79.6	81.5	79.2	87.7	80.9	

^aCrow's Hybrid Corn Co., Milford, IL. High-lysine strains.

^bGreat Lakes Hybrid Corn, Ovid, MI.

^cStauffer Chemical Co., Westport, CT.

^dJacques Seed Co., Prescott, WI.

^eTracy and Son Farms, Janesville, WI.

TABLE III
Composition and Origin of Dried^a Corn Samples Steeped at Initial pHs near 7

	Corn Hybrid				
	Great Lakes	Stauffer 4402	Super Crost 1950 ^b	Black Hawk 2460 ^c	Unknown
	E Southwestern Wisconsin	F Southwestern Wisconsin	G Southeastern Wisconsin	H Southwestern Wisconsin	I Southwestern Wisconsin
Approximate composition					
Moisture (%)	16.6	13.4	12.4	13.3	12.0
Starch (% d.b.)	68.6	66.7	70.5	65.3	64.2
Protein (% d.b.)	9.41	10.51	8.03	9.71	10.30
Oil (% d.b.)	4.25	5.00	4.05	3.85	4.17
Total (% d.b.)	82.3	82.2	82.6	78.9	78.7

^aDrying temperature = approximately 60°C.

^bEdward J. Funk and Sons, Kentland, IN.

^cBlackhawk Seeds, Janesville, WI.

aqueous solution initially at 0.25% SO₂, dried and ensiled corn samples were obtained from each of three central Iowa farms. They had been harvested during the autumn of 1981, after a normal growing season and a somewhat wet fall, and had been stored until early March 1982. Corn hybrid identities and compositions of each of the six samples are given in Table I.

For steeping at an initial pH near 7 and an aqueous solution initially at 0.25% SO₂, five dry and seven ensiled corn samples were obtained from five Wisconsin and three Iowa farms. They had been harvested during the autumn of 1981 and stored until early June 1982. Details of each of the 12 samples are given in Tables II and III.

All high-moisture corn was ensiled in A. O. Smith Harvestore® units, in which oxygen initially present is quickly taken up by respiration and no more is allowed to enter. The other corn was artificially dried on the farm at varying temperatures (Tables I and III). Iowa samples were delivered almost immediately, so no special precautions were taken. Wisconsin samples were placed

under nitrogen or fluorocarbon blankets. All samples received were kept at 4°C until used.

Steeping and Milling Procedure

Approximately 50-g (d.b.) portions of each of the three dried corn and three ensiled corn samples in Table I were added individually to 250 ml of water or aqueous Na₂SO₃ solution made up to 0.25% SO₂ and adjusted to pH 4–5 with concentrated H₂SO₄. Roughly 100-g quantities of the five samples of dried corn and seven samples of ensiled corn in Tables II and III were added to 500-ml solutions of aqueous 0.25% SO₂ adjusted to approximately pH 7 with concentrated H₂SO₄.

Each sample was steeped and milled, roughly following the procedure of Anderson (1963). The mixtures were held at 52°C with shaking hourly for the first half day or so and less often thereafter. Samples of 20 ml of steepwater and 2 g (d.b.) of corn were removed at specified times for composition and moisture determination, respectively. After 48 hr, the steepwater was decanted and analyzed, and excess liquid was removed from the corn by blotting. Samples of approximately 25 g of corn, with about 15 ml of added water, were coarsely ground with a Janke & Kunkel type A10 blender with two 55-mm diameter blades and screened in turn with 40, 200, and 270 U.S. Standard sieves (0.420-, 0.074-, and 0.053-mm openings, respectively). The fraction that was held up by the first sieve was ground somewhat more finely twice more with a mortar and pestle with extra water added, taking care not to break up germ and fiber particles, and was screened again with the first sieve. Material stopped by the 200- and 270-mesh sieves was washed with water to more completely separate starch, which passed through, from gluten, which was held up. After settling, starch was separated from water by decanting. The procedure is shown schematically in Fig. 1.

To further investigate the steeping and milling of ensiled corn, 2.5 kg (w.b.) samples of corn were added to each of three 7-L baffled New Brunswick fermentors filled with 3 L of aqueous 0.25% SO₂ adjusted to pH 7 and held at 52°C. The supernatant in each vessel was stirred at 10–15 rpm. A solution of SO₂ or steepwater flowed at 160 ml/hr to the top of each fermentor and was withdrawn from the bottom, so that each vessel operated batchwise for 6 hr and received flow for the following 18 hr on a staggered basis (Fig. 2). Samples were treated as before, except that steeped corn was initially ground with a Quaker City 3–3/4X mill.

Analyses

Moisture in corn kernels was determined by drying for 1.5 hr at 120°C; in processed samples it was removed by drying at 49°C overnight (Anderson 1963). Starch was measured polarimetrically by the CaCl₂ procedure of Earle and Milner (1944). Protein (N × 6.25) was analyzed by the macro-Kjeldahl method with a Tecator digestion system 1005-20 heating unit, Kjeldahl system 1003 distillation system, and 1002-21 titration unit (Tecator, Inc., Herndon, VA). Oil was determined by Soxhlet extraction (Barnett 1954) for 16 hr with 8–10 g of sample and 200–250 ml of an equal volume mixture of diethyl ether and 1,1,1-trichloroethane (Hoffman 1960). Lactic acid was measured by either the method of Smith (1953) or by gas chromatography. In the latter case, a 150-mm long, 3.2-mm o.d. stainless steel column containing 10% SP-1200 and 1% H₃PO₄ adsorbed on 80–100 mesh Chromosorb WAW in a Varian 3700 gas chromatograph was employed. Column temperature was maintained at 160°C, the injector at 200°C, and the flame-ionization detector at 220°C. Nitrogen passed through the column at the rate of 30 ml/min; 30 ml/min H₂ and 300 ml/min air were fed to the detector. Sample pH was decreased to below 3.5 with 0.5N H₂SO₄ (Theune 1978). Volatile acids and ethanol were analyzed by the same technique, except that column temperature increased from 60 to 120°C at 10°C/min.

RESULTS AND DISCUSSION

The 18 samples in Tables I, II, and III were individually steeped, the six in Table I with both SO₂ and water. Kernel moisture and steepwater pH, protein, and lactic acid were periodically

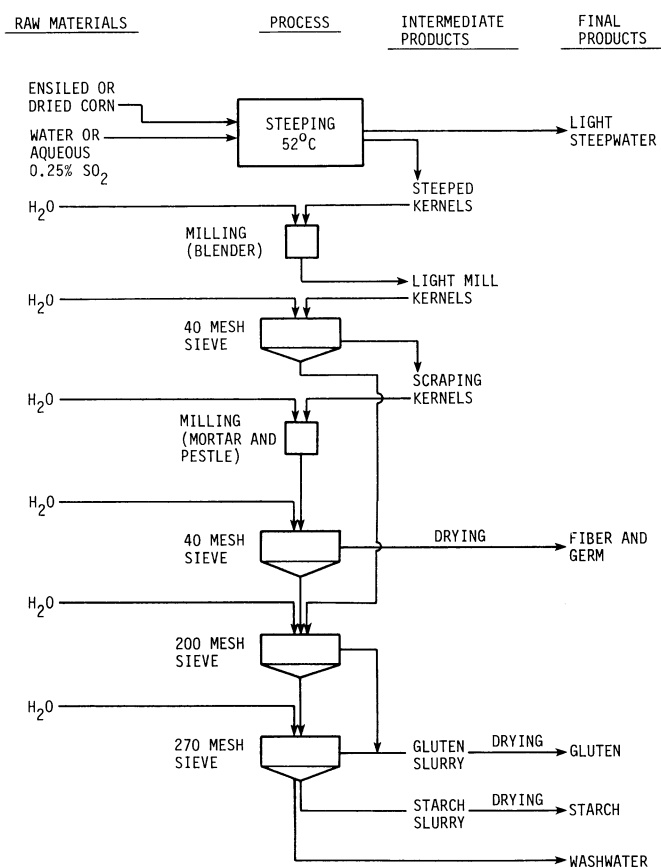


Fig. 1. Laboratory procedure for steeping and separation of ensiled or dried corn.

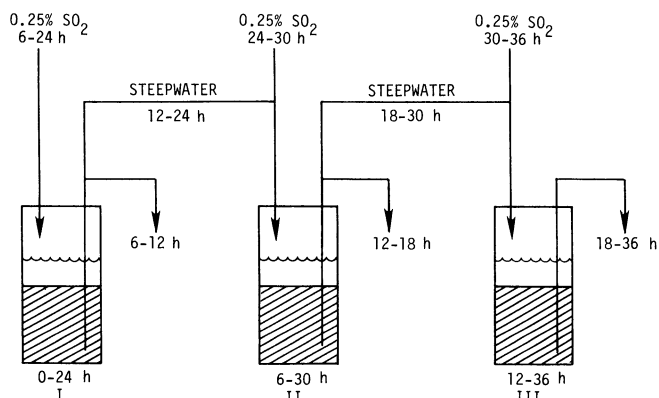


Fig. 2. Simulated countercurrent steeping of ensiled corn in three vessels.

determined. In addition, kernels were milled after 48 hr of steeping, and fraction yields and compositions were obtained for each sample. There was no correlation between initial composition of either dried or ensiled corn samples or between drying conditions, which were only poorly controlled, and any experimental results. Therefore data from individual samples were grouped into the four categories shown in Table IV. There were no significant differences at the level of $P = 0.05$ across the four categories in any component except moisture. For these reasons, it may be assumed that the four groupings are essentially equivalent and, because of their breadth of hybrids and origins, are a fair sample of dent corn produced in the upper midwestern United States during a good growing season.

In addition, presence or absence of SO_2 caused no noticeable differences in moisture content of kernels or pH of steepwater. Therefore, data for water and SO_2 steeping at initial pHs of 4-5 were combined for moisture and pH. As shown in Fig. 3, corn samples rapidly imbibed water during steeping, reaching final moisture contents between 40 and 45%, as expected. Water uptake appears more rapid at lower initial pHs.

Steepwater pHs of those samples having initial pHs between 4 and 5 rose rapidly and then decreased, starting at higher values and ending at lower ones in the case of ensiled corn (Fig. 4). Though pHs of steepwater initially near 7 decreased throughout steeping, those associated with ensiled corn again reached lower values, caused undoubtedly by extraction of already formed lactic acid from the kernels.

Protein contents of steepwater above ensiled corn samples initially near pH 7 rose somewhat more quickly and reached appreciably higher final values than those of steepwater associated with dried corn (Fig. 5). This could have been caused by the higher

microbial content initially present in the ensiled corn or more soluble proteins in ensiled corn. Data from two runs conducted with SO_2 and water initially at pH 4 (not shown) were similar to those in Fig. 5, though final protein values were somewhat higher.

Lactic acid in steepwater followed the same pattern as did protein, increasing more quickly and attaining higher final levels with ensiled corn (Fig. 6). Operation at higher pHs increased lactic acid. Use of SO_2 did not decrease it, contrary to earlier published results. Ethanol and acetic, propionic, butyric, isobutyric, and isovaleric acids were also produced, but none other than ethanol and propionic acid ever exceeded 0.1% at the end of steeping. Only in rare cases did the latter two exceed 0.2%.

Yields of each fraction on a dry basis are shown in Table V. Tests of significant differences between means were conducted for single changes in type of sample, initial pH, and use of SO_2 or water for steeping. In no case were differences at the $P < 0.05$ level found after substitution of water for SO_2 in steeping. Ensiled corn gave more of the starch fraction and less of the gluten fraction than did dried corn, and the differences became highly significant when steeping occurred at initial pHs near 7. Yields of fiber and germ were also significantly lower for ensiled corn at pH 7. Yields of individual insoluble fractions were often lower at pH 7 than at pH 4-5, and the tendency was especially noticeable for the total yield of all three fractions.

TABLE IV
Mean Composition of Corn Samples Used for Steeping and Milling

	Initial pH 4-5		Initial pH 7	
	Dried (3) ^a	Ensiled (3)	Dried (5)	Ensiled (7)
Moisture (%)	13.2 ± 3.0 ^b	23.3 ± 4.0	13.5 ± 1.8	24.2 ± 4.1
Starch (% d.b.)	70.7 ± 1.3	67.6 ± 3.4	67.1 ± 2.5	67.7 ± 3.6
Protein (% d.b.)	9.46 ± 0.21	9.53 ± 0.88	9.59 ± 0.98	9.74 ± 0.60
Oil (% d.b.)	4.70 ± 0.21	4.39 ± 0.46	4.26 ± 0.44	4.13 ± 0.39
Total (% d.b.)	84.9 ± 1.1	81.5 ± 3.6	80.9 ± 2.0	81.6 ± 3.6

^aNumber of samples.

^bStandard deviation.

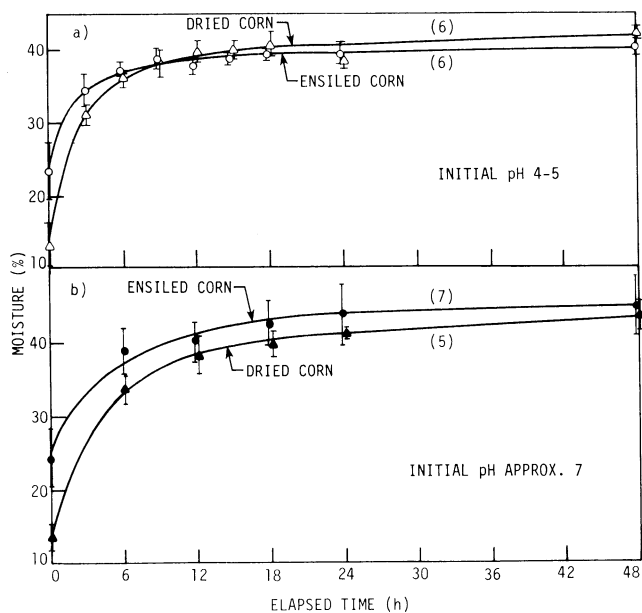


Fig. 3. Moisture content of corn kernels during steeping. Ranges are standard deviations. Numbers in parentheses are numbers of samples used for average values in each curve.

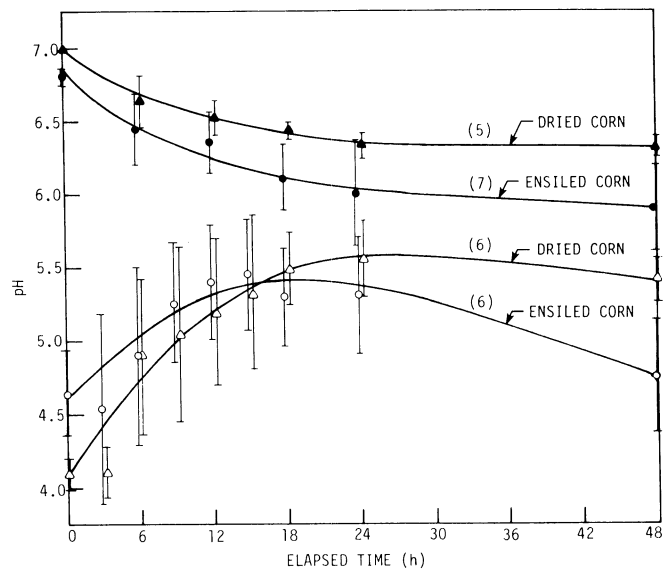


Fig. 4. pH of steepwater during steeping.

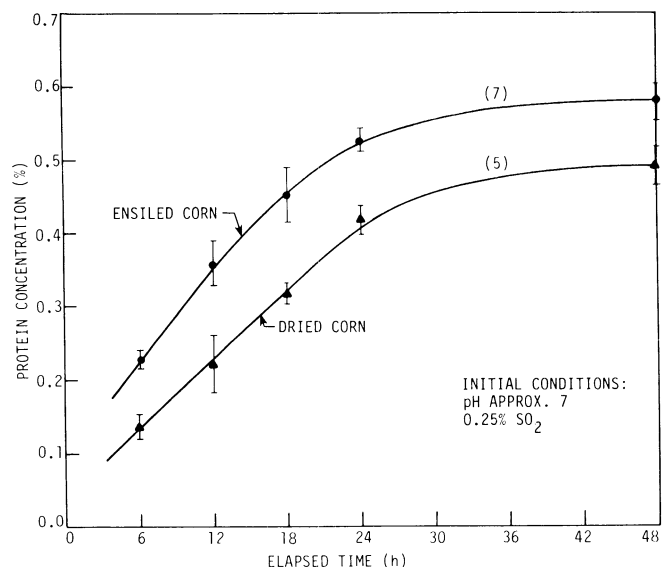


Fig. 5. Protein content of steepwater during steeping.

Compositions of the three insoluble fractions were strongly affected by whether dried or ensiled corn was used (Table VI). Ensiled corn steeped at an initial pH of 7 gave starch significantly freer of protein, and gluten lower in starch and higher in protein.

Fiber and germ was significantly higher in oil at either initial pH range.

When yields and compositions of crude fractions were combined to obtain recoveries of individual components, variations between feedstocks and initial steepwater pH became more evident (Table VII). Use of ensiled corn at an initial pH near 7 gave higher recoveries of starch in the starch fraction and overall, and lower starch recoveries in gluten and fiber and germ fractions. In general, less protein was recovered from ensiled corn, both in the starch and gluten fractions and overall (not including steepwater). Increase of initial steepwater pH appears to cause a decrease of protein recovery, both overall and in the gluten and fiber and germ fractions.

Simulated countercurrent steeping of ensiled corn gave yields and compositions of crude fractions very similar to those obtained by batch steeping, though starch was somewhat purer (Table VIII). This led to a higher recovery of starch than with batch-steeped ensiled corn, both overall and in the starch fraction (Table IX). No countercurrent steeping of dried corn was conducted.

CONCLUSIONS

From this study, we have concluded that ensiled corn is an acceptable feedstock for wet milling. In general, separation of components, especially starch, is more complete. This is probably caused by the fermentation that occurs in ensiled corn even before it undergoes steeping, which is reflected in increased lactic acid and perhaps increased protein content in the steepwater associated with ensiled corn. Although protein recovery in starch and gluten fractions is lower when ensiled corn rather than dried corn is processed, this is partially counterbalanced by the increased protein content of the resulting steepwater.

Operation at higher pHs causes decreased yields of insoluble products, in addition to potential increased difficulty with contaminating microbial growth. Surprisingly, removal of SO₂ in steeping leads to no significant differences in yields or recoveries, though again microbial contamination could be a problem.

It is interesting that change of feedstock from dried to ensiled

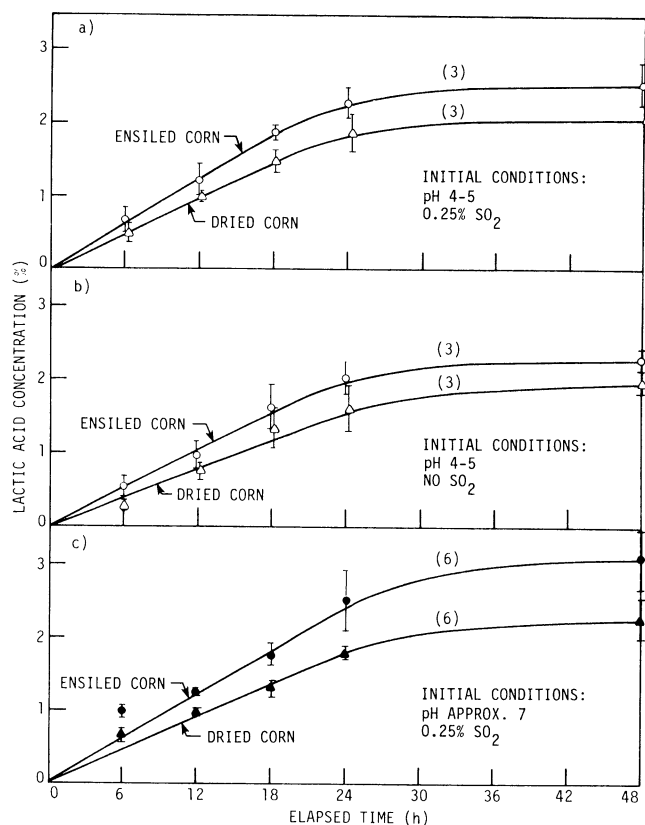


Fig. 6. Lactic acid content of steepwater during steeping.

TABLE V
Mean Yields of Crude Fractions After Steeping and Milling^a

Fraction	Steeping	Yield on Total Sample (% d.b.)			
		Initial pH 4-5		Initial pH 7	
		Dried (3)	Ensiled (3)	Dried (5)	Ensiled (7)
Starch	0.25% SO ₂	59.7 b ± 3.4	61.9 ± 4.0	54.7 b,e ± 2.0	60.3 e ± 2.3
	Water	58.1 ± 3.9	61.5 ± 2.9
Gluten	0.25% SO ₂	15.4 ± 4.0	12.6 b ± 2.5	14.7 e ± 3.0	9.9 b,e ± 1.3
	Water	14.1 ± 3.6	12.7 ± 4.3
Fiber and germ	0.25% SO ₂	21.0 ± 1.9	21.4 ± 5.6	22.6 d ± 1.5	18.3 d ± 2.7
	Water	21.7 ± 5.6	23.2 ± 2.9
Total yield	0.25% SO ₂	96.1 ± 1.7	96.0 e ± 2.9	92.0 ± 3.5	88.5 e ± 2.1
	Water	94.0 ± 2.8	97.4 ± 1.7

^a Means in the same row designated by the same letter are significantly different. b, 0.05 > P > 0.025; d, 0.01 > P > 0.005; and e, 0.005 > P.

TABLE VI
Mean Compositions of Crude Fractions After Steeping and Milling^a

Fraction	Component	Steeping	Composition (% d.b.)			
			Initial pH 4-5		Initial pH 7	
			Dried (3)	Ensiled (3)	Dried (5)	Ensiled (7)
Starch	Starch	0.25% SO ₂	95.6 ± 0.3	95.9 ± 0.7
	Protein	0.25% SO ₂	1.29 e ± 0.11	0.90 e ± 0.10
Gluten	Starch	0.25% SO ₂	24.4 e ± 0.9	21.3 e ± 0.8
	Protein	0.25% SO ₂	38.2 ± 4.2	39.6 ± 2.5	35.0 e ± 2.7	41.2 e ± 1.3
Fiber and germ	Protein	Water	39.1 ± 3.3	39.5 ± 3.2
	Starch	0.25% SO ₂	20.0 ± 1.2	19.0 ± 1.9
	Protein	0.25% SO ₂	11.7 b ± 1.2	12.8 ± 2.0	9.7 b ± 1.1	12.5 ± 2.8
	Protein	Water	13.4 ± 0.7	12.0 ± 2.0
	Oil	0.25% SO ₂	8.18 c ± 0.30	9.16 c ± 0.28	8.53 e ± 0.32	9.39 e ± 0.15
	Oil	Water	8.51 b ± 0.25	9.15 b ± 0.33

^a Means in the same row designated by the same letter are significantly different. b, 0.05 > P > 0.025; c, 0.025 > P > 0.01; and e, 0.005 > P.

TABLE VII
Recoveries of Components in Various Fractions After Steeping and Milling

Component and Fraction	Steeping	Recovery (%)			
		Initial pH 4-5		Initial pH 7	
		Dried (3)	Ensil'd (3)	Dried (5)	Ensil'd (7)
Starch in starch	0.25% SO ₂	77.9	85.4
in gluten	0.25% SO ₂	5.3	3.1
in fiber and germ	0.25% SO ₂	6.7	5.1
Total starch recovery	0.25% SO ₂	89.9	93.6
Protein in starch	0.25% SO ₂	7.4	5.6
in gluten	0.25% SO ₂	62.2	52.3	53.6	41.9
in gluten	Water	58.3	52.6
in fiber and germ	0.25% SO ₂	26.2	28.7	22.8	23.5
in fiber and germ	Water	30.7	29.2
Total protein recovery ^a	0.25% SO ₂	83.8	71.0
Oil in fiber and germ	0.25% SO ₂	36.5	44.7	45.3	41.6
	Water	39.3	48.4

^aExcluding steepwater protein.

TABLE VIII
Yields and Composition of Crude Fractions from Ensil'd Corn After Simulated Countercurrent Steeping and Milling^{a,b,c}

Fraction	Fermentor II		Fermentor III	
	Yield on Total Sample (% d.b.)	Composition (% d.b.)	Yield on Total Sample (% d.b.)	Composition (% d.b.)
Starch	62.4	97.6 Starch 0.60 Protein	63.2	97.1 Starch 0.48 Protein
Gluten	10.5	24.3 Starch 40.6 Protein	9.3	22.5 Starch 42.5 Protein
Fiber and germ	17.2	18.5 Starch 12.3 Protein 9.25 Oil	17.0	18.1 Starch 13.8 Protein 9.34 Oil

^aInitial corn composition was 23.0% moisture, 67.2% starch (d.b.), 9.45% protein (d.b.), and 4.1% oil (d.b.).

^bpH of steepwater was 7.0 (0 hr), 6.65 (12 hr), 5.78 (18 hr), and 5.85 (24 hr).

^cLactic acid concentration in steepwater was 3.32% (36 hr).

TABLE IX
Recoveries of Components in Various Fractions After Simulated Countercurrent Steeping and Milling

Component and Fraction	Recovery (%)	
	Fermentor II	Fermentor III
Starch in starch	90.6	91.3
in gluten	3.8	3.1
in fiber and germ	4.7	4.6
Total starch recovery	99.1	99.0
Protein in starch	4.0	3.2
in gluten	45.1	41.8
in fiber and germ	22.4	24.8
Total protein recovery ^a	71.5	69.8
Oil in fiber and germ	38.8	38.7

^aExcluding steepwater protein.

corn leads to greater changes in product composition and recoveries of various components than do changes in either SO₂ content or initial pH of steepwater. That the use of ensil'd corn leads to potentially superior wet-milling performance is noteworthy, and may suggest that this feedstock could play a role in the industrial production of starch and other wet-milling products.

ACKNOWLEDGMENTS

This project is supported by A. O. Smith Harvestore Products, Inc., the Engineering Research Institute of Iowa State University, and the Cooperative Graduate Research Program at Iowa State, which is funded by

the U.S. International Communications Agency. The authors thank David Mroch for help with the experimental work, Richard Vetter for advice and for arranging the delivery of samples, and Robert Serfass for use of the Kjeldahl apparatus.

LITERATURE CITED

- ANDERSON, R. A. 1963. Wet milling properties of grains: Bench-scale study. *Cereal Sci. Today* 8:190.
- ANDERSON, R. A., VOJNOVICH, C., and GRIFFEN, E. L., Jr. 1961. A note on the effect of steeping time on wet-milling high-amylose corn containing 57-percent-amylose starch. *Cereal Chem.* 38:84.
- BARNETT, A. J. G. 1954. *Silage Fermentation*. Academic Press, New York.
- BOUNDY, J. A., TURNER, J. E., WALL, J. S., and DIMLER, R. J. 1967. Influence of commercial processing on composition and properties of corn zein. *Cereal Chem.* 44:281.
- COX, M. J., MacMASTERS, M. M., and HILBERT, G. E. 1944. Effect of the sulfurous acid steep in corn wet milling. *Cereal Chem.* 21:447.
- DOSTAL, L. 1953. Über die Zusammensetzung der Maisquellwassers und die rheologischen Eigenschaften der Stärke in Abhängigkeit von die Quellbedingungen. *Stärke* 5:116.
- EARLE, F. R., and MILNER, R. T. 1944. Improvements in the determination of starch in corn and wheat. *Cereal Chem.* 21:567.
- FAN, L.-T., CHEN, H.-C., SHELLENBERGER, J. A., and CHUNG, D. S. 1965. Comparison of the rates of absorption of water by corn kernels with and without dissolved sulfur dioxide. *Cereal Chem.* 42:385.
- HOFFMAN, H. H. 1960. Study of chlorinated solvents for crude fat in feeds. *J. Assoc. Off. Agric. Chem.* 43:315.
- KEMPF, W. 1961. Die Abhängigkeit von Ausbeuten und Qualität der End- und Nebenprodukte von Wassergehalt des Rohmaises. *Stärke* 13:404.
- KEMPF, W., and TEGGE, G. 1961. Die Eignung verschiedener Maissorten für die Stärkefabrikation. *Stärke* 13:363.
- KROCHTA, J. M., LOOK, K. T., and WONG, L. G. 1981. Modification of corn wet-milling steeping conditions to reduce energy consumption. *J. Food Process Preserv.* 5:39.
- LINDEMANN, E. 1954. Der Einfluss der Maissorte und des Verarbeitungprozesses auf die Qualität der Maisstärke. *Stärke* 6:274.
- MASLENNIKOVA, T. N. 1970. Zur Frage der Verwendung von Natriumdisulfid statt schwefeliger Säure bei der Maisstärkeproduktion. *Stärke* 22:270.
- MacMASTERS, M. M., EARLE, F. R., HALL, H. H., RAMSER, J. H., and DUNGAN, G. H. 1954. Studies on the effect of drying conditions on the composition and suitability for wet-milling of artificially dried corn. *Cereal Chem.* 31:451.
- MacMASTERS, M. M., FINKNER, M. D., HOLZAPFEL, M. M., RAMSER, J. H., and DUNGAN, G. H. 1959. A study of the effect of drying conditions on the suitability for starch production of corn artificially dried after shelling. *Cereal Chem.* 36:247.
- PELSHENKE, P. F., and LINDEMANN, E. 1954. Untersuchungen über die Eignung verschiedener Maissorten für die Stärkefabrikation. *Stärke* 6:177.
- ROUSHDI, M., FAHMY, A. A., and MOSTAFA, M. 1981b. Role of lactic acid in corn steeping and its relation with starch isolation. *Stärke* 33:426.
- ROUSHDI, M., GHALI, Y., and HASSANEAN, A. 1981a. Factors improving the steeping process of corn grains. III. Conditions favoring lactic acid formation during corn steeping and its effect. *Stärke* 33:49.
- SMITH, R. J. 1953. Determination of lactic acid in heavy corn steep liquor. *Anal. Chem.* 25:505.
- THEUNE, H. H. 1978. Gaschromatographische Bestimmung der kurzkettigen Fettsäuren einschliesslich Äthanol und Milchsäure aus Gärfutter. Ein Methodenvergleich. *Landwirtsch. Forsch.* 35:540.
- WAGONER, J. A. 1948. Microscopical observations and procedures used in a study of industrial corn steeping. *Cereal Chem.* 25:354.
- WAHL, G. 1966. Maistrocknung und Nassmüllerei. I. Untersuchung der Verarbeitungsfähigkeit von künstlich getrocknetem Mais. *Stärke* 18:11.
- WAHL, G. 1969. Biochemische-Technologische Studien über die Nassverarbeitung von Mais. I. Zum Stand der Kenntnisse über den Maisquellenprozess. *Stärke* 21:68.
- WAHL, G. 1970a. Biochemische-Technologische Studien über die Nassverarbeitung von Mais. 3. Die Milieubedingungen im Maiskorn während des Quellprozesses. *Stärke* 22:77.
- WAHL, G. 1970b. Biochemische-Technologische Studien über die Nassverarbeitung von Mais. 4. Über die qualitative Zusammensetzung des Mais-Hydrolasensystems und Aktivität einzelner Enzyme bsw. Enzymgruppen. *Stärke* 22:232.
- WAHL, G. 1971a. Biochemische-Technologische Studien über die Nassverarbeitung von Mais. 5. Untersuchung des amylolytischen Enzymsystems des Mais. *Stärke* 23:85.

- WAHL, G. 1971b. Biochemische-Technologische Studien über die Nassverarbeitung von Mais. 6. Untersuchung des proteolytischen und lipolytischen Enzymsystems des Mais. *Stärke* 23:145.
- WATSON, S. A. 1967. Manufacture of corn and milo starches. Page 1 in: *Starch: Chemistry and Technology*. Vol. II. R. L. Whistler and E. F. Paschall, eds. Academic Press, New York.
- WATSON, S. A., and HIRATA, Y. 1962. Some wet-milling properties of artificially dried corn. *Cereal Chem.* 39:35.
- WATSON, S. A., HIRATA, Y., and WILLIAMS, C. B. 1955b. A study of the lactic acid fermentation in commercial corn steeping. *Cereal Chem.* 32:382.
- WATSON, S. A., and SANDERS, E. H. 1961. Steeping studies with corn endosperm sections. *Cereal Chem.* 38:22.
- WATSON, S. A., SANDERS, E. H., WAKELY, R. D., and WILLIAMS, C. B. 1955a. Peripheral cells of the endosperms of grain sorghum and corn and their influence on starch purification. *Cereal Chem.* 32:165.
- WATSON, S. A., WILLIAMS, C. B., and WAKELY, R. D. 1951. Laboratory steeping procedures used in a wet-milling research program. *Cereal Chem.* 28:105.
- WIGHT, A. W. 1981. Changes in properties of some maize cultivars associated with artificial drying at elevated temperatures. I. Wet-milling properties. *Stärke* 33:122.

[Received October 22, 1982. Accepted July 17, 1983]