

Wet Milling of Grain Sorghum Using a Short Steeping Period

PING YANG^{2,3} and PAUL A. SEIB^{2,4}

ABSTRACT

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Commercial yellow grain sorghum was steeped in a vented container at 50–70°C for 2–10 hr with 1.5 parts (by weight) of water containing initially 0.1–0.5% sulfur dioxide (SO₂). During steeping, ≈75% of the SO₂ was lost through the vent. When grain sorghum and dent corn were steeped at 58°C with an initial concentration of 0.25% SO₂, steepwater uptake by grain sorghum was faster during the first 4 hr, but leveled off at 4% below that of corn after 10 hr. In addition, the steepwater of grain

sorghum became 25% more acidic than that of corn after 2–10 hr. The optimum recovery of starch from grain sorghum steeped 4 hr was 51% at 58°C with an initial concentration of 0.3% SO₂. Decortication of the grain sorghum produced 70% recovery of pericarp-free, broken kernels, which gave a somewhat reduced recovery of milled starch with only a slight improvement in brightness. The hot paste characteristics of the starches from two samples of yellow grain sorghums matched those of a commercial corn starch.

Grain sorghum and corn starches, both waxy and normal, are practically interchangeable, although corn starch granules are smaller (Watson 1970). Wet milling of large tonnages of corn is practiced around the world, yet no wet milling of grain sorghum is done. Grain sorghum is a less consistent commodity than corn in terms of size, color, and defects. Wet milling of sorghum grain yields ≈50% less cooking oil than corn, although the potential for controlling oil composition appears less complex for grain sorghum than for corn (Davis et al 1990). The recovery of starch is not so complete from grain sorghum as from corn. Sorghum starch is lost in its germ and fiber fractions because some starch occurs in its pericarp, and because some of its peripheral cells are not opened during grinding of the steeped grain (Rooney 1973, Watson 1984). Furthermore, sorghum starch is associated with more highly crosslinked proteins than corn starch (Hamaker et al 1992), and separating starch from sorghum protein is more difficult. Finally, sorghum starch usually is less bright than corn starch and may be stained with pericarp or glume pigments in the field or during processing (Watson 1970, Munck 1995).

One method of adding value to a grain is to separate, purify, and modify its chemically distinct components for specialty uses. Because of the problems with exhaustive wet milling of grain sorghum, an abbreviated low-input process yielding starch and a feed by-product may be a better approach. In our first attempt (Yang and Seib 1995), sorghum grain was wet-ground at a 1.2 ratio of water and grain. After sieving and centrifuging, and recycling of water with no liquid effluent, two products were isolated: wet starch (40% moisture) and an animal feed (50% moisture). The animal feed product was mixed directly with other ingredients to give a growing ration for cattle. Alternatively, a preserved feed ingredient was produced by ensiling a mixture of the wet by-product and dry alfalfa. The yield of dried starch amounted to 20%, which represented a 27% recovery of the starch present in the grain. The starch contained 0.8% protein, half of which was removed by treatment with protease or high-shearing in water.

In the present investigation, we have explored short-time steeping in aqueous sulfite to improve the recovery of starch from grain sor-

ghum and from decorticated grain. We also determined the protein, color, and pasting properties of the starches.

MATERIALS AND METHODS

Materials

Yellow No. 2 dent corn was a 1994 Iowa cultivar. One sample of grain sorghum, commercial yellow No. 2, was kindly provided in clean condition by Grain Products Inc., ADM, Dodge City, KS. A second commercial sample, which also appeared to be yellow grain sorghum, was purchased from a local farmers' cooperative. The second sample was cleaned with a dockage testing machine, and the cleaned grain was stored at 5°C before use.

Glucosylase (from *Rhizopus* mold); glucose oxidase (type II, from *Aspergillus niger*); peroxidase (type I, from horseradish); protease (type II, from *Aspergillus oryzae*); tris[hydroxymethyl]aminomethane, and corn starch were purchased from Sigma Chemical Co., St. Louis, MO. *o*-Dianisidine dihydrochloride was purchased from Aldrich Chemical Co., Milwaukee, WI. All chemicals were reagent-grade unless otherwise specified.

General Methods

All analyses were done in duplicate, except those for grain hardness, which were done in triplicate. Moisture was assayed by oven-drying for 1 hr at 130°C (Method 44-15A); protein by Kjeldahl nitrogen (Method 46-13); ash by dry combustion (Method 08-01), and fat by extraction with petroleum ether (Method 30-25) (AACC 1995). Total starch content was determined by AACC Method 76-11, except gelatinization of starch (0.5 g) was accomplished in 1M sodium hydroxide (40 ml) at 25°C rather than by high-pressure cooking. Color was measured by a tristimulus meter with readings given in the *L* a* b** spherical color space (Minolta, model CR-210, Minolta, Osaka, Japan). Before measuring color, grain was ground with a Falling Number laboratory mill at a setting of zero (Perten Instruments AB, Huddings, Sweden). Total titratable acidity in steep liquor (10 ml) was measured by titration to pH 7 with 0.01M sodium hydroxide, and pH was determined by a pH meter (model 350, Corning Inc., New York, NY). The concentration of sulfur dioxide (SO₂) in steepwater (10 ml) was measured directly by iodometric titration with 0.16N iodine (Eckhoff and Okos 1983). Whenever the steepwater had pH > 3, it was acidified with acetic acid (0.2M) before SO₂ assay.

Endosperm hardness of grain sorghum was estimated by particle size index after grinding using the method of Kirleis and Crosby (1982), and by floatation in a sodium nitrate solution with a density of 1.32 g/ml (Hallgreen and Murty 1983). Decortication of grain sorghum (unknown grade, 13.7% moisture) was done on

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²Graduate research assistant and professor, respectively, Department of Grain Science & Industry, Kansas State University, Manhattan, Kansas 66506-2201.

³Present address: Department of Agricultural Engineering, University of Illinois, Champaign-Urbana, Urbana, IL 61801.

⁴Corresponding author. Fax: 913/532-7010.

two vertical VRM Satake mills operated in tandem (Satake Engineering Co. Ltd., Hiroshima, Japan) at a grain flow rate of 22 kg/min. The first, third, and fifth passes were through machine 1, and the second and fourth passes were through machine 2. Abrasion of the kernels was effected by carborundum-coated (grit Nos. 24 and 33) rings on the rotors, whereas attrition was effected by back-pressure applied to the grain and slotted screens (Munck 1995).

Pasting curves were recorded on a Brabender Viscograph-E (C. Brabender Instruments, Inc., Hackensack, NJ) fitted with a torque sensor of 700 g-cm (Tipples 1980). A starch slurry at 7.5% solids (dry basis) in water (465 ml) was heated from 30 to 95°C at 1.5°C/min, held at 95°C for 60 min, and then cooled to 30°C at 1.5°C/min. The temperature to reach the peak consistency immediately after the pasting temperature was noted, and the rate of reduction in consistency (shear thinning) from the peak to the consistency after 1 hr at 95°C was calculated (Subrahmanyam and Hosney 1995).

Wet-Milling Procedure

A series of starch isolations were done after steeping the yellow No. 2 sample in 0.20 and 0.25% SO₂ (initial concentrations) at 50 and 58°C for 2–10 hr. One sample was steeped in 0.25% SO₂ at 58°C for 72 hr. Decorticated grain sorghum was steeped in 0.25% SO₂ at 58°C for 4, 6, and 10 hr, and the starch was isolated. Two replicates were run for each condition.

The procedure with a 4-hr steep at 58°C in 0.25% SO₂ is described to illustrate the method. A solution of sodium hydrogen sulfite (0.61 g) in water (150 ml) was prepared in a 500 ml Erlenmeyer flask, and the 0.25% concentration of SO₂ was confirmed by iodometric titration (100% of theory). Yellow No. 2 grain sorghum (100 g, dry basis) was added, and the flask was plugged with a two-holed rubber stopper equipped with a thermometer and fitted with a short length (4 cm) of plastic tubing. The mixture was maintained at 58°C in a water bath with gentle shaking. After

4 hr, the steepwater was drained, and its pH, total titratable acidity, and SO₂ concentration were measured. Fresh water (150 ml) was added to the steeped grain, and the mixture was ground for 1 min at low speed (15,500 rpm) and for 2 min at high speed (22,000 rpm) in a Waring blender (model 7010G, Waring Products Division, New Hartford, CT). The blade of the blender was reversed so that its blunt edges impacted the grain. The slurry at ≈38°C was poured onto a 80-wire mesh screen (opening 180 μm), and the overs were washed with water (3 × 50 ml). The combined throughs were placed atop a two-tier stack of sieves, the top being a 200-wire mesh (opening 75 μm) screen, and the bottom a 400-mesh nylon bolting cloth (opening 40 μm). The overs on both sieves were washed with water (1 × 30 ml, and 1 × 20 ml). The throughs were then combined and centrifuged at 12,000 × g for 20 min, the supernatant layer was discarded, and the tailings layer was scraped off and combined with the overs from the sieving steps. That mixture, together with some additional tailings, formed the by-product stream. The bottom sedimented layer was slurried with water (30 ml), the slurry was centrifuged at 12,000 × g for 20 min, and the process was repeated twice. The supernatants from the washing steps were discarded, and the tailings were added to the by-product stream. The sedimented starch was dried in an oven at 40°C overnight, and was assayed for moisture, protein, and color.

Modeling Experiment

Response surface analysis was used to obtain the multiple correlation coefficients in the equation for the influence of steeping temperature (50, 55, 60, 65, and 70°C) and the initial SO₂ concentration (0, 0.1, 0.2, 0.3, 0.4, and 0.5%) on starch recovery. Steep time, the amount of grain, and the ratio of steep liquor to grain were kept constant at 4 hr, 100 g (dry solid), and 1.5:1.0 (w/w), respectively. A computer program (SAS 1989) provided predictions for untested values, and the response surface contour maps

TABLE I
Composition, Moisture, and Hardness of Starting Materials for Wet Milling

Material	Component ^a (% dwb)				Moisture %	Hardness ^{b,c}	
	Starch	Protein	Fat	Ash		PSI	PF
Dent corn	74.3	9.4	3.8	1.4	12.6
Grain sorghum, yellow No. 2	77.5	10.6	3.3	1.5	13.0	51	54
Grain sorghum, yellow (?) ^d	76.9	9.6	2.1	1.5	14.5	52	61
Decorticated grain sorghum ^e	88.7	8.8	2.1	0.5	13.7

^a Least significant differences typical for the assays are given in Table II.

^b Least significant differences for PSI and PF were 0.6 and 1.5, respectively.

^c PSI = particle size index after grinding. PF = percentage of kernels floating in a sodium nitrate solution with a density of 1.32 g/ml at 25°C.

^d Commercial sample, grade is uncertain.

^e After five passes through one of two Satake mills in tandem.

TABLE II
Decortication of Grain Sorghum and Composition of Fractions

Passes ^a	Mill		Yield ^b %	Bran							Decorticated Grain								
	1	2		Color ^c			Component ^d				Color			Component					
				L*	a*	b*	MC	P	Starch	Fat	Ash	L*	a*	b*	MC	P	Starch	Fat	Ash
G	0	0	14.5	9.6	75.6	2.1	1.5	71.5	3.1	11	14.5	9.6	75.6	2.1	1.5
1	1	0	4.5	63	4.3	10	11.7	10.1	46.4	4.9	3.6	74.4	2.7	11	14.3	8.8	76.2	2.0	1.5
2	0	1	6.3	64	4.9	13	11.3	11.7	41.4	6.3	4.0
3	1	0	10.7	69	3.9	11	11.2	11.5	57.4	7.0	3.7	79.6	1.5	11	13.9	8.1	83.9	1.1	0.8
4	0	1	5.1	73	3.4	11	11.0	10.2	67.8	3.7	2.1	81.1	1.1	11	13.6	8.1	84.5	0.8	0.6
5	1	0	5.6	77	2.5	9	10.9	9.2	77.8	2.7	1.8	82.7	0.5	12	13.7	8.8	88.7	0.7	0.5
LSD ^f				0.3	0.2	0.5	0.4	0.6	2.1	0.7	0.2	0.22	0.15	0.5	0.7	0.6	2.3	0.02	0.06

^a G = grain sorghum, 1–5 = products at sequential levels of decortication.

^b Yields of bran calculated based on initial weight of grain sorghum. Fiber levels in the fractions were not determined.

^c L* = brightness, a* = redness, b* = yellowness.

^d MC = moisture content. P = protein.

^e Sample lost.

^f P = 0.05.

showed the estimated optimum conditions. Data were evaluated statistically using the one-way analysis of variance procedure with the least significant difference test (SAS 1989).

Removal of Protein from Starch

To remove contaminating protein from sorghum starch, the wet starch isolated from 100 g of grain was mixed with water (30 ml), and the slurry was cooled in an ice bath and stirred vigorously for 120 sec using a tissue homogenizer (model SDT99708, 170W, Tekmar Co., Cincinnati, OH). The sheared slurry was centrifuged at $12,000 \times g$ for 20 min. The supernatant and tailing layers were discarded, and the starch was collected and dried in an oven at 40°C .

RESULTS AND DISCUSSION

Grain Sample

The grain sorghum samples were of similar composition, and both had kernels of intermediate hardness (Table I). Decortication of one of the grain sorghum samples after five passes through a decortivating machine removed $\approx 30\%$ of the outer portion of the kernels. This sample, decorticated-5 grain, appeared free of pericarp. The starch level in this sample was increased by 17%, and its protein was reduced by 8% (Table II). Almost all the kernels were broken during decortication, and the size distribution of the decorticated-5 grain was: over a No. 6 wire mesh, 3.2% (non-broken kernels); 7 mesh, 40.8%; 8 mesh, 26.9%, and 9 mesh, 14.8%. The five bran fractions collected at each pass contained an ever-increasing level of starch after the second pass (Table II). The germ was concentrated in the bran from the passes 1 through 3 as indicated by the fat levels. The brightness (L^*) of the decorticated grain, after grinding to flour size, improved markedly after pass 3 (Table II).

Effect of Grinding Procedure

Watson (1970) reported a 85–90% recovery of starch by laboratory wet-milling of sorghum grain that had been steeped 40–50 hr at $48\text{--}52^\circ\text{C}$ in 0.1–0.16% SO_2 . We used a laboratory wet-milling procedure (Fig. 1), where starch was purified by centrifugation instead of tabling, and where fine grinding was done with a Waring blender rather than with a plate mill. A sorghum sample that had been steeped exhaustively in 0.25% (initial concentration) SO_2 for 72 hr at 58°C was separated from the steeping liquor and placed in water (Fig 1). That mixture was ground 10 times in a Waring blender over a 30-min period at 30°C . The exhaustive grinding gave a 81% recovery of sorghum starch (1.8% protein) from the yellow No. 2 sample according to the scheme in Figure 1. If the same steeped grain sorghum was ground twice in the blender for a total of 3 min (Fig. 1), then starch recovery was 66%, whereas dent corn steeped and ground under the same conditions gave 78% starch. Those results show that the recovery of sorghum starch in this work was dependent on the grinding time. We chose the two-step grinding (3 min) procedure for the remainder of the work.

Wet-Milling of Grain Sorghum with a Short Steeping Period

Steeping grain sorghum at 50°C in 1.5 parts of 0.2% aqueous SO_2 (initial concentration) gave a sharp increase in starch yield after 2 hr, but only a slight increase over the next 8 hr of steeping (Fig. 2). Elevating the steep temperature from 50 to 58°C increased starch yield from 45 to 50%, whereas increasing the initial concentration of SO_2 from 0.20 to 0.25% did not increase yield. The SO_2 concentration declined with steeping time, principally because the steeping container was vented to the atmosphere. Starch yield from our samples of grain sorghum appeared largely independent of SO_2 when its initial concentration was $>0.20\%$ in the steepwater, indicating an excess of SO_2 to soften the grain in the short (<10 hr) steep period. Eckhoff and Tso (1991a,b) found that adding lactic acid and protease to the sulfited steepwater of corn and corn grits, respectively, improved recovery

of corn starch. Those additives were not tried in this investigation, although Watson and Hirata (1955) employed lactic acid with sulfite in a 48-hr steep of grain sorghum.

Steepwater uptake at 58°C was somewhat faster initially for the grain sorghum sample than for the dent corn sample, in agreement with Fan et al (1963). But after 5 hr of steeping, the uptake by grain sorghum was ≈ 4 percentage points below that of corn (Fig 3A). Fully steeped corn usually has a moisture content of $\approx 45\%$ (Blanchard 1992). The acidity was always higher in the sorghum steepwater than in corn steepwater, as shown by pH and titratable acidity (Fig 3B). That difference in acidity was not caused by differences in SO_2 concentration, because titration of SO_2 showed that, after 2 hr, the steepwater of grain sorghum contained less SO_2 than did corn (Fig 3A). In a laboratory countercurrent steep-

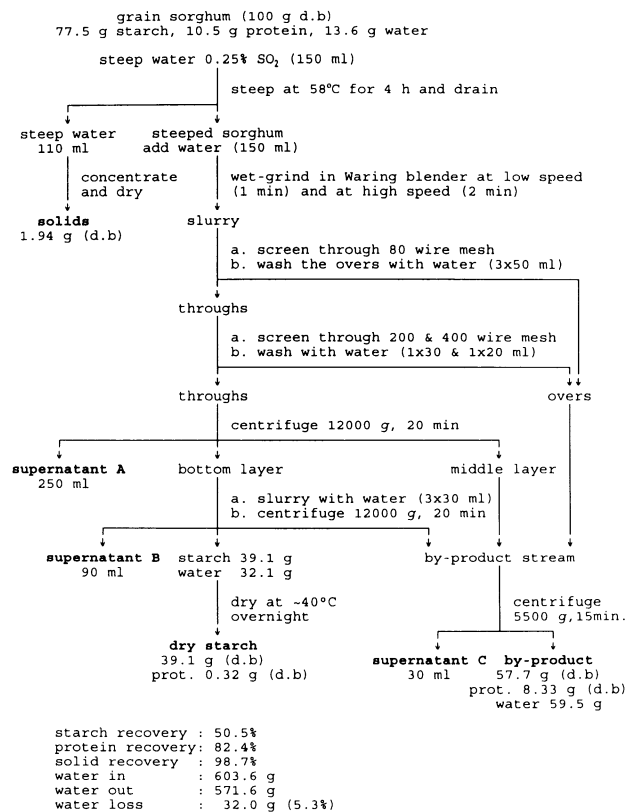


Fig. 1. Wet milling of grain sorghum using a short steeping period.

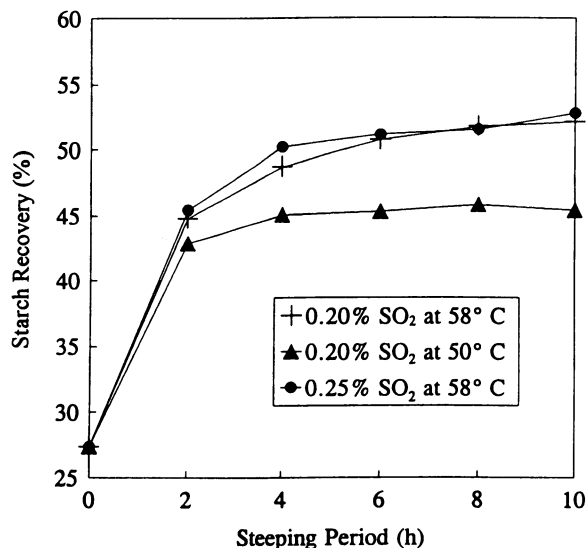


Fig. 2. Starch recovery after steeping grain sorghum in 1.5 parts (by weight) of steepwater to grain. Levels of SO_2 are the initial levels in the steepwater.

ing experiment on grain sorghum at 50°C starting with steepwater containing 0.1% SO₂ at pH 3.1 (Watson et al 1951), the SO₂ level declined to 0.078% after 8 hr, while the pH rose to 3.6 and the titratable acidity rose to 0.5% lactic acid (0.055 meq/ml).

Venting the steeping grain caused the SO₂ concentration in our samples to decline with steep time, which also occurs in the counter-current steeping of grain practiced commercially (Watson 1984, Blanchard 1992). During a 10-hr steep period at 58°C (Fig. 3), the SO₂ concentration in a blank sample containing no grain declined 78% compared to 76 and 70%, respectively, for grain sorghum

and corn. The disappearance of SO₂ was somewhat larger than the 50–60% lost after 8 hr in a batch steeping of 80 MT of corn done on commercial equipment with continuous circulation of liquor at 50–52°C and an initial SO₂ level of 0.10–0.15% (Watson et al 1955). The SO₂ remaining in the 10-hr steep liquor with our samples of grain sorghum and corn, on a dry basis, amounted to 0.90 and 1.12 g/kg, respectively, which constitutes a two- to threefold excess compared to the 0.2–0.4 g/kg of SO₂ absorbed by corn during commercial countercurrent steeping with 1.1 L/kg of 0.1–0.2% aqueous SO₂ (Watson 1967, Blanchard 1992). Of the SO₂ unaccounted for after the 10-hr steep (2.6–2.8 g/kg), the proportion absorbed by the grain sorghum is unknown.

The reason for the increased acidity of the steepwater and its consequences in grain sorghum is not clear, nor is it known that all grain sorghum would behave in the same manner. The titratable acidity (Fig. 3B) indicated that bacterial fermentation began in the grain sorghum steep after ≈6 hr, whereas no fermentation occurred in the corn steeped up to 10 hr. It seems plausible that fermentable solubles were leached from the small kernels of grain sorghum earlier than they were from corn (Watson 1970), and that the higher surface area of sorghum may have carried an extra larger inoculum. Furthermore, the concentration of the bacteriostatic SO₂ in the grain sorghum steep was below that in the corn steep after 1–2 hr (Fig 3A).

Those results indicate the initial bottleneck to starch isolation from grain sorghum is the diffusion of steep liquor into the kernels (Eckhoff and Tso 1991a). The second barrier is probably the high extent of crosslinking of sorghum protein (Watson and Hirata 1955). Increasing the steeping temperature to 58°C did give some improvement in accessibility of the protein matrix to sulfite (Fig. 2), but increasing steeping temperature to 65–70°C decreased starch recovery as shown in a 4-hr steep modeling experiment (Fig. 4). The decrease in recovery was attributed to gelatinized and swollen starch that began to blind the screens. Increasing the concentration of SO₂ beyond 0.3% during steeping also gradually decreased

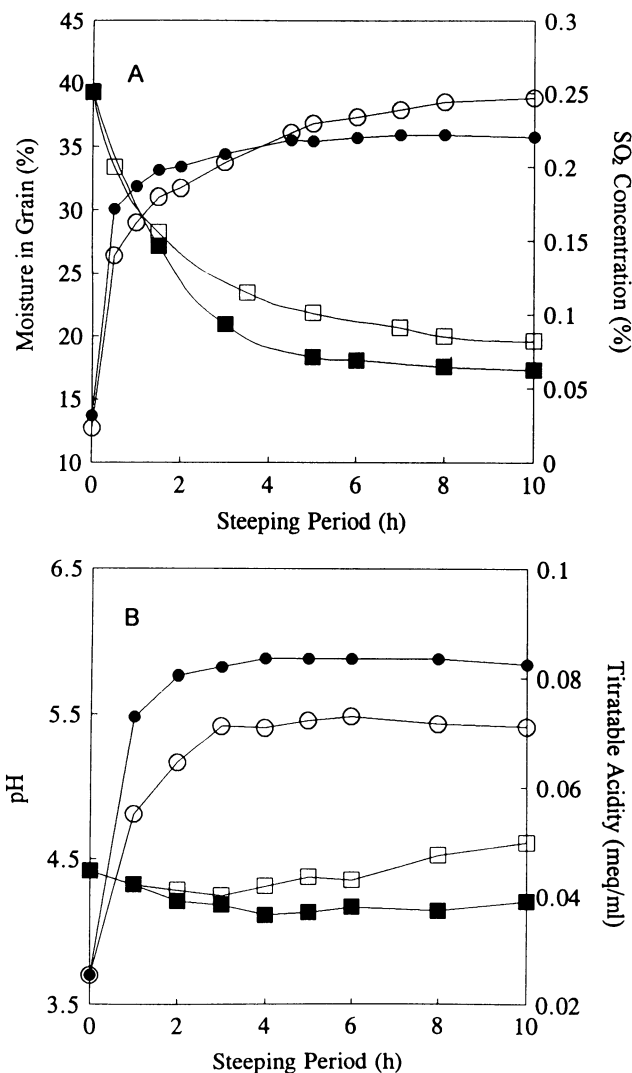


Fig. 3. Steeping of grain at 58°C in 1.5 parts (by weight) of steepwater. A, Moisture content in grain sorghum (●) and corn (○), and SO₂ in steepwater of grain sorghum (■) and corn (□). B, Steepwater pH of grain sorghum (●) and corn (○), and titratable acidity (alkali equivalents per 10 ml of steepwater) in grain sorghum (■) and corn (□).

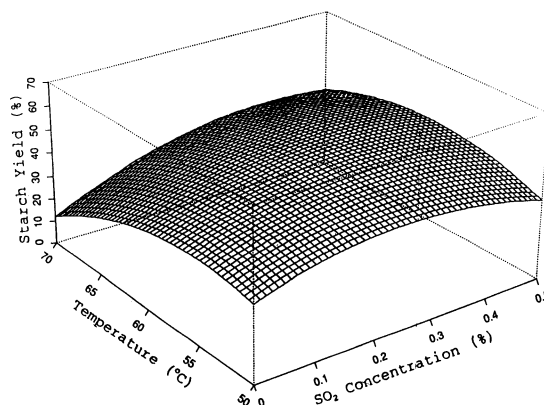


Fig. 4. Starch recovery in the wet milling of grain sorghum (yellow No. 2) using a 4-hr steep with a steepwater-to-grain ratio of 1.5:1.0 (w/w).

TABLE III
Protein, Color, and Pasting Properties of Sorghum and Commercial Corn Starches

Starch	Steeping Method	Protein (%)	Color ^a			Peak Temp. (°C)	Consistency (BU)			
			L*	a*	b*		Peak	95°C	After 1 hr ^b	Breakdown ^b
Corn	Commercial	0.31	94.5	-2.8	3.8	89.0	460	360	250	210
Sorghum 1	Wet grind with water ^c	0.83	92.3	-0.8	4.4	89.0	580	480	360	220
	2 4 hr, 58°C, 0.25% SO ₂	0.79	93.2	-1.2	3.5	90.5	540	450	300	240
	3 24 h, 58°C, 0.20% SO ₂	0.80	92.6	-1.0	3.9	90.0	470	400	290	360
	4 Decorticated, 4 hr, 58°C, 0.25% SO ₂	0.74	93.9	-1.1	2.3	90.5	620	490	260	360

^a L* = brightness, a* = redness, b* = yellowness.

^b At 95°C.

^c Ping and Seib (1995).

starch recovery, perhaps by acid-catalyzed hydrolysis of starch. The optimum recovery of sorghum starch in a 4-hr steep at 58°C with initial concentration of 0.31% SO₂ was 51% (Fig. 4). Zipf et al (1950) investigated starch recovery from grain sorghum steeped for 65 hr. They found sorghum starch recovery was independent of SO₂ concentration between 0.1–0.25% at 47°C, but was reduced dramatically if steeping temperature was 60–65°C at 0.25% SO₂.

Wet Milling and Color of Sorghum Starch

Starch recovery from the decorticated grain sorghum after steeping at 58°C in initial concentration of 0.25% SO₂ for 4, 6, and 10 hr was 50.9, 51.7, and 55.9%, respectively, which was comparable to the recovery of starch from whole grain (Fig. 2). Because of dry milling losses of starch, starch recovery based on grain was reduced ≈3% when decortication was used. Freeman and Watson (1969) found that wet-peeling of grain sorghum facilitated the separation of sorghum starch and protein, but some starch was lost in that process. Zipf et al (1950) concluded that pearling grain sorghum before exhaustive wet-milling was impractical due to losses of starch with the bran.

Starch from the decorticated sample had approximately the same brightness ($L^* = 93.9$) as that of the control ($L^* = 93.2$) (Table III), although it had less color, as indicated by a somewhat lower b^* value. None of the sorghum starches were as bright as commercial corn starch. Extraction of the sorghum starch with 70% ethanol at 25°C slightly improved brightness (data not shown). Zipf et al (1950) reported whiter starch from pearled grain sorghum.

Pasting Characteristics and Removal of Contaminating Protein

Sorghum starch isolated without use of SO₂ (Yang and Seib 1995) had a higher hot-paste consistency when compared to starches that had been isolated in the presence of sulfite including commercial corn starch (Table III). Traces of sulfite are known to reduce the swelling volume and, therefore, the consistency of a hot starch paste at 95°C (Paterson et al 1994), even though corn starch was shown to be the least sensitive to sulfite. Presumably, sorghum starch also would have low sensitivity. Reduced paste consistency might also be due to the acidity of SO₂ solution, which catalyzed depolymerization of starch.

The hot pastes of our samples of sorghum starch did not undergo excessive shear thinning (Table III) as previously observed for other samples (Subramanian and Hosney 1995). The breakdown in consistency upon stirring the hot pastes of the sorghum starch for 1 hr at 95°C almost equaled the breakdown observed for commercial corn starch, except that for the starch isolated from decorticated grain sorghum. At 50°C, the paste consistency of cooked sorghum starch was equal to that of commercial corn starch. High-shear treatment of a slurry of a sorghum starch contaminated with 1.3% protein decreased protein by 50%.

CONCLUSIONS

Wet-milling of yellow grain sorghum with a steep at 58°C for 2–4 hr and an initial concentration of 0.25% aqueous SO₂ released only about half its starch, despite rapid hydration of the small kernels. Increasing steep temperature and SO₂ concentration reduced starch recovery. Decortication of the grain was not beneficial to starch recovery using the abbreviated-steep process. About one-half of the starch in grain sorghum appears associated with crosslinked proteins.

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