Fractional Composition of Grain Sorghum (Sorghum bicolor) After Wet-Peeling in a Centrifugal Pump

K. R. Lochte-Watson
Gilroy Foods

Curtis L. Weller
University of Nebraska-Lincoln, cweller1@unl.edu

Kent M. Eskridge
University of Nebraska-Lincoln, keskridge1@unl.edu
**Fractional Composition of Grain Sorghum (Sorghum Bicolor) after Wet-peeling in a Centrifugal Pump**

K. R. Lochte-Watson, C. L. Weller, K. M. Eskridge

**Abstract.** Use of a centrifugal pump to peel and facilitate separation of grain sorghum kernels into three fractions was investigated. Special emphasis was given to separating fractions to achieve a high concentration of wax in one fraction and a high concentration of starch in another. Trials were conducted to determine the effects of both soak time and recirculation time on wet-peeling. After peeling, three fractions, peeled kernel, bran, and suspended solids, were collected. Together, the bran and suspended solids fractions contained 12.7% of the initial total dry solids, 90% of initial wax, and 8.1% of the initial starch. Peeled kernel fraction accounted for 84% of the initial dry material, which contained 94% of the initial starch with 8.5% of the initial wax. Circulation through the pump for 5 min resulted in over 90% of the wax being concentrated in bran and suspended solids fractions, and over 90% of the starch was recovered in peeled kernel fractions. Soaking for 10 min versus no soaking increased the starch available in the peeled kernel fraction to 94%. Circulation time through the centrifugal pump had a more significant effect on separation of constituents than the soak periods studied.

**Keywords.** Milling, Bran, Decortication, Wax, Starch, Protein.

Sorghum wax, or more specifically, precipitated hexanes extract is found on the outer layer or epicarp of sorghum kernels at levels of 0.20 to 0.25% d.b. (Weller et al., 1998; Lochte-Watson et al., 1996). At such low levels, recovering wax from whole kernels would not be commercially viable unless the rest of the kernel is utilized. Higher economic returns could be realized if wax was recovered as a co-product from a current commercial process. Additionally, extracting wax from bran fractions as opposed to whole kernels would reduce the amount of material handled.

Sorghum wax has been shown to be similar to cannauba wax, an extract from Brazilian palm leaves (Kummerow, 1946a; Bunger and Kummerow, 1951; Cannon and Kummerow, 1957; Freeman and Watson, 1969; Weller et al., 1998). In addition to hexanes (Kummerow, 1946a; Kummerow, 1946b; Bunger and Kummerow, 1951; Kehm, 1951; Hsu, 1955; Cannon and Kummerow, 1957; Dalton and Mitchell, 1959; Freeman and Watson, 1969; Lochte-Watson et al., 1996; Weller et al., 1998) used to remove lipid material from the outer layer of sorghum, petroleum ether (Sariava, 1995), chloroform (Kummerow, 1946b; Bianchi et al., 1979; Avato et al., 1990), benzene (Hubbard et al., 1950; Seitz, 1977; Bianchi et al., 1979; Avato et al., 1990), and trichloroethylene (French, 1948) have been used. Hsu (1955) compared extraction yields of wax and oil from sorghum bran using a Soxhlet extractor with the following solvents: hexanes or Skellysolve B, acetone, methyl ethyl ketone, secondary butyl alcohol, n-butyl ether, ethylene dichloride, methanol, and urea. Hsu found absolute ethanol recovered the most wax.

Grain sorghum is most often dry ground or flaked for animal feed. It is also used as a source of starch for ethanol production. In ethanol production, whole kernels are ground, wetted, and then cooked to gelatinize starch. Starch is then broken down enzymatically into sugars, which are subsequently converted to ethanol by yeast. Non-starch materials such as lipids, proteins, cellulose, and other minor constituents of the kernel make up distiller’s dried grain, a co-product of ethanol production.

Lochte-Watson et al. (1996) studied abrasive decortication of sorghum kernels and its effects on wax yield of removed bran. They observed that 75% of the total available wax was recovered from abrasively removed bran fractions containing over 10% total available starch. As a result, they judged abrasive decortication to be ineffective for bran removal from which wax would be recovered.

Other investigators have studied peeling (Morgan et al., 1964; Freeman and Watson, 1969) and wet-milling (Zipf et al., 1950; Watson, 1970) of sorghum to separate bran from the endosperm. Freeman and Watson (1969) developed a method for removing bran from whole sorghum kernels by peeling kernels using a Waring® blender. Preliminary investigations by the authors using Freeman and Watson’s (1969) method to separate bran and endosperm fractions, found large amounts of wax concentrated in the bran fraction. A major drawback of Freeman and Watson’s (1969) method was that it only processed 100 g (3.5 oz) of...
kernels at a time, leaving the need for a more scalable method to peel whole sorghum kernels. Initial lab work by the authors found circulating sorghum kernels through a pumping system showed promise for peeling bran off whole kernels.

An industrial wet-milling grain sorghum plant was operated by the Corn Products Company in Corpus Christi, Texas, from 1948 to the late 1970s. The plant produced sorghum gluten meal, refined oil, glucose, dextrin, and dry starch (Watson, 1970). In this process, germs were removed first by a plate mill, then the kernel was ground in an attrition mill, followed by the ground pericarp being separated with screens.

The objective of this research was to explore the use of a centrifugal pump to peel sorghum. The effects of precirculation soak time and circulation time on the yield of grain sorghum constituents recovered in kernel fractions were determined. Constituents of dry matter, starch, protein, ash, wax, and residual hexanes extract (RHE) were evaluated for peeled kernel, bran, and suspended solids fractions.

**MATERIALS AND METHODS**

**KERNEL PREPARATION**

*Sorghum bicolor, cv NC+7R37E,* was mechanically harvested in October 1996 at Mead, Nebraska. Sorghum kernels were cleaned and dried to 11.6% MC (w.b.) by the Nebraska Foundation Seed Division, and then stored in paper bags at 10°C (50°F) until used.

**EQUIPMENT SETUP**

Sorghum kernels and water were circulated through a system as shown in figure 1. The recirculating system consisted of a stainless-steel, funnel-shaped hopper; a Pacer centrifugal S-series pump (Model No. S2A450-698-PED, Pacer Pumps, Lancaster, Pennsylvania) equipped with a 1.5 kW (2 hp) Baldor industrial ac motor (rated at 3450 rpm), and a wire-reinforced hose. The hopper had a 5-cm (2-in.) diameter discharge outlet. A 90° elbow was attached to the discharge outlet, followed by a 0.3-m (1-ft) transfer pipe leading into the pump. The pump housing held a check valve preceding a three-vaned impeller with a center height of 1.27 cm (0.5 in.) and an outer edge height of 0.64 cm (0.25 in.).

From the pump, sorghum and pump water were returned to the hopper via a 1.8 m (6 ft) of 5-cm (2-in.) ID flexible hose. The pump had a measured water pumping rate of approximately 380 L/min (100 gpm). The recirculating system with hose outlet positioned at the bottom of the hopper for optimum operation had a capacity of about 15 L (4 gal). If the hose outlet was not positioned at the bottom of the hopper, a vortex formed in the hopper and the pump operated at less than full capacity.

**PEELING IN PUMP**

The hopper was first filled with 3.5 kg (7.7 lbs) of sorghum kernels at 11.6% MC (w.b.) and 17.5 L (4.6 gal) of water at 20°C (70°F) for a total volume of 20 L (5.3 gal). During recirculation, a constant liquid level of approximately 4 L (1 gal) was maintained in the hopper. Tests were conducted to determine the effects of both soak time and recirculation time on wet-peeling. Kernels were soaked for either 0 min or 10 min prior to being pumped through the system. During recirculation, the pump was operated for either 3 min or 5 min, which resulted in kernels passing through the pump roughly 70 or 120 times, respectively, based on the water pumping rate of 380 L/min (100 gpm), and a system capacity of 16 L.

After the kernels were circulated through the pump, the pump was turned off and the free-end of the flexible hose was moved into a 175 L (45 gal) collection vessel. The solution was then pumped into the collection vessel, followed by flushing of the recirculating system with 20 L (5 gal) of water. The free-end of the hose was then placed at the floor drain, the pump drain plug removed, and the pump was further flushed and drained to remove any residual sorghum particles and water before the next trial was performed. Further analysis showed that over 90% of the sorghum dry matter placed in the hopper for peeling was collected in the collection vessel before the pump was drained to the floor. Three fractions (bran, peeled kernels, and suspended solids) were produced during recirculation.

**SEPARATING FRACTIONS**

After collecting the sorghum-water solution in a large collection vessel, the three fractions were then separated from each other. The bran was removed first. This was accomplished by agitating the solution to suspend the bran, followed by skimming the pump or “milk” water and bran off the top. The skimmings were poured onto a standard U.S. No. 40 sieve, retaining the bran on the sieve while the water and suspended starch passed through. Peeled kernels in the skimmings were removed by hand and placed back into the large collection vessel. “Milky” rinse and pump water was retained for collection of suspended solids fraction. Bran removal was continued as above until all visible bran was collected. The cleaner rinse water collected after rinsing the bran fraction was used to rinse the peeled kernels.

Peeled kernels were collected on a standard U.S. No. 16 sieve then rinsed for 15 s to remove small particles such as germs, small broken kernels, or small particles of bran which could pass through the strainer. The small particles were collected on the standard U.S. No. 40 sieve and added to the bran fraction. The rinse and pump water collected...
during the process was added to the rinse and pump water collected during bran removal. A total of about 50 L (13 gal) of rinse water was added to the initial 17.5 L (4.6 gal) of pump water from which the suspended solids were collected.

The suspended solids fraction was collected by sedimentation. First, the pH of captured rinse and pump water was lowered from 7 to 5 with 30 mL (1 oz) of lactic acid. After storage at 5°C (40°F) for 12 h, a red-tinted supernatant was decanted off and the settled slurry was dried to less than 12% MC (w.b.) in a forced-air dryer at 35°C (100°F) in 12 h. After drying, the hard cake-like mass was weighed and its moisture content was measured in duplicate using AOAC method 925.10 (AOAC, 1990). The cake-like mass of suspended solids was ground for approximately 30 s in a household coffee grinder (Chefmate Model GC-6000, Dayton Hudson Corp., Minneapolis, Minnesota) before wax extraction and proximate analysis were performed. Suspended solids fractions were then stored at 10°C (50°F) for 14 to 21 days until wax extractions and proximate analyses were completed.

Bran and kernel fractions were weighed, and moisture content was measured in duplicate using AOAC method 925.10 (AOAC, 1990). They were then dried at 50°C (120°F) for 36 to 48 h with stirring every 5 to 7 h. Samples were stored at 10°C (50°F) for 14 to 21 days until analyzed for wax, starch, protein, ash, and moisture contents.

### PROXIMATE ANALYSIS

Approximately 10-g sub-samples were used for moisture content, ash, protein, and starch analysis for all fractions and whole kernels. Samples were ground to 0.5 mm (0.02 in.) diameter in a Cyclotec 1093 Sample Mill (Tecator, Herndon, Virginia). Ash content was determined in triplicate using AOAC method 923.03 (AOAC, 1990). Protein content was determined in triplicate using a Kjeltech automated system (Tecator Inc., Herndon, Virginia) following AOAC method 920.53 (AOAC, 1990). Protein content was estimated as percent N times 6.25. Starch content was determined in triplicate using the Total Starch Assay Procedure (Megazyme International, Wicklow, Ireland) following AACC method 76-13 (AACC, 1995). Moisture content was determined in triplicate using AOAC method 925.10 (AOAC, 1990). Values were reported as percent component of sample dry matter or converted to percent component recovered of total available. Values of percent component (dry matter, starch, protein, ash or wax) of total available were the weight of component in fraction (bran, peeled kernel or suspended solids) divided by the weight of component available in whole kernel (dry basis, d.b.) from which the fraction was derived times 100%.

### WAX EXTRACTION

Wax was extracted from each fraction using a solvent containing a minimum of 95% n-hexane, called hexanes (Product No. N169-01, J.T. Baker, Phillippsburg, New Jersey). The hexanes/sorghum mixture was refluxed for 30 min at a ratio of approximately 500 mL (17 oz) hexanes to either 400 g (14 oz) of peeled kernels or whole kernels, or 40 g (1.4 oz) of bran or suspended solids. After refluxing, the mixture was vacuum filtered through a commercial coffee/tea filter (Product No. 20100, Bunnomatic Corp., Springfield, Illinois) held down by a flat stainless-steel washer [ID-12.5 cm (4.9 in.), OD-15 cm (5.9 in.)], on top of a Whatman No. 2 filter paper [(dia. 15 cm (5.9 in.))] on a Büchner funnel [ID-15.5 cm (6.1 in.)]. Larger, coarse particles were captured on the coffee/tea filter and with the stainless-steel washer, the filter prevented them from bypassing around the finer filter paper and entering the filtrate. The filtrate was stored at -20°C (40°F) for 24 h, allowing a precipitate to form. It was then vacuum filtered through a hexane-rinsed, 24-h air-dried Whatman No. 50 paper [9 cm (3.5 in.)] on a Büchner funnel [9.5 cm (3.7 in.)]. The process of cold storing and vacuum filtering was repeated two more times. The weight of wax or precipitated hexanes extract was considered to be the total weight collected on the three hexane-rinsed, 24-h air-dried Whatman No. 50 papers. Wax values for each of the 84 samples are a mean of two extractions.

A second portion, termed residual hexanes extract (RHE), recovered similarly to “foots oil” found when refining petroleum waxes (International Group Inc., 1999), was also quantified. This material was primarily lipid material which did not precipitate from hexanes at low temperatures. It was recovered by evaporating the hexanes from the filtrate collected after removing the wax. The value was a mean of two extractions.

### EXPERIMENTAL DESIGN AND STATISTICAL ANALYSIS

The experiment was conducted as a randomized complete block design with a 2 × 2 (circulation time × soak time) factorial treatment structure with seven blocks. Four whole kernel samples were taken from each of seven different bags, where each bag served as a block. Each whole kernel sample from each bag was processed separately using one of the four circulation time × soak time treatment combinations. A total of 28 samples (four treatments × seven blocks) were processed, creating three fractions for each sample. Each fraction and total recovered constituents were analyzed separately. Proc GLM (SAS, 1996) was used to evaluate main effects for circulation time and soak time, and their interaction. Dependent variables were: dry matter, starch, protein, ash, wax, and residual hexanes extract (RHE). These dependent variables were analyzed for each fraction and the total recovered for each constituent. Total recovered constituent was calculated by adding each percent component recovered in each fraction of total available. A value of 100% meant all of the constituent measured in the whole kernel (not peeled) samples was accounted for between the three fractions. Pair-wise t-test comparisons were used to determine significant (p < 0.05) differences of means for significant main effects and interactions.

### RESULTS AND DISCUSSION

#### FRACTION COMPOSITION

Whole kernel grain sorghum (NC+7R37E) samples, on average (n = 7), contained 75.0 ± 3.0% starch, 9.5 ± 0.3% protein, 1.4 ± 0.1% ash, 0.23 ± 0.01% wax, and 0.20 ± 0.02% residual hexanes extract (RHE) (d.b.). Fat and fiber were not measured and likely accounted for the remaining weight. Whole grain sorghum composition used in this
After circulating for 3 and 5 min, peeled kernel fractions accounted for a maximum 94% and a minimum 80% of the total available dry matter recovered. After wet-peeling, the bran fraction accounted for 1.5% to 4.6% of the total dry matter recovered. Chemical compositions of each fraction recovered after soaking for 10 min and a circulation time of 5 min are listed in Table 1.

Over 90% of total dry matter, starch, protein, and wax available in initial whole kernels were recovered between the three fractions after recirculation for all 28 treatments. Up to 10% of the original total dry matter was lost during purging residual kernels from pump and collecting, drying, and moving of fractions between various containers, screens, and sample bags. Total available ash recovered between the three fractions was from 60% up to 95% for circulation times of 5 to 3 min, respectively. The large loss of ash after 5 min of circulation time was attributed to the particles which did not settle from the rinse and pump water when the suspended solids were collected.

**RESIDUAL HEXANES EXTRACT (RHE)**

RHE is composed of lipids found throughout the kernel, especially in the germ. A 30 min hexane reflux period extracted an average 0.2% RHE from the whole kernels. Because of the intact state of whole kernels, a 30-min reflux period was not long enough to remove all the lipid material. For the smaller, detached particles, 30 min of exposure to solvent removed as much as four to seven times more, or 0.75 to 1.3% RHE from all the fractions together, than was removed from intact whole kernels. More RHE lipids were extracted from each fraction than the whole kernels because the various fractions had a smaller particle size with more exposed surface area per volume, smaller distances across which lipid diffusion occurred, and removed barriers. Serna-Saldivar and Rooney (1995) reported sorghum to have a fat content of 3.6% with 13.2% of the total fat found in the endosperm, 76.2% in the germ, and 10.6% in the pericarp. Percarp lipids are mostly wax, leaving about 3.3% fat to be extracted by hexane and termed RHE. Therefore, the methods used to extract wax from wet-peeled fractions in this article also removed almost one-half (1.5%) of the total lipids found in sorghum kernels.

**EFFECTS OF SOAKING AND CIRCULATION TIME**

The effect of soaking the kernels for 10 min versus no soak varied, depending on fraction and constituent. In general, soaking time had a significant (p < 0.05) influence...
(table 2) on bran and suspended solids fractions and total constituent recovered between the three fractions.

Circulation time had a significant (p < 0.05) impact on nearly all constituents for all fractions. Circulating for 5 min, or approximately 120 passes through the pump, resulted in concentrating over 90% of the total available wax recovered from the bran and suspended solid fractions, while leaving 90% of the total available starch in the peeled kernel fractions (table 2). Lochte-Watson et al. (1996) found when using a tangential abrasive dehulling device (TADD) to mill sorghum, only 58% of the original wax was recoverable from the abraded bran fraction leaving 30% of the wax on the kernel. Further milling to remove the wax would have increased the removed starch content to greater than 10% of the original starch. Comparatively, using a centrifugal pump to peel the bran separated the starch and wax into two separate fractions with less than 10% cross-contamination of wax and starch. In both cases the wax was concentrated to less than 15% of the initial dry matter.

After wet peeling in the pump, significant amounts of wax were found in both bran and suspended solids fractions. Wax located in the suspended solids fraction was likely a result of wax detaching from the bran as it fractured and broke. The wax, as found by Lochte-Watson and Weller (1999) and Lochte-Watson et al. (1996), breaks, flakes or grinds off with abrasion. Therefore, wax was recovered from both suspended solids and bran fractions.

Of the 24 dependent variables evaluated, only the following six had significant (p < 0.05) soak by circulation time interactions: recovered starch, protein, ash, and RHE for peeled kernel fractions, and starch and ash for total recovered component (table 3). There were no significant differences for samples circulated for 3 min, regardless of soak time. For samples circulated for 5 min, a 10-min pre-soak resulted in an increased recovery of constituents. Soaking was considered parallel to tempering in dry milling processes.

In dry milling of wheat, tempering or increasing the moisture content is generally performed to toughen the bran and soften the starchy endosperm (Mattern, 1991).

Processing Method Effect on Fractional Separation

During hand separation of fractions, the water/sorghum mixture was stirred and agitated with no specific control on the amount of agitation. Any extra agitation may have torn additional pericarp loose allowing it to be recovered in the bran fractions instead of the peeled kernel fractions. For samples circulated for 5 min, most of the bran was removed by the pump and separation methods did not affect the recovery of dry matter. For samples circulated for 3 min, pericarp and germ were still attached to the kernel and the varying amounts of agitation during separation increased the standard deviation for dry matter content of each fraction. This was a problem inherent to the laborious hand separation techniques and could be controlled using mechanical separation techniques.

Conclusions and Implications

Exploration of wet peeling sorghum kernels using a centrifugal pump resulted in separation of the kernel into three fractions; peeled kernel, bran, and suspended solids. Circulation time through a centrifugal pump had a more significant effect on separation of constituents than the soak periods studied. A soak time of 10 min affected the bran and outer edges of the endosperm as a longer soak time began to soften the endosperm considerably. Of the constituents measured, the amount of ash and RHE recovered were the most affected. Wet peeling in a centrifugal pump separated the starch from the wax. The bran and suspended solids fractions contained 12.7% of the initial total dry solids, 90% of initial wax, and 8.1% contamination of starch. The peeled kernel fraction accounted for 84% of the initial dry material, which contained 94% of the initial starch with 8.5% of the initial wax. Another 7% of the starch was recovered from the suspended solids fraction after wax extraction.

Further development of a process using a centrifugal pump or other shearing forces to peel bran from kernels should investigate tempering before peeling in the pump. Tempering may increase the toughness of the bran thereby

### Table 3. Treatment means for those constituents with significant (p < 0.05) soak time by circulation time interaction

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Constituent (%)</th>
<th>Soak, Circulation Time (min)</th>
<th>S.E.†</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0, 3</td>
<td>10, 3</td>
</tr>
<tr>
<td>Peeled kernel</td>
<td>Starch†</td>
<td>96.0 b</td>
<td>93.6 b</td>
</tr>
<tr>
<td>Protein</td>
<td>90.8 c</td>
<td>89.7 c</td>
<td>81.2 a</td>
</tr>
<tr>
<td>Ash</td>
<td>75.2 c</td>
<td>72.3 c</td>
<td>52.6 a</td>
</tr>
<tr>
<td>RHE</td>
<td>193 c</td>
<td>164 bc</td>
<td>121 a</td>
</tr>
<tr>
<td>Total</td>
<td>Starch†</td>
<td>99.1 ab</td>
<td>98.5 ab</td>
</tr>
<tr>
<td>Ash</td>
<td>80.5 c</td>
<td>79.8 c</td>
<td>61.4 a</td>
</tr>
</tbody>
</table>

* Each value is a mean of seven samples.
† Values are reported as percent constituent recovered in fraction over constituent available in whole sorghum on a dry basis. Whole sorghum kernel content was 75% starch, 9.6% protein, 1.4% ash, 0.23% wax, and 0.20% RHE.
‡ Standard error of the mean was the same for both parameters, soak time, and circulation time.
§ Means with the same letter in each row are not significantly (p > 0.05) different from each other.
¶ Residual hexanes extract.
allowing the bran to be removed in larger particles with less fracturing. Investigating the effect of circulating for an initial period of time, followed by soaking, and then additional circulation may yield additional information for a commercial type peeling process. Theoretically, the circulating, soaking, followed by circulating would cause an initial fracture in the bran, toughen it, then peel it off in one or two larger particles, respectively.

As circulating through a single pump would be inefficient, scaled-up processes should look at multiple pumps, venturis, or other methods which create turbulent flow. Processes which create shearing forces, simulating multiple passes through a centrifugal pump would emulate the process developed in this study.

Agitating during separation will cause additional material to be removed from the peeled kernel. The additional agitation during separation of fractions should be controlled. Suggested methods of separation include centrifugation and hydroclones after peeling.

Implementation of this method into an ethanol fermentation or flaking mill process could be accomplished for recovery of wax as a co-product of either process. The three fractions could be used as follows. The bran, after wax extraction, would be dried and used for animal feed or recovery of cellulose (fiber). The peeled kernels could either be flaked or ground for starch utilization. The suspended solids fraction could be used in a variety of ways depending upon the use of the starch and bran. It could be combined with the bran fraction, both extracted for wax, then used as animal feed. It could be extracted for wax separately, followed by starch utilization with the peeled kernel, pelletizing, or possibly extrusion.

Acknowledgments. The authors are grateful to Laurie Keeler at the University of Nebraska Food Processing Center for providing space and equipment to perform experiments and to Dr. Steven R. Eckhoff, Rene J. Shunk, and Larry Prueitt at University of Illinois, Agricultural Engineering Department and Corn Milling Pilot Plant for use of equipment and help to run initial trials.

References
French, R. O. 1948. The solvent extraction of wax from sorghum grain bran. M.S. thesis. Manhattan, Kans.: Dept. of Chemical Eng., Kansas State University.
Kehm, R. J. 1951. The solvent extraction of wax from sorghum bran. M.S. thesis. Manhattan, Kans.: Dept. of Chemical Eng., Kansas State University.
Kummerow, F. A. 1946b. The composition of the oil extracted from 14 different varieties of Andropogon Sorghum var. vulgaris. Oil & Soap 23(9): 273-275.