

NOTE

Thermal Properties of Corn Starch Extracted with Different Blending Methods: Microblender and Homogenizer¹

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Thermal properties of starch can be evaluated by using differential scanning calorimetry (DSC). DSC generates data of the thermal parameters of starch, such as onset temperature (T_o), peak temperature (T_p), and enthalpy of gelatinization (ΔH). These parameters may help determine the functionality of specific starches and, therefore, their utility in the food industry. Thermal properties of corn starch can also be used as criteria for selection of desirable lines for breeding purposes to obtain starch with specific properties useful in industry (Meuser et al 1989).

White et al (1990) reported the use of a small-scale wet-milling starch extraction process for corn in which the endosperm is blended in a microblender after the kernels are steeped in a meta-bisulfate solution. The meta-bisulfate separates bonds between protein and starch granules, simplifying the starch purification process (Meuser et al 1985). In oat grains, presoaking combined with high shear was necessary for adequate separation of protein and starch granules (Lim et al 1992). Physical methods such as blending of the endosperm can further enhance the separation of starch and protein (Lim et al 1992).

Blending entails both grinding of the endosperm with a mortar and pestle and then blending for 4 min in a Waring commercial blender equipped with an Eberbach microcontainer assembly (White et al 1990). The method of White et al (1990) is complete, but it is also time-consuming. A modification of this method might be to improve sample throughput and efficiency of starch separation by using a homogenizer instead of a microblender after steeping. Others have noted, however, that homogenizing corn endosperm during starch extraction could potentially damage starch granules and change starch properties (Meuser et al 1985).

The objectives of this study were to: 1) compare thermal property measurements of starch obtained from endosperm blended with a microblender and from endosperm blended with a homogenizer as a way to increase sample throughput, and 2) examine starch granules for possible damage due to blending endosperm in a homogenizer.

MATERIALS AND METHODS

Maize Populations

Ten hybrids of dent corn with the same maturity dates were obtained from MBS Incorporated and Pioneer Hi-Bred International, Inc. Hybrids were planted in two-row plots at Ames, IA.

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The ears of hybrid plants within a plot were harvested and shelled in bulk. Sixty kernels per plot were randomly selected with a seed counter to conduct six starch extractions, three starch extractions per blending method with 10 kernels per extraction.

Starch Extraction

Two blending methods were used, each with half the samples. The first blending method, a microblender, was used as described by White et al (1990). The microblender consisted of a microcontainer assembly attached to a Waring blender, which had a no-load speed of 20,000 rpm (120 V, 50–60 Hz, and 3 amps). Time requirements were 4 min per blending to blend the endosperm and water as uniformly as possible in the microblender. For each extraction (10 kernels per extraction), endosperm had to be blended twice in the microblender (endosperm from five kernels at a time) and then combined because the blending assembly was not large enough to hold endosperm from all 10 kernels. Total blending time for the microblender was 8 min per extraction. For the second method, a homogenizer was used instead of the microblender to increase the speed of the procedure. For this modification, endosperm from 10 kernels per sample were put in a 100-mL centrifuge tube and 50 mL of distilled water was added. A Tekmar tissue homogenizer (Ultra-Turrax T25, 600W, Cincinnati, OH) at 17,000 rpm blended the endosperm and distilled water for 25 sec in the centrifuge tube. The total mixing time for the homogenizer was 25 sec per extraction. The speed and time chosen for the tissue homogenizer was necessary to uniformly blend the endosperm and water. The screening and drying processes were conducted as described by White et al (1990). Starch isolated using the method of White et al (1990) had a protein content of $\approx 0.88\%$. After extraction, samples from both blending procedures were stored at room temperature in air-tight vials until evaluation by DSC.

Differential Scanning Calorimetry

Starch (≈ 4 mg, dwb) was weighed in an aluminum pan (Perkin-Elmer 0219-0062) and 8 μ L of distilled water was added. The pan was sealed and the sample was allowed to equilibrate for 1 hr at room temperature. A Perkin-Elmer DSC-7 analyzer (Norwalk, CT) equipped with a thermal analysis data station measured and recorded onset temperature (T_o), peak temperature (T_p), and enthalpy of gelatinization (ΔH). The analyzer was thermally programmed to heat samples from 30 to 120°C, at a rate of 10°C/min. During the DSC run, a reference pan containing 8 μ L of distilled water was run at the same time as the sample. After gelatinization, samples were stored at 4°C for seven days and rerun on the DSC to obtain retrogradation values for the same parameters noted for gelatinization. Peak height index (PHI) was calculated from $\Delta H/(T_p - T_o)$. Ranges of gelatinization and retrogradation peaks were recorded as $R_n = 2(T_p - T_o)$, and % retrogradation [$(\Delta H$ retrogradation/ ΔH gelatinization) $\times 100$] was calculated. All DSC parameters for gelatinization and retrogradation were compared between blending methods.

TABLE I
Thermal Properties^a of Starch Gelatinization and Retrogradation Obtained by Two Different Blending Methods of Endosperm During Starch Extraction^b

	T_o	SD	T_p	SD	ΔH	SD	PHI	SD	R_n	SD	%R	SD
Gelatinization												
Microblender	69.38	1.36	73.62	0.88	3.29	0.13	0.81	0.67	8.47	1.66	42.18	12.68
Tissue homogenizer	68.86	1.66	73.00	1.13	3.33	0.19	0.84	0.66	8.29	1.60	40.63	10.82
LSD ^c	0.43	...	0.32
Retrogradation												
Microblender	43.00	3.17	53.30	2.36	1.33	0.41	0.13	0.038	20.60	3.22
Tissue homogenizer	43.80	4.74	53.44	2.21	1.41	0.38	0.14	0.037	19.33	6.10
LSD ^c	0.0135

^a T_o , onset temperature (°C); T_p , peak temperature (°C); ΔH , enthalpy of gelatinization (cal/g); PHI, peak height index; R_n , retrogradation peak (°C); %R, % retrogradation; SD, standard deviation.

^b Means of 10 hybrids (30 extractions) per blending method.

^c Least significant difference values are given only for parameters that differed between blending methods.

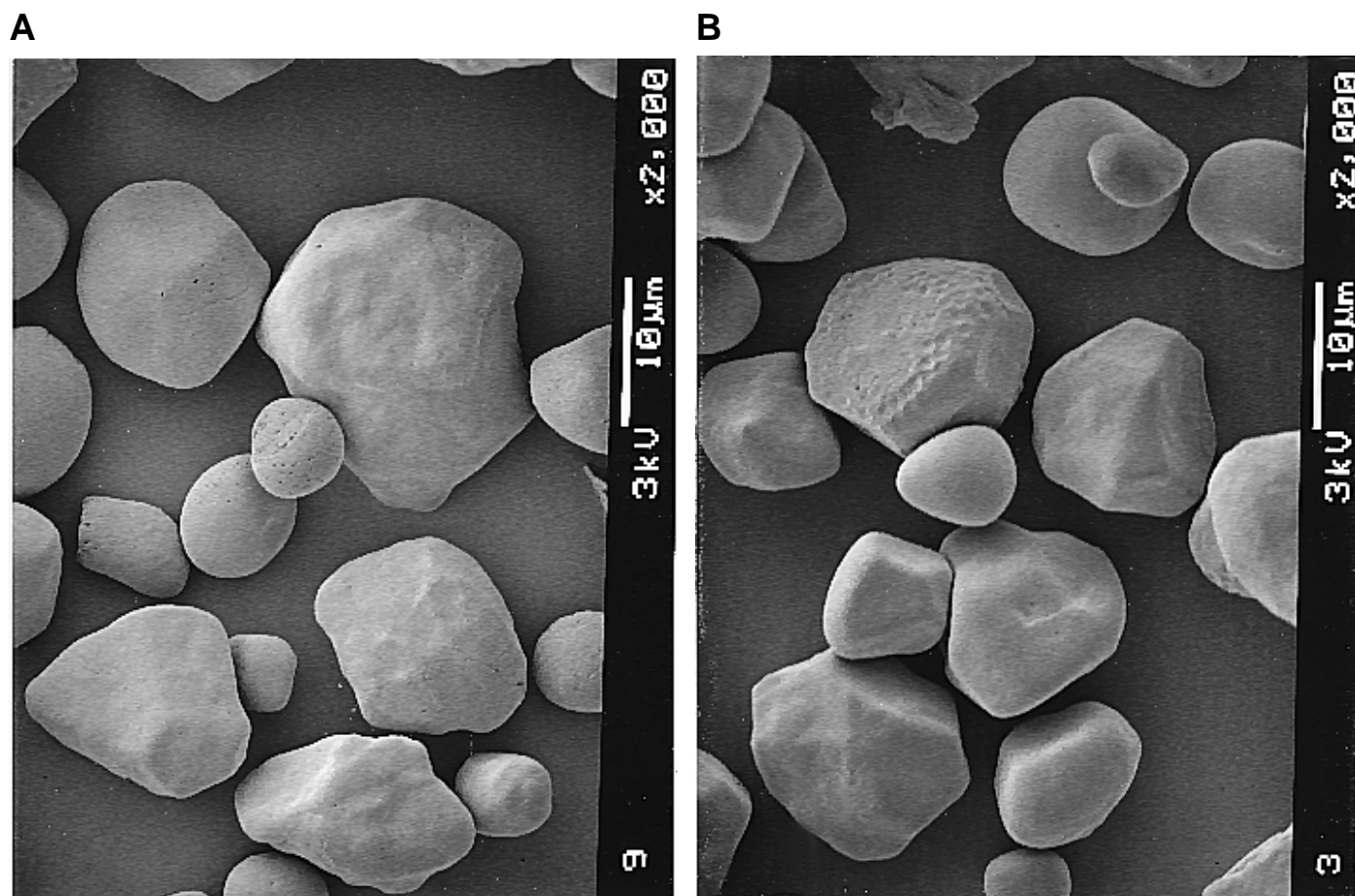


Fig. 1. Electron micrographs of hybrid 13008 × MBS501 representing starch extracted by the microblender method (A) and tissue homogenizer method (B). Magnification ×2,000, bar = 10 µm.

Electron Scanning Microscope

Extracted starch samples were dusted onto silvertape and mounted to aluminum cylinders with silverpaint. Samples were sputter-coated with gold pladium. Electron micrographs were generated from a JOEL JSM 5800LV scanning microscope at high vacuum, 10 kV accelerating voltage, and ×2,000 magnification (bar = 10 µm). A 1-cm² area on a slide was observed for starch granule damage from each sample. About 15 microscopic fields per slide were observed for starch granule damage (≈300 starch granules per slide).

Hydration Capacity

Two starch samples (three separate starch extractions per sample) for each blending method were analyzed for hydration capac-

ity as an indication of possible starch damage (AACC 1995). To obtain enough starch for the analyses, starches from two hybrids were combined (13008 × MBS501 and Tester A × MBS280, and Pioneer Hybrid 3394 and 13008 × MBS9541). One modification of the method was adopted to account for the small sample size. For each sample, 0.2 g of starch was weighed into a 50-mL centrifuge tube and 4 mL of water was added before centrifuging. Samples were centrifuged for 15 min at 1,000 × g.

Statistical Analysis

The proc general linear models (GLM) procedure from Statistical Analysis Systems (SAS Institute, Cary, NC) was used to determine significant differences ($P \leq 0.05$) between blending methods and to determine interaction between blending method and hybrid.

TABLE II
Hydration Capacity of Starch Obtained by Blending with a
Microblender and Tissue Homogenizer During Starch Extraction

Blending Method	Hydration Capacity
Microblender	1.63 ^a
Tissue homogenizer	1.69

^a Average of two starch samples (three extractions per sample) for each blending method. There were no significant differences between blending methods for hydration capacity.

RESULTS AND DISCUSSION

Table I summarizes means and standard deviations of DSC parameters for starch from 10 hybrids (three replicates per hybrid = 30 extractions) for each blending method. There were no interactions noted between blending method and hybrid (data not shown). Least significant difference (LSD) values are given where there were differences ($P \leq 0.05$) between blending methods.

Starch extracted with a tissue homogenizer had slightly, but significantly, lower T_o and T_p of gelatinization than starch extracted with the microblender. Perhaps the tissue homogenizer separated starch granules better than the microblender, thus allowing gelatinization to occur at a slightly lower temperature. Values for ΔH , PHI, gelatinization R_n , and %R were not different between blending methods.

DSC parameters for means of retrogradation runs and standard deviations are also shown in Table I. The data generated from the DSC shows that the PHI for the tissue homogenizer method was slightly (0.14 vs. 0.13), but significantly, greater than the microblender method ($P \leq 0.05$). The values of T_o , T_p , ΔH , and retrogradation R_n for the microblender and tissue homogenizer methods were not different from each other.

Analyses of both gelatinization and retrogradation runs suggest that the microblender and tissue homogenizer are reliable blending methods. Although T_o and T_p from gelatinization runs were significantly different, and PHI from retrogradation runs were significantly different, the actual differences were very small and would not be important in determining differences in functional properties of starches. Both methods produced isolated starch with consistent thermal property data when measured by DSC, as noted by the low standard deviations reported in Table I for gelatinization, except for R_n . Retrogradation is influenced by amylopectin chain length, starch concentration, and the stability of starch paste during storage (Yuan et al 1993, Shi and Seib 1992). Therefore, values for retrogradation tend to have greater standard deviations because of the unpredictability of the structure of retrograded starch after initial gelatinization. Both methods, however, resulted in DSC parameters with similar standard deviations.

The hydration capacities of starch from the tissue homogenizer (1.69) and microblender (1.63) were not significantly different ($P \leq 0.05$) (Table II). These results give additional evidence that

starch blended with a tissue homogenizer was not damaged and was comparable to starch blended with a microblender.

Figure 1 provides visual evidence of intact and individual starch granules for both microblender and tissue homogenizer extraction methods of hybrid 13008 × MBS501. Micrographs representing all extractions of two randomly selected hybrids (13008 × MBS501 and Pioneer hybrid 3394) showed similar and discreet starch granules. All micrographs suggest starch isolation with either the microblender or the tissue homogenizer does not damage starch granules. The tissue homogenizer, however, grinds endosperm seven and a half times faster per extraction than the microblender because of its capability to blend large amounts of endosperm with good efficiency. The entire original starch extraction process (White et al 1990) takes three days to complete with blending and filtering as the most tedious and labor intensive step. To decrease the time of sample throughput, the preferred method for grinding starch endosperm would be to replace the microblender with a tissue homogenizer when conducting starch extractions as described by White et al (1990). Each homogenization of five kernels would reduce extraction time from 4 min to 25 sec. If 10 kernels were being used, the extraction time would be 8 min, with the homogenizer still taking only 25 sec. Within a day, a laboratory worker could increase the number of 10-kernel extractions from 24 to 240. An additional benefit is the noise reduction in the laboratory when using a homogenizer.

Thermal property measurements from starch blended with either a tissue homogenizer or a microblender are so similar that either blending method could be used for starch evaluation. The tissue homogenizer is an efficient and dependable blending method for starch extraction because it blends endosperm faster than the microblender and it produces undamaged, intact starch granules.

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