Effects of Degree of Milling and Lipid Removal on Starch Gelatinization in the Brown Rice Kernel

E. T. CHAMPAGNE, W. E. MARSHALL, and W. R. GOYNES[1]

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ABSTRACT

The effects of the degree of milling (unmilled to 9.1% removal of outer surface) and of surface, nonstarch lipid removal on starch gelatinization in the rice kernel were examined by differential scanning calorimetry. Onset (T_o) and peak (T_p) gelatinization temperatures of full-fat and defatted kernels were determined to be logarithmic functions of the degree of milling. The removal of the outer 1.3% of the full-fat or defatted rice kernels by milling led to the largest decreases in T_o and T_p values. The greater the degree of milling, the larger the percentage of lipid solvent extracted from the kernels and the larger the decrease in T_p. Decreases in T_p followed logarithmic and linear functions of the percentage of lipid removed from unmilled and milled rice kernels, respectively. A relatively large decrease in T_p was observed for a small percentage of lipid extracted from unmilled rice kernels compared with the decreases in T_p observed for much larger percentages of lipid extracted from 1.3–9.1% milled rice kernels. Evidence is presented indicating that waxes and nonstarch lipids in the rice kernel have a role affecting starch gelatinization.

Brown rice is rich in fiber, minerals, and vitamins that are removed during milling to make conventional white rice. Brown rice is noted for requiring twice the cooking time of white rice. The long cooking time of brown rice has been attributed to its reduced rate of water absorption compared with white rice. Desikachar et al (1965) demonstrated that removing the outer 1% of the brown rice kernel increases the water absorption during cooking to that of highly milled rice. The wax content of this outer 1% layer was implicated in reducing the rate of water absorption. The differences in the cooking times of brown and milled rice may also be related to differences in their starch gelatinization temperatures. Rice starch samples with higher gelatinization temperatures have been shown to require longer cooking times than those with lower gelatinization temperatures (Juliano and Perez 1983). Binding of lipids to rice amylose and amylopectin may result in higher gelatinization temperatures (Maningat and Juliano 1980, Ohashi et al 1980).

The purpose of this investigation was to study the effects of the degree of milling, from unmilled (brown rice) to 9% milled (white rice), and lipid removal on the thermal parameters of starch gelatinization in the rice kernel. Recently Normand and Marshall (1989) reported the first differential scanning calorimetric study on whole grain milled rice; their method was employed in this investigation.

MATERIALS AND METHODS

Rough rice samples of Lemont (1987) were obtained from the Louisiana State University Rice Experiment Station, Crowley, LA. The samples were dehulled in a McGill sheller (H. T. McGill, Houston, TX) and milled in a Satake mill, model TM-05 (Satake Engineering Co., Japan). To prepare rice flours, brown or milled rice was ground to a fine powder in a Udy cyclone mill (Udy Corp. Fort Collins, CO) equipped with a 40-mesh sieve screen.

Methods

The degree of milling was determined by weighing the milled rice from which bran particles were removed with a 14-mesh sieve. Surface, nonstarch lipids were extracted from the unmilled or milled rice kernels by the method of Hogan and Deobald (1961), except that chloroform-methanol (2:1, v/v) was used as the lipid extraction solvent instead of petroleum ether. Extraction times were 25 min and 1 hr. The amount of lipid extracted from the rice kernel is expressed as the percentage of nonstarch lipid removed from the same quantity of rice flour by an 8-hr chloroform-methanol extraction. The extraction procedure outlined in AACC method 30-25 (1983) was followed for removing the nonstarch lipids from the rice flours. The defatted rice kernels were desolventized by air-drying overnight. Moisture contents of the full-fat and defatted rice kernels were determined by oven drying ground material at 130°C for 1 hr according to the Agricultural Marketing Service method (AMS 1959).

Differential scanning calorimetry was conducted using a thermal analyzer (model 7701, Hart Scientific, Provo, UT) equipped with a Hart Scientific 707 cell base designed for a system that includes a reference and one to three samples. Samples were prepared and run on the differential scanning calorimeter according to the procedure of Normand and Marshall (1989). Sufficient water was added to the rice kernels and flour to obtain approximately 70% moisture. There were no significant differences in the thermal curves obtained for unmilled rice kernels presoaked for 1, 6, or 18 hr at room temperature (data not shown). To expedite data acquisition, all samples were presoaked for approximately 1 hr at room temperature, and the ampoules were equilibrated to 20°C. Thermal curves were obtained from 200 to 110°C with a heating rate of 1.0°C/min. Thermal transitions of the brown rice kernels or brown rice flours were defined as T_o (onset), T_p (peak gelatinization), and T_c (conclusion). The enthalpy associated with starch gelatinization, ΔH, was calculated as the area of the endotherm enclosed by drawing a straight line between T_o and T_c and is expressed in joules per gram of sample, dry basis.

A Hitachi S-510 scanning electron microscope was used to examine the surfaces of the full-fat and defatted rice kernels. The rice grains were attached to aluminum sample stubs with double adhesive tabs. No fixation and dehydration processes were necessary. Prepared stubs were sputter-coated with gold/palladium to prevent charging in the electron beam. The stubs were observed at 5 and 10 kV accelerating voltage.

Statistical Analyses

Significant differences in thermal parameters between full-fat and defatted rice kernels and flours were determined by paired t tests using the SAS statistical package (SAS Institute, Inc., Cary, NC). The SAS statistical package was also utilized for stepwise regression analysis of the degree of milling and lipid removal data.

RESULTS

Effects of Degree of Milling on the Thermal Parameters of Brown Rice Kernels

Each thermal curve for full-fat or defatted rice kernels was characterized by a broad starch gelatinization endotherm (Normand and Marshall 1989), as illustrated in Figure 1A and

[1]Southern Regional Research Center, Agricultural Research Service, U.S. Department of Agriculture, Box 19687, New Orleans, LA 70179.

B for full-fat unmilled and full-fat 9% milled rice, respectively. Increasing the degree of milling of the rice kernels resulted in the thermal curve shifting to lower temperatures (Fig. 1A and B). The calculated T_o and T_p values of the starch gelatinization endotherm of full-fat and defatted rice kernels milled to different degrees are listed in Table I. In samples in which the outer 1.9 to 9.1% of the rice kernel was removed by milling, a low-temperature shoulder appeared on the starch gelatinization endotherm. T_o values were calculated to commence with the initiation of the low-temperature shoulder in these samples. T_c and ΔH values could not be calculated for the brown rice kernels since no final baseline was observed, due to temperature limitations of the instrument.

The removal of the outer 1.3% of the full-fat or defatted rice kernels by milling led to the largest decreases in T_o and T_p values. Using stepwise regression, T_o and T_p values of the full-fat and defatted kernels were determined to be logarithmic functions of the degree of milling. The general equation that described T_o or T_p as a function of the degree of milling was:

$$Y = m \times \log(\text{degree of milling} + 1) + b_0$$

where $Y = T_o$ or T_p, m = slope of the regression line, and b_0 = y-intercept. The regression and correlation values for full-fat and defatted rice kernels are listed in Table II.

Thermal curves of flour samples obtained from grinding comparable full-fat rice kernels milled to different degrees are shown in Figure 1 (D and E). Similar thermal curves were obtained for flours obtained from defatted milled rice kernels. The starch gelatinization endotherm in each flour thermal curve was narrower than that observed in the corresponding whole kernel thermal curve. The T_o value for the full-fat, unmilled rice kernels was approximately 2°C higher than that for the flour made from these kernels. In contrast, the T_o values for the full-fat milled kernels were not significantly different ($P = 0.7953$) from those for the flours made from these kernels (Tables I and III).

Peak gelatinization temperatures in the rice flour samples were markedly lower (approximately 12–22°C) than those of the corresponding whole kernels (Tables I and III). Heat transfer effects account only in part for the lower T_p values of the rice flours. Normand and Marshall (1989) demonstrated that when heat transfer effects are eliminated at zero heating rate, T_p values for 10% milled rice kernels were still 9°C higher than those for flour samples. The T_p values of the rice flours correspond roughly to the positions of the low-temperature shoulders that were present in the 1.9–9.1% milled whole kernel rice samples. No low-temperature shoulder was observed in the flour scans. However, a small higher temperature endotherm in the 98–100°C range was present in the flour thermal curves. Biliaderis et al (1986) have proposed that endotherms in this region of the thermal profile are due to the melting of an amylose-lipid complex formed during heating in the calorimeter or already present in the flour.

Effects of Lipid Removal on the Thermal Parameters of Rice Kernels and Flours

Table IV compares the lipid contents of full-fat and defatted rice kernels and lists the percentages of lipid extracted from the kernels by chloroform-methanol (2:1, v/v). The 25-min and 1-hr solvent extractions were effective in only extracting 13 and 16%, respectively, of the total lipid in the unmilled rice kernels. In contrast, 50–64% and 62–85% of the kernel lipid was extracted from the 1.3–9.1% milled rice kernels for 25-min and 1-hr extraction times, respectively.

Extracting surface nonstarch lipids from the milled rice kernels increased the magnitudes of the low-temperature shoulders (Fig. 1C). Scanning electron micrographs (Fig. 2) illustrate the cracking of the rice surface upon lipid removal. Extraction by chloroform-methanol partially dehydrates the kernel surface. When the extracted kernels are exposed to humid air, they rehydrate and expand, which results in cracking. The extent of cracking and the width and depth of the cracks appeared to increase as functions of lipid extraction time (percentage of lipid extracted) and the degree of milling. Figure 2A shows the undamaged surface of a full-fat, unmilled kernel. Figure 2B illustrates the surface change in a full-fat, 9.1% milled kernel. With 25-min lipid extraction

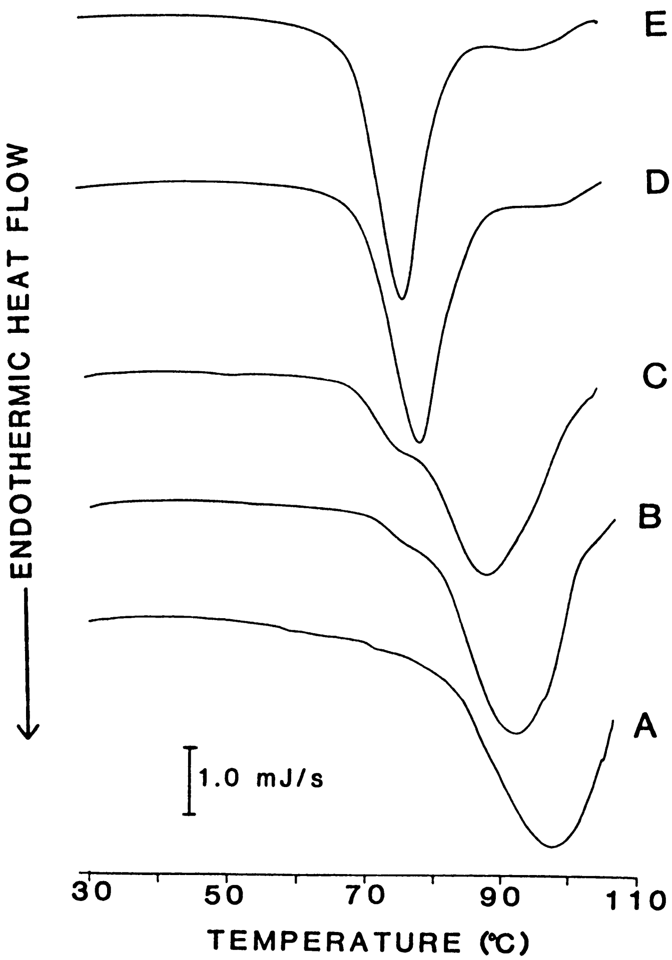

Fig. 1. Thermal curves of full-fat unmilled (**A**), full-fat 9.1% milled (**B**), and 1-hr defatted 9.1% milled kernels (**C**). Thermal curves **D** and **E** are for flour samples obtained from grinding full-fat unmilled and 9.1% milled rice kernels, respectively.

TABLE I
Effects of Degree of Milling and Defatting on Onset (T_o) and Peak (T_p) Gelatinization Temperatures (°C) of Rice Kernels[a]

% Milled	Full Fat T_o	Full Fat T_p	Defatted 25 min T_o	Defatted 25 min T_p	Defatted 1 hr T_o	Defatted 1 hr T_p
Unmilled	76.4 ± 0.1	101.5 ± 0.1	75.6 ± 0.3	100.7 ± 0.1	74.2 ± 0.4	100.5 ± 0.1
1.3	74.8 ± 0.2	96.7 ± 0.2	74.4 ± 0.3	95.7 ± 0.1	73.3 ± 0.0	94.5 ± 0.2
1.9	74.1 ± 0.3	96.4 ± 0.1	73.8 ± 0.3	94.6 ± 0.3	72.8 ± 0.1	94.4 ± 0.0
3.1	73.6 ± 0.1	95.1 ± 0.1	72.5 ± 0.2	93.0 ± 0.0	72.3 ± 0.3	92.7 ± 0.2
5.0	73.6 ± 0.3	94.2 ± 0.0	72.6 ± 0.1	92.0 ± 0.2	72.3 ± 0.3	90.4 ± 0.3
7.2	72.8 ± 0.3	94.0 ± 0.2	72.7 ± 0.2	91.9 ± 0.2	72.2 ± 0.1	89.4 ± 0.0
9.1	72.5 ± 0.2	94.1 ± 0.1	72.3 ± 0.1	91.5 ± 0.2	71.4 ± 0.3	89.3 ± 0.3

[a] Moisture content of samples was 70%. Values given are means ± standard error of the mean of duplicate determinations.

times, the unmilled and 1.3–3.1% milled rice kernels exhibited no cracking, while the 5.0–9.1% milled kernels appeared etched with very shallow longitudinal and transverse cracks when observed at 20× magnification. This etching is shown in Figure 2C for a kernel after 9.1% milling and 25 min lipid extraction. Very minute cracks were revealed on the surface of the 25-min defatted 1.3–3.1% milled rice kernels at 100× magnification. With 1-hr lipid extraction times, the unmilled rice kernels showed no cracking, the 1.3–3.1% milled kernels had minute cracks, and the 5.0% milled kernels were moderately cracked. The 7.2 and 9.1% milled kernels exhibited extensive radial and transverse fractures when observed at 20× magnification (Fig. 2D).

Nonstarch lipid extraction from unmilled and milled rice kernels significantly ($P < 0.0001$) lowered T_o and T_p values of the kernels and the flours obtained from the kernels. Increasing lipid extraction time of the kernels from 25 min to 1 hr further decreased T_o ($P < 0.002$) and T_p ($P < 0.0001$) values. The T_o values of the unmilled rice kernels and flours decreased by approximately 2 and 0.8°C, respectively, upon 1 hr of lipid extraction (Tables I and III). The T_o values of the 1-hr defatted, milled (1.3–9.1%) rice kernels and flours were approximately 10 and 0.60, respectively, lower than those of the full-fat rice samples with the same degree of milling. The differences in T_p values between the full-fat rice kernels and the defatted rice kernels ranged from approximately 0.8 to 5°C, with the temperature difference increasing with the length of lipid extraction time and the degree of milling (Table I). The T_p values of the flours from defatted rice kernels were approximately 0.4–0.9°C lower than those for the flours from full-fat kernels with the temperature difference decreasing with the degree of milling (Table III). There were no significant differences ($P = 0.4007$) between the T_c values of the full-fat flours and those of the defatted flours. The gelatinization enthalpy (ΔH) values for rice flours obtained from unmilled and 1.3–9.1% milled rice samples are listed in Table V. The ΔH values of the flours from full-fat kernels were significantly ($P < 0.001$) smaller than those of the flours from defatted kernels.

Figure 3 depicts the relationship between the decrease in T_p values and the percentages of nonstarch lipids removed from the unmilled and 1.3–9.1% milled rice kernels. The greater the degree of milling, the larger the percentage of lipid extracted and the larger the decrease in peak gelatinization temperature. The data points for the defatted unmilled rice kernels, however, did not fall on the same curve as the data points for the defatted milled rice kernels. Note that extraction of 16% of the lipid of the unmilled rice kernel resulted in a 1.0°C decrease in T_p, whereas extraction of 50% of the lipid from the 1.3% milled rice also decreased T_p by 1.0°C. Using stepwise regression, the following equations were determined to describe the decrease or change (Δ) in T_p as a function of the percentage of lipid removed from unmilled and milled rice kernels:

$$\Delta T_p \text{ unmilled} = 2.71 \times \log(\% \text{ lipid removed} + 1) - 2.29$$

$$\Delta T_p \text{ milled} = 0.11 \times (\% \text{ lipid removed}) - 4.60$$

TABLE II
Regression and Correlation Values for Onset (T_o) and Peak (T_p) Gelatinization Temperatures of Full-Fat and Defatted Unmilled and Milled (1.3–9.1%) Rice Kernels
$Y = m \cdot \log(\text{degree of milling} + 1) + b_o$[a]

Sample	Y	b_o	m	R^2
Full fat	T_o	−3.71	76.16	0.93
Defatted 25 min	T_o	−3.33	75.38	0.85
Defatted 1 hr	T_o	−2.50	74.11	0.86
Full fat	T_p	−7.03	100.09	0.86
Defatted 25 min	T_p	−9.21	99.58	0.92
Defatted 1 hr	T_p	−10.96	99.44	0.94

[a] $Y = T_o$ or T_p, b_o = the regression estimate of the y-intercept, m = the estimate of the slope of the regression line, R^2 = the coefficient of determination.

DISCUSSION

The results of this investigation suggest that two factors appear to lower T_o and T_p of starch gelatinization of rice kernels: increasing the degree of milling and lipid extraction. Removal of the pericarp and seed coat, which was accomplished by 1.3% milling, led to the largest decrease in T_p values (approximately 5°C) and T_o values (approximately 1°C) in both the full-fat and partially defatted rice samples compared with subsequent decreases in these thermal parameters with further milling. The T_o value of the full-fat unmilled rice was dependent on rice kernel integrity, as indicated by the 2°C difference in T_o between the

TABLE III
Effects of Degree of Milling and Defatting on Onset (T_o), Peak (T_p), and Conclusion (T_c) Gelatinization Temperatures (°C) of Rice Flour[a]

	Full Fat			Defatted 1 hr		
% Milled	T_o	T_p	T_c	T_o	T_p	T_c
Unmilled	74.3 ± 0.1	78.9 ± 0.1	88.1 ± 0.9	73.5 ± 0.0	78.0 ± 0.1	86.6 ± 0.3
1.3	73.8 ± 0.0	78.5 ± 0.0	86.3 ± 0.0	73.4 ± 0.0	78.1 ± 0.2	86.5 ± 0.0
1.9	73.7 ± 0.1	78.4 ± 0.1	86.7 ± 0.2	73.3 ± 0.1	78.0 ± 0.1	86.9 ± 0.0
3.1	73.6 ± 0.2	78.4 ± 0.2	87.0 ± 0.1	73.4 ± 0.1	77.8 ± 0.1	87.0 ± 0.1
5.0	73.5 ± 0.0	78.3 ± 0.0	86.7 ± 0.2	72.8 ± 0.1	77.6 ± 0.1	86.6 ± 0.1
7.2	73.3 ± 0.0	78.0 ± 0.0	86.7 ± 0.4	72.4 ± 0.0	77.6 ± 0.1	86.5 ± 0.0
9.1	73.2 ± 0.1	77.7 ± 0.0	86.6 ± 0.1	72.5 ± 0.0	77.3 ± 0.0	86.7 ± 0.7

[a] Rice flours obtained by grinding full-fat and defatted unmilled and milled (1.3–9.1%) rice kernels. Moisture content of samples was 70%. Values given are means ± standard error of the mean of duplicate determinations.

TABLE IV
Lipid Contents of Lemont Rice Kernels[a]

	Full Fat	Defatted 25 min		Defatted 1 hr	
% Milled	% Lipid	% Lipid	% Lipid Extracted	% Lipid	% Lipid Extracted
Unmilled	3.71	3.22	13.3	3.12	15.8
1.3	3.20	1.59	50.4	1.18	63.2
1.9	3.12	1.25	60.0	1.17	62.4
3.1	2.79	1.08	61.3	1.02	63.5
5.0	2.53	0.96	62.1	0.55	78.4
7.2	2.09	0.76	63.5	0.34	83.9
9.1	1.68	0.60	64.3	0.25	85.0

[a] Chloroform-methanol (2:1) extractable lipid. Values given are means of duplicate determinations. Standard error of the mean < 0.05% for % lipid values. Values given are on dry weight basis.

kernel and flour samples. In contrast, the initiation of starch gelatinization in the 1.3–9.1% milled rice kernels was independent of the physical form of these samples. The pericarp and seed coat appear to provide a barrier that controls water penetration to the starch granules. Evidence of the pericarp and seed coat restricting solvent penetration was the small percentages of nonstarch lipids extracted from the unmilled rice by the chloroform-methanol solvent compared with the percentages extracted from the 1.3–9.1% milled rice kernels. Although defatting the unmilled rice did not appear to crack the kernels, the pericarp and seed coat tissues were apparently disturbed by the extraction since the difference in T_o values between the kernel and flour samples was smaller (0.7°C) following 1-hr defatting.

Waxes are located in the pericarp and seed coat of the rice kernel (Bechtel and Pomeranz 1977) and are part of the lipid fraction extracted by the chloroform-methanol solvent (Ito et al 1983). We suggest that it was the extraction of wax from these tissue layers in the unmilled rice that led to the relatively large decrease in T_p for the small percentage of lipid extracted compared with the decreases in T_p observed for much larger percentages of lipid extracted from the 1.3–9.1% milled rice samples, which were devoid of wax.

The data presented in Tables I and III and in Figure 3 suggest that lipid extraction lowers T_o and T_p values. However, the question is raised as to whether removing lipid lowers T_o and T_p values or if the lipid extraction process leads to cracking of the rice kernels, which is then responsible for lowering these parameters. The following observations in this study indicated that the lipid itself played a role affecting starch gelatinization in brown rice: 1) Milling from 1.3 to 9.1% resulted in approximately a 52% decrease in the lipid/starch weight ratio of the rice without any evidence of kernel cracking. The difference in T_o and in T_p between 1.3 and 9.1% milled rice kernels was 2.3 and 2.6°C, respectively. 2) The thermal parameters for the flours made from defatted rice kernels were significantly ($P < 0.0001$) lower than those for the flours obtained from full-fat rice kernels, suggesting a dependence on lipid and not structure. We propose that solvent extraction of lipid from the rice kernel allows easier water access to the starch granules and thus lowers starch gelatinization temperatures.

The increase in magnitude of the low-temperature shoulder in the thermal curves of rice kernels with increased degree of

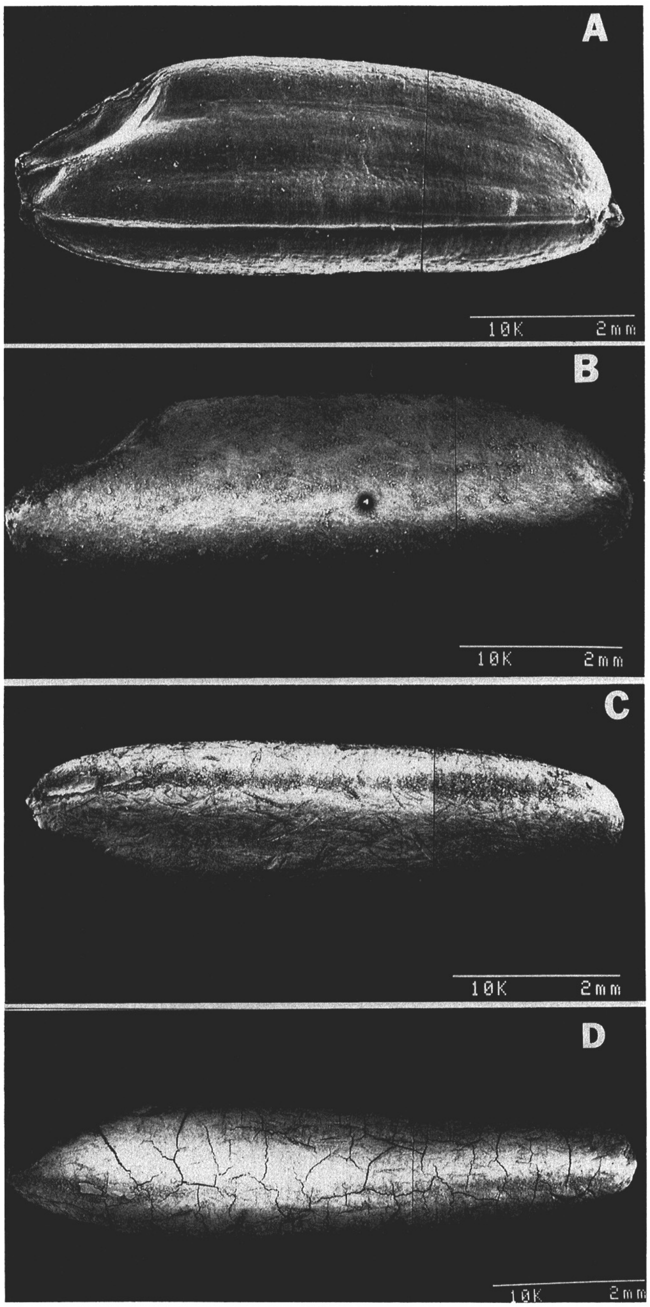

Fig. 2. Scanning electron micrographs of full-fat unmilled (**A**), full-fat 9.1% milled (**B**), 25-min defatted 9.1% milled (**C**), and 1-hr defatted 9.1% milled (**D**) rice kernels.

TABLE V
Enthalpy Values[a] for Flours Obtained by Grinding Full-Fat and 1-hr-Defatted Unmilled and Milled (1.3–9.1%) Rice Kernels

	Enthalpy ΔH (J/g)	
% Milled	**Full-Fat Flour**	**Defatted Flour**
Unmilled	9.1 ± 0.2	9.7 ± 0.3
1.3	9.5 ± 0.1	9.5 ± 0.1
1.9	9.1 ± 0.2	9.9 ± 0.2
3.1	9.2 ± 0.2	10.2 ± 0.2
5.0	10.0 ± 0.2	10.1 ± 0.0
7.2	9.7 ± 0.5	10.2 ± 0.2
9.1	10.2 ± 0.0	10.3 ± 0.1

[a]Values given are means ± standard error of the mean for duplicate determinations.

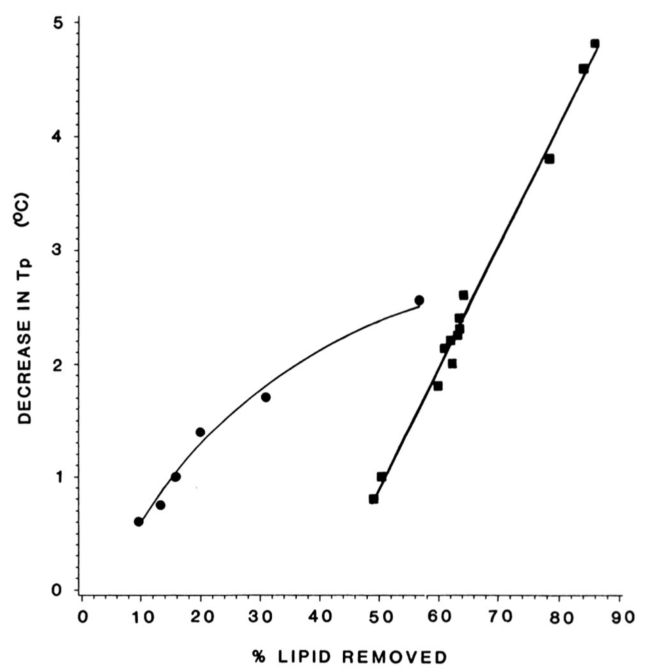

Fig. 3. The relationship between the decrease in T_p values and the percentages of nonstarch lipids removed from unmilled (●) and 1.3–9.1% milled (■) kernels. Data points are included for extraction times other than 25 min and 1 hr. Additional points on curve for unmilled kernels are for 15-min, 2-hr, 4-hr, and 8-hr lipid extraction times. Lowest point on curve for milled kernels is for 1.3% milled rice kernels that were solvent extracted for 15 min.

milling and increased solvent extraction time also suggests that lipid removal by these means provides easier water access to starch granules. Normand and Marshall (1989) have proposed that starch granules are organized into two types of compartments in the rice kernel. One compartment would be readily accessible to water, whereas a second compartment would be less accessible. Normand and Marshall suggest that the former population is associated with the low-temperature shoulder observed in thermal curves of intact kernels or the major endotherm in thermal curves of rice flours. Producing a flour from whole rice kernels would destroy the natural barriers separating these compartments leading to a larger population of starch granules more easily accessible to water.

Cracking of the kernel could also increase the population of starch granules highly accessible to water (Marshall et al 1989). Unfortunately, there is no way of definitively determining if kernel cracking played a role in decreasing the starch gelatinization temperatures of the 1.3–9.1% milled rice kernels and if it did, how much. The flour data in Table III eliminate the effect of kernel structure on the thermal parameters. The difference in the T_p values of the full-fat flours and the defatted flours was less than 1°C. A 1°C decrease in T_p was also observed for the 1.3% milled rice kernels after 25-min solvent extraction, which removed approximately 50% of the lipid and yielded kernels that had only a few very minute cracks (100×). These observations indicate that the effect of lipid content on T_p was probably small (approximately 1°C), and that perhaps kernel cracking led to the additional 1–4°C decreases in T_p values observed upon defatting the 1.9–9.1% milled rice kernels.

The results of this study may be helpful in designing treatments that alter the surface of the brown rice kernel through partial milling or lipid extraction to produce quicker-cooking brown rice. Scanning electron microscopic studies are underway to examine further the effects of solvent extraction of lipids on the external and internal structure of the rice kernel.

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