Thermophysical Properties of Milled Rice Starch as Influenced by Variety and Parboiling Method

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ABSTRACT

Rices differing in amylose content and starch final gelatinization temperatures (GT) were parboiled at 100, 120, and 127°C to determine the formation of amylose-lipid complex I (melting at <100°C) and complex II (melting at >100°C), which are observed in model systems. In rices parboiled at 100°C, only the low-GT (<70°C) nonwaxy rices exhibited the amylose-lipid complex I melting, without residual (unmelted) starch crystallites. Intermediate-high-GT starches (≥70°C), with residual annealed starch, did not form complex I, nor did waxy IR65. The amylose-lipid complex II melting endotherm was observed in all rices parboiled at 120 and 127°C. It was highest for IR64 parboiled at 120°C. Reduction in hot-water-soluble amylose and increase in X-ray diffraction peak at $2\theta = 19.9°$ for V complex corresponded to maximum ΔH of amylose-lipid complex II melting for the pressure-parboiled samples of intermediate-high-amylose rices.

In model systems of solution-crystallized complexes of amylose with monoacyl lipids (fatty acids, monoglycerides, etc.), Biliaderis and Galloway (1989) demonstrated the formation of two distinct polymorphic structures, depending on the crystallization temperature: complex I with melting temperature (T_m) below 100°C and complex II with T_m above 100°C. These structural forms differ mainly in the degree of chain organization in the solid state, as evidenced by X-ray diffraction and differential scanning calorimetry (DSC) measurements (Biliaderis and Galloway 1989, Biliaderis and Seneviratne 1990). Complex I, expected with rapid nucleation, predominates at low crystallization temperatures (50–60°C). It is morphologically described by a random distribution of helical chains having little crystallographic register in the aggregated state. In contrast, complex II appears to have the structure of discrete crystallites and is the preferred polymorph at high crystallization temperatures (>90°C). The nature of the complexing ligand also seems to affect the supermolecular structure of the complex (Biliaderis et al 1986a); fatty acids and monoglycerides produce both structural forms (I and II) with amylose, but lysophosphatidylcholine gives only one type of complex (T_m = 104°C, heated in excess water).

Parboiled rice is a natural model for studying the two types of amylose-lipid complexes and their influence on the physical properties of the product. The starch lipids of nonwaxy rice grains (mainly lysophosphatidylcholine, lysophosphatidylethanolamine, and free fatty acids [Choudhury and Juliano 1980]) form complexes with the linear starch component (amylose) during the parboiling process (Mahanta et al 1989). Rough rice for parboiling is steeped in water and boiled or steamed to gelatinize the starch with minimum leaching of the endosperm constituents, then it is cooled and dried. In modern processing plants, parboiling uses steam under pressure. The hydrothermal conditions affect the complexing of amylose with lipids, which, in turn, might be responsible for the restricted swelling and solubilization of starch upon cooking the parboiled grain (Priestley 1976, Mahanta et al 1989).

In this study, we examined the two forms of amylose-lipid complexes for parboiled rice in seven rices with differing starch properties using DSC and X-ray diffraction. An attempt was made to relate the formation of these polymorphs to the physical properties of the parboiled grain.

MATERIALS AND METHODS

Parboiling treatments were carried out in an autoclave at 100°C (gauge pressure 0 kg/cm^2 for 30 min), 120°C (gauge pressure 1 kg/cm^2 for 10 min), and 127°C (gauge pressure 1.5 kg/cm^2 for 10 min) as described by Biswas and Juliano (1988). The parboiled IR65, IR24, IR48, IR64, IR5, and IR8 samples were characterized in an earlier study (Biswas and Juliano 1988). IR2071-137-5 was also parboiled at 100°C for 30 min and at 120 and 127°C for 10 min. Rough rice was dehulled with a Satake THU-35A sheller. The brown rice was milled in a Satake TM-05 pearler with a 5330 abrasive disk at 1,730 rpm to approximately 8% bran-polish removal.

Alkali-spreading value was determined by the method of Little et al (1958) using duplicate samples of six grains soaked for 23 hr in 10 ml of 1.7% KOH at 30°C. Milled rice flour was prepared in a Udy Cyclone mill with a 40–60 mesh sieve. Apparent amylose content (AC) was determined by iodine colorimetry at 620 nm using potato amylose and waxy rice starch standard mixture (Juliano et al 1981). Gel consistency was determined by the procedure of Cagampang et al (1973) for 100 mg of flour in 2 ml of 0.2N KOH in 13- × 100-mm culture tubes.

Cooked rice samples were prepared in duplicate by soaking 10 g of milled rice for 30 min in 150-ml beakers in a predetermined amount of water based on amylose content (13 ml for waxy rice, 17 ml for low-AC rices, 19 ml for intermediate-AC rices, and 21 ml for high-AC rices) as previously established by Juliano and Pascual (1980). The four samples were cooked simultaneously for 20 min in Toshiba RC-4B automatic cookers with 200 ml of water in the outer pot (Biswas and Juliano 1988). After standing 10 min, the cooked rice was drained and transferred into polyethylene bags to cool for 1 hr. The cooled rice (17 g) was placed into a 10-cm^2 Ottawa texture measuring system cell with a perforated base (5.2-mm holes); pressed with a 145-g weight for 1 min; and extruded in an Instron model 1140 food tester with a 5–50-kg load cell at crosshead and chart speeds of 30 cm/min (Perez and Juliano 1979). Hardness was the maximum force needed to extrude rice through the perforated base, reexpressed as kg/7 cm^2.

Nonstarch and starch lipids were extracted from rice flours. Nonstarch lipids were extracted three times, for 8 hr each, at ambient temperature with five volumes of chloroform and methanol (1:1), then extracted twice with water-saturated 1-butanol for 30 min, including 10 min of centrifugation at 2,000 × g (Choudhury and Juliano 1980). Starch lipids were extracted from the residue of nonstarch lipid extraction by refluxing three times with five volumes water-saturated 1-butanol (63:37 butanol-to-water ratio) for 8 hr (Choudhury and Juliano 1980). Extracts were pooled, evaporated, dried with anhydrous sodium sulfate, filtered, evaporated to dryness, and weighed.

Digestible starch was determined using the α-amylase-pullulanase method of Berry (1986), followed by amyloglucosidase treatment and glucose assay by glucose oxidase-peroxidase. The β-amylase-pullulanase method of Kainuma at al (1981) was also used.

Soluble Starch, Amylose, and Solids

Milled rice (1 g) was soaked for 30 min in 10 ml of water

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in a 150-ml beaker covered tightly with aluminum foil. The rice was boiled in a Toshiba RC 4B rice cooker for 25 min, followed by 10 min of additional standing after power turn-off. Excess rice water was decanted into a 100-ml volumetric flask, made to volume with distilled water, and designated as "A." Aliquots of A (10 ml) were added to 10 ml of 0.18*N* NaOH and allowed to stand overnight before iodine-colorimetric AC determination (Juliano et al 1981). For soluble starch determination, 1-ml aliquots of A were further diluted with 4 ml of distilled water. An aliquot of 0.10 ml of diluted A was used for starch assay using the phenol-sulfuric acid method with glucose as standard (Dubois et al 1956). The total amount of dissolved solids on cooking was determined by evaporating 40-ml aliquots of A to dryness in an oven at 80°C and weighing the residue.

Thermal and X-Ray Analyses

DSC studies were carried out using a DuPont 9900 thermal analyzer equipped with a DuPont 910 cell base and a pressure DSC cell (Biliaderis et al 1985, 1986b). The system was calibrated with indium. Pressure of 1,400 kPa with N_2 was used for all experiments to eliminate the problem of pan failure due to moisture loss at temperatures >120°C. All samples (30% w/w aqueous dispersions of ground rice grains) were prepared in DuPont coated hermetic pans by adding deionized water to a preweighed dry sample. A heat-sink compound (340, Dow Corning, Midland, MI) was used to improve the thermal contact between the pans and the thermocouple detectors. Data were collected at 0.4-sec intervals (heating rate 10°C/min). The melting-transition characteristics (enthalpy and peak temperature) were determined using the DuPont software analysis programs.

X-ray diffraction patterns of milled rice flours were obtained with a Philips PW 1710 diffractometer (at 30% moisture) equipped with a graphite crystal monochromator: radiation CuK_α, voltage 40 kV, recorder time constant 0.5 sec, sampling interval time 0.4 sec, scan speed 0.1 × 2 θ/sec, recorder speed 10 mm/2θ.

Statistical Analysis

Analysis of variance statistical evaluation of the data was used in a randomized block design. Differences among means were assessed using the LSD test.

RESULTS AND DISCUSSION

Properties of Raw Milled Rices

Rice AC confirmed that IR65 was waxy; IR24 and IR2071-137-5 were low AC; IR48 and IR64 were intermediate AC; and IR5 and IR8 were high AC (Table I). Alkali-spreading values confirmed that IR65, IR24, IR48, and IR8 had low gelatinization temperature (GT, alkali-spreading value 6–7), whereas the other rices had intermediate-high GT (alkali-spreading value 3–5). Starch lipids were higher in nonwaxy rices than they were in waxy IR65 and IR2071-137-5 rices (10.9% AC). IR48 had the highest starch lipids but the lowest nonstarch lipids. IR65 had the highest nonstarch lipids. Total lipids ranged from 2.2 to 2.4% for all rice samples.

Dissolved solids in cooking water of milled rices after 25 min of cooking was highest for IR48 and lowest for IR2071-137-5 (Table I). Among the paired samples (of similar amylose content) differing in GT, only the high-AC/intermediate-GT IR5 had higher dissolved solids than the low-GT IR8. The opposite was true for IR2071-137-5 versus IR24 and IR64 versus IR48. Among the milled rices, dissolved starch was highest for IR48 and lowest for IR2071-137-5. Among nonwaxy rices, IR5 had the highest proportion of dissolved amylose (6.0 out of 25.8%); IR2071-137-5 the lowest (1.0 out of 10.9%). The lowest dissolved amylose for IR65 was expected because of its low AC (0.9%), but it represented the greatest proportion of dissolved amylose (0.5 of 0.9%) among the samples.

Dissolved solids during cooking decreased substantially on parboiling except for IR65, IR2071-137-5, and IR24 (Table I). Hot-water soluble starch also showed the same trends. Hot-water soluble amylose tended to decrease with parboiling at 100 and 120°C, but it did not change much with increased parboiling temperature from 120 to 127°C (Fig. 1). Even the low-AC rices, IR24, IR2071-137-5, and waxy IR65, showed the trend of decreased extractable amylose with additional parboiling.

Hardness of freshly cooked rices was not significantly affected by parboiling of IR65, IR24, and IR2071-137-5 rough rice (Table II). Cooked IR8 and IR48 rices were slightly harder than IR5 and IR64 rices as raw milled rice samples. These differences were not significant for most of the samples parboiled at 120 and 127°C.

Digestible starch, using α-amylase and pullulanase (Berry 1986), for the cooked rices was low for IR5 and consistently high for IR65, IR24, and IR8 (Table III). In general, cooked raw rices exhibited starch digestibilities similar to those of their cooked parboiled counterparts. Only the cooked parboiled rices of IR65 and IR24 had digestible starch close to 100%. In contrast to retrograded amylose (resistant starch), retrograded amylopectin (Ring et al 1988) and all forms of V-amylose (Seneviratne and Biliaderis 1991) are completely digested by α-amylases. Although no substantial reduction in starch digestibilities was observed after the parboiling treatments (Table III), amylose in these products most likely exists in the form of inclusion complexes with lipids. Determination of digestible starch using the β-amylase-pullulanase method of Kainuma et al (1981) gave much lower digestibility values (as low as 75.6%), with the exception of IR65 (data not shown).

Thermal Properties

The DSC analysis confirmed the GT ranking of the rice starches. The four low-GT samples had peak T_m of 67.4–70.8°C. The intermediate-high-GT had peak T_m of 76.8–80.6°C (Table IV). Waxy IR65 showed mainly the gelatinization endotherm in raw rice and a trace of retrograded amylopectin (melting endotherm T_m at 45.5–58.5°C) in the product parboiled at 100°C. In addition to gelatinization endotherms, the nonwaxy low-GT rices showed the staling endotherm of amylopectin in IR48 parboiled at 120°C and in IR8 parboiled to 127°C. There was no amylopectin staling endotherm for the IR24 parboiled products.

All intermediate-high-GT rices showed the presence of annealed

TABLE I
Properties[a] of Milled and Parboiled Rices Differing in Amylose Content and Starch Final Gelatinization Temperature

Rice Variety	Apparent Amylose Content (%, db)	Lipids (%, db)		Dissolved Solids in Cooking Water (%)				Hot-Water Soluble Starch (%, db)				Hot-Water Soluble Amylose (%, db)			
		Starch	Nonstarch	Raw	100°C	120°C	127°C	Raw	100°C	120°C	127°C	Raw	100°C	120°C	127°C
IR65	0.9	0.3	1.8	8.6	7.6	9.4	10.6	8.1	6.9	9.9	10.3	0.5	0.4	0.1	0.1
IR24	15.8	0.9	1.5	9.6	6.4	6.6	8.4	10.0	4.9	5.9	7.1	2.0	1.3	0.2	0.2
IR2071-137-5	10.9	0.6	1.6	5.6	4.6	5.2	5.7	5.4	3.4	4.2	4.8	1.0	0.6	0.1	0.1
IR48	21.6	1.0	1.3	12.3	5.1	4.2	4.2	10.6	3.9	3.1	3.4	2.7	2.6	0.4	0.5
IR64	21.9	0.9	1.6	8.7	5.0	4.8	4.6	6.7	3.8	3.8	4.2	3.3	2.2	0.7	0.1
IR8	25.0	0.8	1.6	8.0	4.4	3.8	3.7	6.0	2.8	2.8	2.6	3.3	1.5	0.1	0.1
IR5	25.8	0.9	1.5	9.6	6.0	4.8	4.6	8.8	4.8	3.8	3.8	6.0	4.6	1.0	0.9
$LSD_{0.05}$	0.2	0.1	0.1	0.3	0.2	0.3	0.4	0.5	0.4	0.3	0.4	0.2	0.3	0.1	0.1

[a] Means of triplicate analyses.

ungelatinized starch crystallites (endotherm T_m of 85.1–87.5°C) for the samples parboiled at 100°C. IR2071-137-5 showed residual granular crystalline structures even after parboiling at 120°C (ΔH: 0.8 J/g, T_m: 87.5°C). The endotherm of residual annealed granular structures decreased progressively with the increasing severity of parboiling. IR2071-137-5 showed retrograded amylopectin in parboiled samples, while only the pressure-parboiled (120 and 127°C) products of IR5 had the staling endotherm. All IR64 samples were free of retrograded amylopectin.

IR2071-137-5 exhibited the highest gelatinization enthalpy for raw rice and residual granular structures left after parboiling (Table IV). The starch granules of this variety show a glass transition of the amorphous regions of the starch when heating in the calorimeter, as reported earlier by Biliaderis et al (1986b). The amounts of retrograded amylopectin in the parboiled samples (based on melting enthalpy at T_m 45.5–58.5°C) were highest for IR48 parboiled at 120°C and for IR5 parboiled at 127°C.

Among the low-GT samples, only waxy IR65 showed small amounts of amylose-lipid complex II (Table V). All other low-GT rices (IR24, IR48, and IR8) gave endotherms with T_m characteristic of complex I for both raw and parboiled products at 100°C. For these rices, the samples parboiled at 120 and 127°C

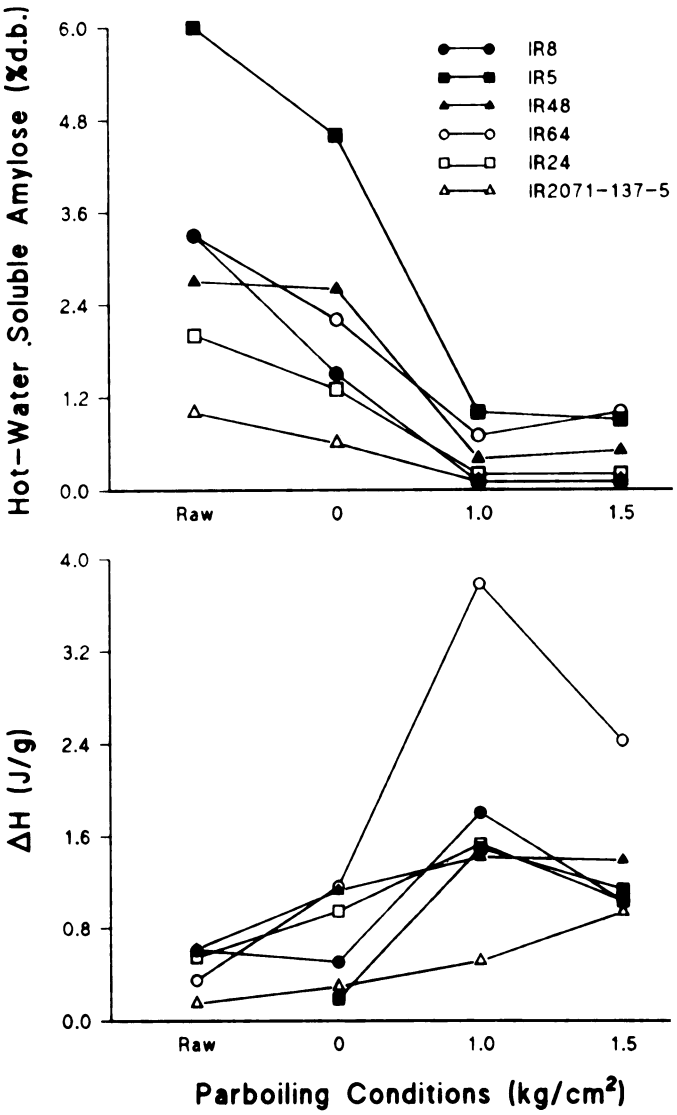

Fig. 1. Effect of parboiling conditions (100°C, 0 kg/cm^2; 120°C, 1.0 kg/cm^2; 127°C, 1.5 kg/cm^2) on hot-water soluble amylose and melting endotherm (ΔH) of amylose-lipid complex II of six nonwaxy rices.

TABLE II
Cooked Rice Hardness[a] (kg 17/cm^2) of Seven Freshly Cooked Raw and Parboiled Milled Rices

Rice Sample	Freshly Cooked Rice			
		Parboiled		
	Raw	100°C	120°C	127°C
IR65	7.6	6.4	7.0	5.8
IR24	7.1	7.6	7.9	7.6
IR2071-137-5	7.1	7.5	7.9	7.8
IR48	9.0	11.6	12.2	11.8
IR64	8.4	9.6	11.0	11.3
IR8	9.7	12.2	10.6	10.6
IR5	8.6	10.8	10.8	11.0
$LSD_{0.05}$	0.9	1.0	0.7	0.7

[a] Means of triplicate analyses.

TABLE III
Digestible Starch[a] (% of total) of Seven Cooked Raw and Parboiled Milled Rices by the α-Amylase-Pullulanase Method

Rice Sample	Cooked Milled Rice			
		Parboiled		
	Raw	100°C	120°C	127°C
IR65	101.1	99.5	99.4	101.1
IR24	100.0	99.6	98.7	100.4
IR2071-137-5	97.3	95.2	94.4	94.8
IR48	99.8	99.1	94.6	97.8
IR64	100.4	95.5	96.8	97.2
IR8	101.2	99.9	100.0	96.7
IR5	95.8	96.8	96.6	96.0
$LSD_{0.05}$	1.3	1.5	0.9	1.0

[a] Means of triplicate analyses.

TABLE IV
Differential Scanning Colorimetry Characteristics of Raw and Parboiled Rices

Rice Sample	Endotherm	Melting Enthalpy, Joules/g[a]			
		Raw	100°C	120°C	127°C
IR65	Gelatinization	8.0 (68.4°C)	0	0	0
	Retrograded amylopectin	...	0.1 (49.9°C)	0	0
IR24	Gelatinization	9.4 (70.8°C)	0	0	0
IR2071-137-5	Gelatinization	12.9 (80.6°C)	3.1 (87.5°C)[b]	0.8 (87.5°C)[b]	0
	Retrograded amylopectin	...	0.3 (55.5°C)	0.8 (55.6°C)	1.0 (55.6°C)
IR48	Gelatinization	7.5 (67.4°C)	0	0	0
	Retrograded amylopectin	...	0	3.7 (45.5°C)	0
IR64	Gelatinization	8.3 (78.0°C)	2.0 (85.1°C)[b]	0	0
IR8	Gelatinization	8.6 (68.6°C)	0	0	0
	Retrograded amylopectin	...	0	0	2.8 (50.0°C)
IR5	Gelatinization	8.5 (76.8°C)	1.8 (85.5°C)[b]	0	0
	Retrograded amylopectin	...	0	1.7 (55.0°C)	7.1 (58.5°C)

[a] Means of triplicate differential scanning calorimetry measurements (30% w/w aqueous dispersions of milled rices, heating rate 10°C/min); $LSD_{0.05}$ for melting enthalpy values 0.3 Joules/g.
[b] Annealed structures of starch granules.

showed only complex II. In contrast, the intermediate-high-GT rices (IR2071-137-5, IR64, and IR5) did not show any evidence of the presence of complex I in raw or parboiled products. The only polymorph found in these specimens was complex II. Figure 2 shows this contrasting DSC behavior of low-GT (IR48) and intermediate-GT (IR64) rices.

The results of this study clearly indicate that complex I is found only in low-GT rices parboiled under mild conditions. This can be rationalized as follows. First, an absolute requirement for amylose-lipid complex formation is sufficient molecular mobility of the starch chains. This is usually accomplished by gelatinization of granules, which is confirmed by calorimetry (Biliaderis et al 1986b). Second, between the two polymorphs, complex I is the kinetically preferred structure that is formed rapidly at relatively low temperatures (Biliaderis and Galloway 1989). In contrast, complex II is a thermodynamically favored structure (state of lower free energy than that of complex I) and is formed at high crystallization temperatures. DSC data (Table V) indicate that mild parboiling (100°C) is adequate to gelatinize the starch in low-GT rices; thereby fostering rapid association of lipids with amylose in the form of complex I. However, if these samples are processed at higher temperatures (120 or 127°C), complex II would be the developed structure. The melt-mediated transformation of complex I to complex II occurs at high temperatures (Biliaderis and Galloway 1989). For the intermediate-high-GT rices, much higher temperatures should be reached before any substantial chain mobility commences. At such high temperatures, formation of the more thermodynamically stable form of the complex (II) is preferred.

The X-ray diffractograms of the rice samples clearly showed the disappearance of the A-type pattern of the raw rices (Fig. 3a,e) and the development of the V-pattern during parboiling, particularly for pressure-parboiled products. For IR5, the diffraction lines of V-amylose (i.e., peak at $2\theta = 19.9°$) increased pro-

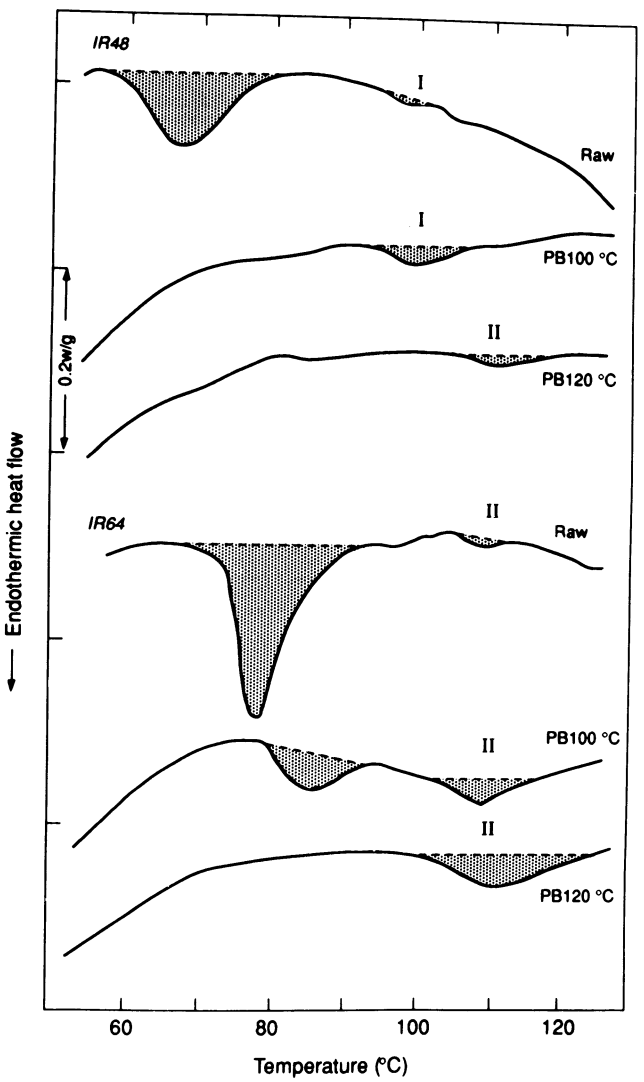

Fig.2. Effect of parboiling temperature (PB100°C and PB120°C) on the differential scanning calorimetry thermal curves of IR48 (low gelatinization temperature) and IR64 (intermediate gelatinization temperature) milled rice. Endotherms I and II correspond to the two types of amylose-lipid complexes.

TABLE V
Differential Scanning Calorimetry Melting Characteristics of Amylose-Lipid Complexes in Raw and Parboiled Milled Rices[a]

		Melting Enthalpy of Amylose-Lipid Complexes in Rices (Joules/g)			
			Parboiled		
Rice Sample	Amylose-Lipid Complex[b]	Raw	100°C	120°C	127°C
IR65	II	0.2	0.4	0.1	0.1
IR24	I	0.6	1.0	0	0
	II	0	0	1.5	1.0
IR2071-137-5	II	0.2	0.3	0.5	0.9
IR48	I	0.6	1.1	0	0
	II	0	0	1.4	1.4
IR64	II	0.4	1.2	3.8	2.4
IR8	I	0.6	0.5	0	0
	II	0	0	1.8	1.0
IR5	II	0	0.2	1.5	1.1

[a] Means of triplicate differential scanning calorimetry measurements on 30% (w/w) aqueous dispersions of raw and parboiled milled rices (heating rate 10°C/min); $LSD_{0.05}$ for melting enthalpies = 0.1 Joules/g.
[b] Complex I with T_m of 97.2–100.1°C, complex II with T_m of 108.0–114.6°C.

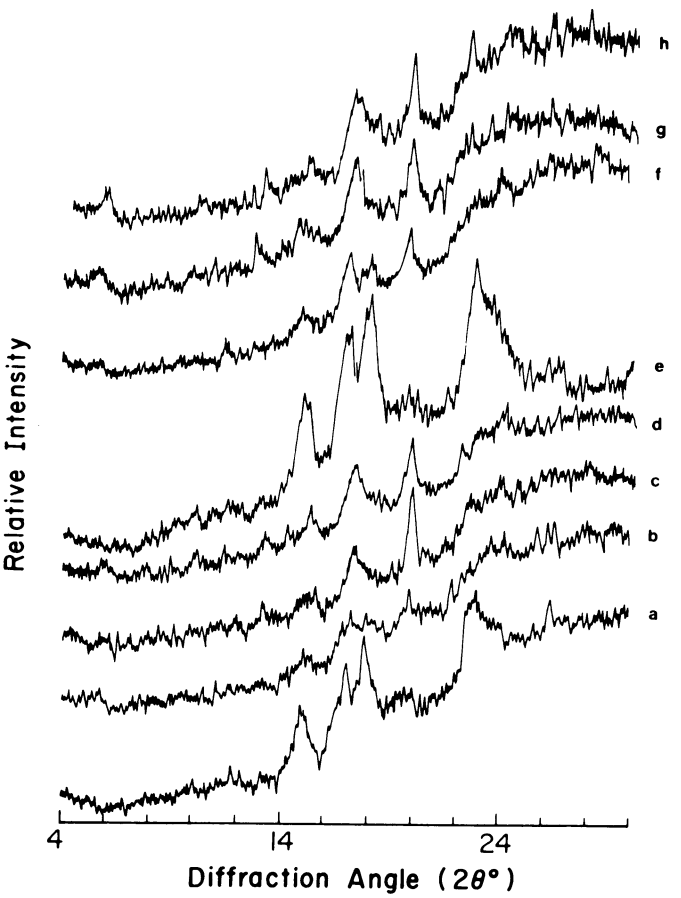

Fig. 3. Effect of parboiling conditions on the X-ray diffractograms of IR64 (**a–d**) and IR5 (**e–h**) milled rice. Treatments are raw (**a** and **e**) and parboiled at 100°C (**b** and **f**), at 120°C (**c** and **g**), and at 127°C (**d** and **h**).

gressively with degree of parboiling. For IR64, the more intense V pattern was observed for the sample parboiled at 120°C; these findings are in agreement with the DSC results (Table V).

The formation of a particular supermolecular structure of amylose-lipid complexes has practical importance for thermally processed, starch-based foods (Biliaderis 1991). For parboiled rice products, end-product quality is related to the type and amount of complex formed during parboiling. For example, the loss of soluble amylose in cooking water is minimized when the parboiling conditions favor extensive formation of complex II (Fig. 1). Trends were similar for dissolved total solids (mainly starch) during cooking. Furthermore, a firmer texture for some pressure-parboiled rice samples may be due, in part, to the crystalline structures in complex II. Amylose-lipid helices of this polymorph are thermally stable at temperatures >100°C and, thus, can act as physical cross-links to stabilize the rice grains during cooking in boiling water. The structure-property profiles presented in Figure 1 could be very useful in modeling and optimizing processing parameters (temperature, moisture, time, raw product characteristics, etc.) during parboiling for desired end-product quality.

ACKNOWLEDGMENT

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