

Physicochemical Properties of Starch in Extruded Rice Flours

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ABSTRACT

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The effects of extruding temperatures and subsequent drying conditions on X-ray diffraction patterns (XRD) and differential scanning calorimetry (DSC) of long grain (LG) and short grain (SG) rice flours were investigated. The rice flours were extruded in a twin-screw extruder at 70–120°C and 22% moisture, and either dried at room temperature, transferred to 4°C for 60 hr, or frozen and then dried at room temperature until the moisture was 10–11%. The dried materials were milled without the temperature increasing above 32°C. XRD studies were conducted on pellets made from extruded and milled flours with particle sizes of 149–248 µm; DSC studies were conducted from the same material. DSC studies showed that frozen materials retrograded more than the flours

dried at room temperature. The LG and SG samples had two distinct XRD patterns. The LG gradually lost its A pattern at >100°C, while acquiring V patterns at higher temperatures. SG gradually lost its A pattern at 100°C but stayed amorphous at the higher extruding temperatures. DSC analysis showed that retrograded flours did not produce any new XRD 2θ peaks, although a difference in 2θ peak intensities between the LG and SG rice flours was observed. DSC analysis may be very sensitive in detecting changes due to drying conditions, but XRD data showed gradual changes due to processing conditions. The gradual changes in XRD pattern and DSC data suggest that physicochemical properties of the extruded rice flours can be related to functional properties.

Rice is an important grain and most of it is consumed as a milled kernel. The United States is an important exporter of rice because its production is far greater than its domestic needs. The United States has one of the most advanced food technology industries to produce novel foods to meet the nutritional convenience and aesthetic needs of its affluent population. Rice is a hypoallergenic, easily digested product with a bland taste and desirable functional properties. Therefore, there is increased interest, in spite of a cost higher than that of corn and wheat, for using rice in novel, value-added foods (Kadan et al 1997, 2001a).

Extrusion cooking, a continuous, high-temperature, short-time process, is used to produce breakfast cereals, snacks, precooked flours, and dietetic foods from grains (Camire 1990). Extrusion cooking has been widely used for corn, wheat, and soybean products for a long time and, in recent years, has been used to make rice food products (Pan et al 1992). During extrusion, cereal components, particularly the starch moiety, undergo structural changes that can affect their functional properties (Bryant et al 2001). Moist heat, as in extrusion, makes the starch granules lose crystallinity and results in physicochemical changes that can be detected by X-ray diffraction (XRD) and differential scanning calorimetry (DSC).

XRD of rice has been used extensively to study changes in starch crystallinity during processing (Bear and French 1941; Roseman and Deobold 1959; Fukui and Nikuni 1969; Juliano et al 1969; Islam et al 1974; Mercier et al 1979; Zobel et al 1988; Mahanta et al 1989; Hibi et al 1990, 1993; Biliaderis et al 1993; Ong and Blanshard 1995; Yamamoto 1995) and to classify rice cultivars (Zhang et al 1993). These studies were conducted on rice cultivars either not grown in the United States (Fukui and Nikuni 1969; Juliano et al 1969; Mercier et al 1979; Mahanta et al 1989; Hibi et al 1993; Biliaderis et al 1993; Ong and Blanshard 1995) or on cultivars no longer grown in the United States (Roseman and Deobold 1959; Islam et al 1974). DSC has been used to study the effects of varietal differences on processing characteristics of rice grain (Juliano 1982), properties of rice flours (Normand and Marshall 1989; Marshall et al 1990; Marshall and Normand 1991), and related studies (Liu and

Lelievre 1991; Chang and Liu 1991). No information is available on the effects of extrusion processing on the XRD and DSC patterns of the two rice cultivars in this study. Such information can help understand the physicochemical and functional properties of samples and thus predict the expected performance of extruded materials in novel foods. Some U.S. rice cultivars have shown promise as a starting material to make a rice-fry-like food using an extrusion process (Kadan et al 2001b).

This study reports the results of extrusion temperatures on XRD and DSC patterns on two rice cultivars. Flours from a popular U.S. long grain (LG), high amylose (Cypress) rice and a short grain (SG), waxy (NFD 108) rice were used to conduct the studies. These two cultivars have shown promise for potential use in rice fry foods (Kadan et al 2001b,c) and whole rice bread (Kadan et al 2001a).

MATERIALS AND METHODS

Rice Cultivars

Two U.S. rice cultivars with high or virtually no amylose content were used in this study. The LG high amylose rice (Cypress) was purchased from Riceland Foods, Stuttgart, AR, and the SG (waxy) rice (NFD 108) was purchased from Farmer's Rice Cooperative, Sacramento, CA. Rice was ground in a pin mill (Kolloplex, Alpine, Augsburg, Germany) to 100 mesh, and the flour was stored at 4°C. Moisture, ash, and fiber level were determined by Official Method 32.1.03 (AOAC 2000). Protein content (N × 5.95) was determined by combustion (Method Ba4e-93, AOCS 1998) using a nitrogen analyzer (model FP-428, Leco, St. Joseph, MI). Lipids were extracted with 100 mL of petroleum ether for 6 hr from 5-g samples of milled flour (Approved Method 30-25, AACC 2000). Amylose content was determined using a spectrophotometric assay developed by Juliano (1971).

Extrusion Conditions

A self-cleaning co-rotating twin-screw extruder (model ZSK-30, Werner-Pflederer, Ramsey, NJ) with a 7-mm die was used to produce the extruded materials. The screws were 975 mm long; the configuration from feed to die was (number of elements-screw flight pitch/length in mm) 1-spacer/10, 2-42/42, 1-42/21, 2-42/42, 5-28/28, 5 kneading block with 45 offset/20, 20-20/20, and 2-spacer/1. The extruder drive capacity was 10 kW at 100 rpm. All zones (2–5) but the first were heated at 70, 80, 90, 100, 110, or 120°C. The extrudate temperature was taken with a thermocouple located behind the die and was within 5°C of the zone 5 temperature. The flour feed rate was 100 g/min. The moisture of the feed was increased to 22% by adding deionized water through a water port in zone 1. The moisture content of rice flour during extrusion could not be measured directly,

¹ USDA, ARS, Southern Regional Research Center, New Orleans, LA 70179. Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by the USDA implies no approval of the product to the exclusion of others that may also be suitable.

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therefore moisture was determined as 20% after extrusion on carefully collected extrudate. Immediately after extrusion, the extrudate was divided into two parts. The first part was collected in a flat tray and dried at room temperature at 10–11% moisture. The second part was placed in an airtight plastic bucket, frozen at –10°C for 60 hr, stored at 4°C for 60 hr, and then dried at room temperature on a flat tray at 10–11% moisture. The dried material was broken into 5-mm particles (Retsch Muhle mill, Germany) and then ground in a Kolloplex impact pin mill. The temperature of the milled material was monitored and did not exceed 32°C. The milled flour was screened with ASTM 60-mesh and 100-mesh sieves, and the material retained by the 100-mesh screen (149–248 μm particle size) was collected for XRD and DSC studies.

X-ray Diffraction

Rice flour samples (≈0.7 g) were pressed into 10 × 25 mm pellets on a hydraulic press (2,300 kg). The pellets were mounted at the center of the goniometer circle, and X-ray diffraction data was obtained with an X-ray diffractometer (model D-Max B, Rigaku/USA, Danvers, MA). The instrument was equipped with a water-cooled rotating copper anode that produced Cu Kα X-rays with an accelerating voltage of 40 kV and a tube current of 50 mA. The goniometer, equipped with a photomultiplier detector (to count X-ray photons), scanned an angular range (2θ) between 8° and 28° at a scan rate of 0.5°/min. Three replicates of each sample were run.

Differential Scanning Calorimetry (DSC)

Milled flours underwent DSC using a differential scanning calorimeter (Hart Scientific model 4100, Calorimetry Sciences, Spanish Fork, UT). The rice flour samples (0.25 g) were weighed directly into the ampules. Deionized water (0.50 g) was added to each ampule. Samples were subjected to DSC analysis (Kadan et al 2001a); enthalpy (ΔH) was calculated as J/g of dried flour by integration of the thermal curves. The average of two separate determinations were made to yield reported values.

RESULTS AND DISCUSSION

Proximate compositions of the two rice flour samples investigated are shown in Table I. The composition of LG and SG rice cultivars are typical. However, the SG rice had virtually 100% amylopectin (<0.1% amylose) while the LG had 21.5% amylose. The LG amylose sample also had higher protein (8.2%) and slightly higher lipid (0.79%) contents than the SG rice (6.4 and 0.59%, respectively).

XRD patterns of LG and SG extruded rices are presented in Fig. 1, while the distribution and average intensities of three replicates of the peaks for the two extruded rice flours are given in Tables II and III. The control rice flours from both cultivars (Fig. 1A and C) showed typical A patterns (Biliaderis et al 1993; Hosoney 1994). However, there are major differences in the peaks of control LG and SG flours. The LG control flour has a peak at 19.3° but no

TABLE I
Proximate Composition (%) of Unextruded Rice Flours

Rice Cultivars	Amylose	Moisture	Protein	Lipid	Ash	Fiber
Short grain (waxy, NFD 108)	0.0	12.0	6.4	0.5	0.4	0.2
Long grain (Cypress)	21.5	12.1	8.2	0.7	0.5	0.1

TABLE II
Distribution of Peak Intensities for Extruded and Frozen Extruded Long Grain (Cypress) Rice^a

Sample	2θ 13.1°/Å 6.75	2θ 15.2°/Å 5.84	2θ 17.2°/Å 5.18	2θ 18.1°/Å 4.91	2θ 19.3°/Å 4.61	2θ 23.4°/Å 3.79
Control	np ^b	284	342	358	292	323
LG-70	np	293	336	348	321	289
LG-80	np	263	290	301	290	253
LG-90	np	229	260	264	266	212
LG-100	233	np	np	np	304	np
LG-110	251	np	np	np	374	221
LG-120	279	np	np	np	310	222
FLG-70	np	257	308	309	286	235
FLG-80	np	203	225	242	214	206
FLG-90	np	np	np	252	261	197
FLG-100	231	np	np	np	313	230
FLG-110	257	np	np	np	391	237
FLG-120	251	np	np	np	183	98

^a Function of X-ray angle 2θ or lattice spacing Å. Values represent averages of three replicate analyses.

^b No peak is present.

TABLE III
Distribution of Peak Intensities for Extruded and Frozen Extruded Short Grain (waxy, NFD 108) Rice^a

Sample	2θ 13.1°/Å 6.75	2θ 15.2°/Å 5.84	2θ 17.2°/Å 5.18	2θ 18.1°/Å 4.91	2θ 19.3°/Å 4.61	2θ 23.4°/Å 3.79
Control	np ^b	347	398	387	np	308
SG-70	np	318	368	387	np	334
SG-80	np	279	321	343	np	301
SG-90	np	307	352	374	np	322
SG-100	np	233	np	251	np	216
SG-110	np	np	np	np	np	np
SG-120	np	np	np	np	np	np
FSG-70	np	242	281	293	np	251
FSG-80	np	366	421	343	np	388
FSG-90	np	259	287	301	np	267
FSG-100	np	167	187	170	np	141
FSG-110	np	np	np	np	np	np
FSG-120	np	np	np	np	np	np

^a Function of X-ray angle 2θ or lattice spacing Å. Values represent averages of three replicate analyses.

^b No peak is present.

peak at 13.1°. The SG flour has no peaks and, hence, no intensities at 13.1° and 19.3°. Islam et al (1974) studied the X-ray diffraction of Bluebelle and Bluebonnet, two long grain cultivars, which are no longer grown in the United States. These workers, unlike the present study, did not observe any 2θ peak intensities at 13.1° and 19.3°. They also reported that Bluebonnet cultivar had an additional peak at 24.4° that was absent in Bluebelle cultivar. Similarly, Mahanta et al (1989) reported a 2θ peak at 20.0° for an Indian-grown LG cultivar not used in this study. Juliano et al (1969) and Zhang et al (1993) reported that cropping season and cultivation conditions can significantly affect the X-ray diffraction intensities. These workers did not report either the proximate composition or the amylose contents of their rice samples. It is not clear whether the absence of 2θ peaks at 13.1° and 19.3° or the presence of additional peaks at 20.0° and 24.4° in these studies is due to differences of amylose content or differences in the growing locations and conditions.

The principal indexes for the LG flour were at 2θ = 13.1°, 15.2°, 17.2°, 18.1°, 19.3°, and 23.4°. These peaks correspond to interplanar distances of 6.75Å, 5.84Å, 5.18Å, 4.91Å, 4.61Å, and 3.79Å, respectively. The diffraction intensities of the extruded LG flour samples dried immediately at room temperature as well as after freezing decreased with increases in extrusion temperatures except for the

2θ intensity at 19.3° and 13.1°. Overall, the frozen long grain (FLG) had lower peak intensities than LG, except at 19.3°. The gradual disappearance of 2θ intensities at 15.2°, 17.2°, and 18.1° (100, 110, and 120°C) coincided with the appearance of 2θ peaks at 13.1° and V patterns. The 2θ intensities at 15.2°, 17.2°, and 18.1° were absent in LG extruded at ≥100°C. The 2θ peak intensities in FLG at 15.2° and 17.2° that disappeared at 90°C and 18.1° were absent in FLG extruded at ≥110°C. The magnitude of the diffraction intensities is influenced by the crystallinity of the starch moiety. The amylose content and gelatinization temperature of starch are considered important characteristics of cooking qualities of milled rice products (Juliano 1965). Therefore, the 2θ peak intensities that decrease with increasing extruding temperatures show the relative loss of rice starch crystallinity. On the other hand, 2θ intensities that increase with the increases in extruding temperature show the degree of cook and the resultant rearrangement of the starch moiety. The appearance of a 2θ peak at 13.1° also suggests complete gelatinization or loss of crystallinity of the starch moiety, as it also coincides with the loss of A form diffractogram. The XRD patterns of LG (Fig. 1A) showed that LG has a typical V pattern (Hoseney 1994) for extruded samples at ≥110°C.

Earlier workers (Roseman and Deobald 1959; Mahanta et al 1989) found that some rice cultivars gave 2θ peaks at 22.0° and

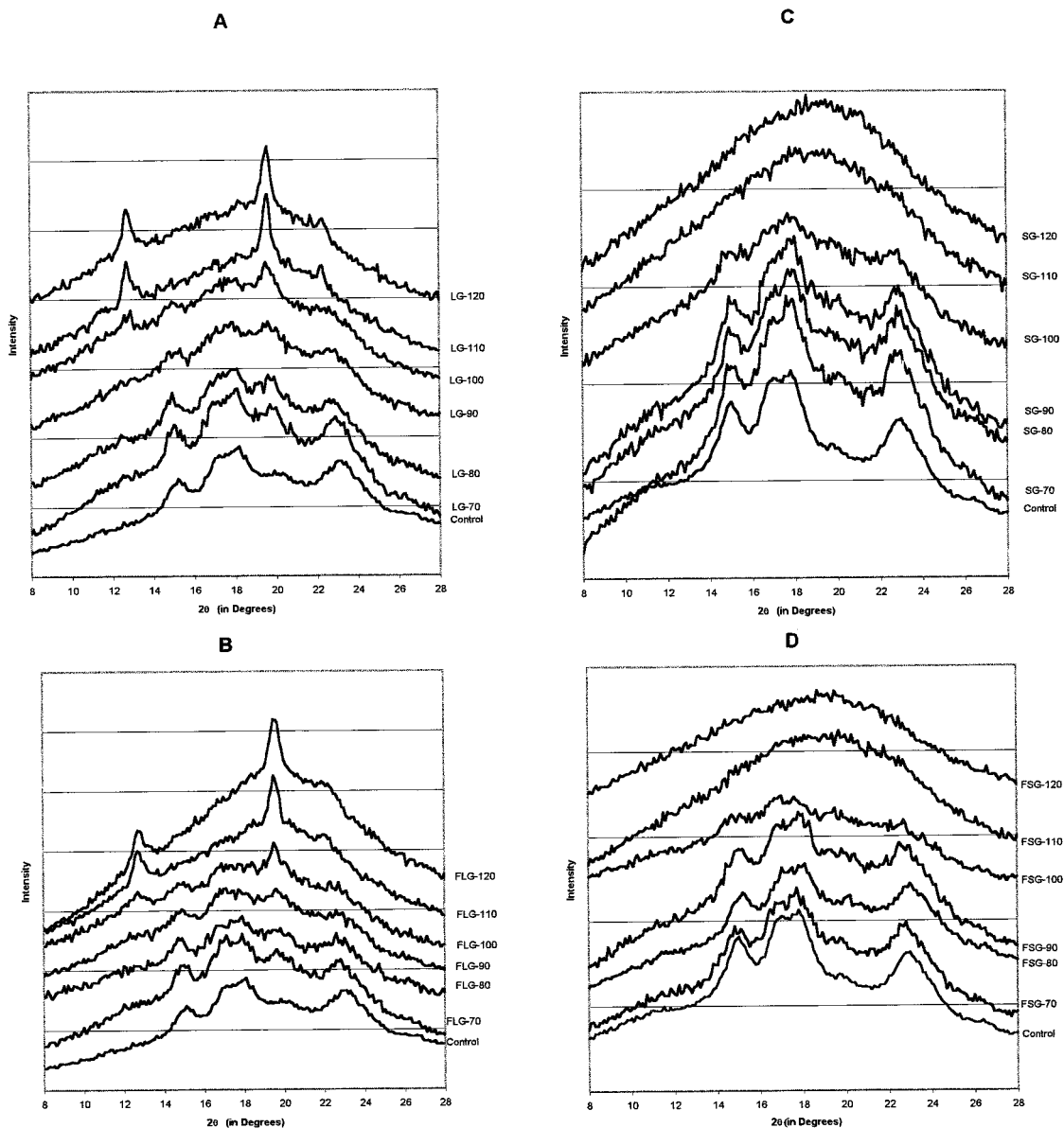


Fig. 1. X-ray diffractogram of long grain (A), frozen long grain (B), short grain (C), and frozen short grain (D) rice samples extruded at 70–120°C.

24.0° and acquired a B pattern during parboiling or freezing before finally changing to a V pattern. These workers used a much higher water content (>60%) than in the present study (22%). Apparently in low moisture extrusion, LG changed from an A pattern to a V pattern without any evidence of undergoing a B pattern because no 2θ peaks at 22.0° and 24.0° were observed. The XRD for FLG generally followed the same pattern as LG, except that the intensities were lower. The decrease in intensities of FLG are probably due to the interaction of rice protein and lipid with the starch moiety during freezing, which can further affect XRD.

The XRD patterns of SG and frozen short grain (FSG) are shown in Fig. 1C and D and their peak intensities are given in Table III. The 2θ peak intensities at 15.2°, 17.2°, 18.1°, and 23.4° generally decreased with the increase in extruding temperatures and completely disappeared at 110°C. The absence of peaks at 19.3° (Table III) in control SG and in high-temperature extruded samples probably reflects the absence of the amylose moiety of the starch. The XRD of the control SG and the 70–90°C extruded samples exhibited characteristic A patterns that were completely lost at 110°C and with no apparent V pattern formed, even at 120°C.

Retrogradation has been described as an attempt toward crystallization of large cooked starch in the gel state (Whistler 1965; Hosney 1994). The conformational changes thus produced can be evaluated by DSC (Normand and Marshall 1989; Marshall and Normand 1991). The DSC enthalpies (ΔH) of extruded LG, FLG, SG, and FSG samples are given in Table IV. The unextruded LG has a peak temperature (T_p) of complete gelatinization at 78.7°C (pk-2) and ΔH of 13.4 J/g. In 70°C (LG-70) extruded flour, the ΔH decreased, as expected, but an unanticipated peak (pk-4) with T_p at 130.0°C and ΔH of 2.6 J/g also appeared. This peak (pk-4) is absent in the other extruded rice flours. Peak 2 disappeared at 110°C. In the 90 and 100°C extruded flours, two other peaks (pk-1 and pk-3) appeared. In this temperature range, the LG was completely gelatinized and appearance of pk-1 and pk-3 indicated the onset of some retrogradation. The appearance of pk-1 was also repeated by Kadan et al (2001a) in retrograded whole rice bread (T_p 52.3/54.4°C) and whole wheat bread (T_p 51.7°C). The substantial decrease

in ΔH for pk-2 of FLG-70 and FLG-80 as compared with LG-70 and LG-80, as well as the absence of pk-4, suggests that the freezing process, unlike drying at room temperature, does not preserve retrogradation but instead promotes it. The FLG samples had a more pronounced retrogradation as indicated by higher ΔH in appearances of pk-1 and pk-3, in FLG-70 and FLG-80, 90, 100, and higher than corresponding LG.

The waxy SG was essentially gelatinized at 80°C as indicated by very small ΔH for pk-2. Unlike LG, all SG 70–100 flours had pk-3 and their ΔH values were higher than those for LG. There was no noticeable pk-1 in any of the SG flour samples, suggesting resistance to retrogradation. All FSG flour samples had pk-1. The pk-2 was absent in FSG-70 (unlike FLG-70), again indicating that freezing does not stop physicochemical changes such as transformation of a starch molecules and, thus, promotes different transformational changes. Amylose is generally assumed to be a linear polymer with minor branching and has a much lower molecular weight than amylopectin, which is a large molecule of branched chain (Hosney 1994). Processed cereals, which are amylose-containing foods with high moistures, undergo retrogradation during storage at $\leq 25^\circ\text{C}$ (Whistler 1965). The waxy SG flours were essentially amylose-free; nevertheless, they underwent noticeable retrogradation, suggesting that amylopectin can also undergo some retrogradation. It appears that pk-1 at T_p of 52.3–54.4°C is characteristic of retrogradational changes as it was also found in retrograded whole and wheat bread (Kadan et al 2001a).

The presence of a B X-ray diffraction pattern is considered to be evidence of retrograded starch (Roseman and Deobald 1959; Hosney 1994). None of the LG or SG samples exhibited any evidence of a B X-ray diffraction pattern. Apparently, some retrogradation can occur without affecting XRD or appearance of a B X-ray diffraction pattern as detected by DSC thermogram (Table IV). According to Ong and Blanshard (1995), absence of a B pattern does not eliminate the occurrence of retrograded starch.

In conclusion, LG and SG flours exhibited two very distinct X-ray diffraction patterns on samples extruded at $>100^\circ\text{C}$. The LG showed the appearance of a 2θ peak at 13.1° in samples dried at room

TABLE IV
Differential Scanning Calorimetry (DSC) Characteristics^a of Long Grain, Frozen Long Grain, Short Grain, and Frozen Short Grain Rice Flours

Sample	Peak 1		Peak 2		Peak 3		Peak 4	
	T_p	ΔH	T_p	ΔH	T_p	ΔH	T_p	ΔH
Unextruded long grain	np ^b	np	78.7	13.4	np	np	136.0	3.0
LG-70	np	np	77.4	11.1	103.0	trace ^c	130.0	2.6
LG-80	np	np	78.3	8.4	105.4	trace	np	np
LG-90	54.5	0.5	80.5	1.8	115.4	0.1	np	np
LG-100	54.2	0.4	81.0	1.2	113.5	1.2	np	np
LG-110	np	np	np	np	111.0	1.3	np	np
LG-120	np	np	np	np	109.0	0.7	np	np
FLG-70	48.0	0.4	78.2	4.5	102.7	1.0	np	np
FLG-80	48.3	0.8	78.6	4.1	105.2	2.0	np	np
FLG-90	47.0	2.1	80.0	2.4	117.0	1.5	np	np
FLG-100	49.7	0.8	81.5	1.5	118.0	1.0	np	np
FLG-110	np	np	np	np	109.3	0.6	np	np
FLG-120	np	np	np	np	108.5	0.2	np	np
Unextruded short grain	np	np	76.2	13.2	111.8	0.0	np	np
SG-70	np	np	73.9	1.2	125.0	4.2	np	np
SG-80	np	np	74.9	1.2	120.0	5.1	np	np
SG-90	np	np	75.9	2.1	111.5	1.3	np	np
SG-100	np	np	np	np	118.5	3.2	np	np
SG-110	59.1	trace	np	np	np	np	np	np
SG-120	np	np	np	np	np	np	np	np
FSG-70	47.4	np	np	np	np	np	np	np
FSG-80	50.0	1.2	74.3	2.0	116.1	3.8	np	np
FSG-90	44.3	trace	74.3	1.6	120.0	5.6	np	np
FSG-100	45.2	0.4	76.3	1.8	110.0	1.8	np	np
FSG-110	54.9	0.7	np	np	np	np	np	np
FSG-120	55.5	0.4	np	np	134.3	0.7	np	np

^a Peak temperature (T_p , °C) and enthalpy (ΔH , J/g).

^b No peak is present.

^c Trace value <0.01.

temperature as well as frozen. Appearance of this peak also indicates a gradual transformation from A-type diffractogram to a V X-ray diffraction pattern. The SG samples had no 2 θ peaks at 13.1° and 19.3° and gradually lost other 2 θ peak intensities at 15.2°, 17.2°, 18.1°, and 23.4° and showed no evidence of V patterns even in samples extruded at $\geq 100^\circ\text{C}$. It is hypothesized that a 2 θ peak at 19.3° is caused by the presence of amylose in the rice starch. Upon extrusion at $>100^\circ\text{C}$, the amylose is responsible for appearance of a 2 θ peak at 13.1° and a V pattern of X-ray diffraction. Apparently, the functional properties of LG or SG flours extruded at 90°C or $>100^\circ\text{C}$ will be very different and may be uniquely suited for certain food products. Extrusion processing conditions of rice flour affect functional properties (Bryant et al 2001). However, only the XRD pattern showed consistent changes with the increase in extrusion temperature. DSC, on the other hand, showed predictable gradual changes only up to complete gelatinization of the starch moiety. The DSC also detected some conformational changes that appeared only in retrograded material. DSC also appeared to be highly sensitive to very minor changes in processing conditions, once the starch had been gelatinized. Conversely, XRD gave more predictable gradual differences in 2 θ peaks.

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