EFFECTS OF POSTHARVEST OPERATIONS ON THE GELATINIZATION AND RETROGRADATION PROPERTIES OF LONG-GRAIN RICE

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ABSTRACT. *The effects of rough rice pre-drying, drying, and storage treatments on the gelatinization and retrogradation properties of long-grain rice (cv. Cypress) were studied via differential scanning calorimetry (DSC). The experimental variables included two pre-drying conditions (immediate and delayed drying), two drying conditions (high and low air temperatures), and four storage treatments (no storage, and storage at 4, 21, and 38°C for 20 wk). Gelatinization of the rice flour in water occurred at temperatures from 73 to 86°C, with an enthalpy of 8.3 to 9.7 J/g. Storage temperature had a significant effect on the gelatinization characteristics. Rice stored at 38°C exhibited higher ($p < 0.0005$) gelatinization enthalpy and peak temperature than did rice stored at 4 and 21°C. Pre-drying treatments had no significant effect on the gelatinization enthalpy and peak temperature, but did on the onset temperature ($p < 0.05$). The pre-drying and/or drying treatments had significant ($p < 0.05$) effects on the retrogradation onset and conclusion temperatures of gelatinized rice. Storage of rough rice significantly ($p < 0.0001$) increased retrogradation enthalpy, but did not affect the peak temperature of the retrogradation endotherm.*
Keywords. *Grain storage, Drying, Gelatinization, Retrogradation, Differential scanning calorimetry.*

It is common practice for freshly harvested rice to be dried to a safe moisture content and then stored for some months prior to milling. This process has a significant effect on rice chemistry and functionality. For example, enzymatic activities change as a function of storage time (Yasumatsu et al., 1964; Dhaliwal et al., 1991). Free fatty acids, carbonyl compounds, and hardness index increase with storage duration (Villareal et al., 1976). Longer storage can also result in more starch degradation (Kester et al., 1956; Tani et al., 1964) and more reducing sugars (Pushpamma and Reddy, 1979), even though total starch content does not markedly change. Additionally, protein quality and protein solubility decrease in stored rice, particularly at higher storage temperatures (Chrastil, 1990a).

The cooking properties and eating quality of rice also change significantly during storage. A major increase in amylograph peak viscosity occurs during storage, particularly within the first few months (Villareal et al., 1976; Perez and Juliano, 1981; Perdon et al., 1997). The swelling ability and water uptake ratio of cooked rice increase with storage time (Villareal et al., 1976; Chrastil 1990b; Perdon et al., 1997). The cooked rice also becomes harder and less sticky after rough rice storage at a higher temperature or moisture content (Perez and Juliano, 1981; Tamaki et al., 1993). Indudhara Swamy et al. (1978) observed a slow decrease, during storage, in the solubility of amylose and in the water uptake ratio of rice cooked at 80 and 96°C, and postulated that there was also a slow increase in the gelatinization temperature during storage. Although some literature is available regarding changes in composition and quality of rice during storage, data on thermal properties of rice and rice flours, as affected by postharvest handling, are currently lacking.

Differential scanning calorimetry (DSC) has been used extensively to investigate phase transitions of starch and starch-based foods during heating or cooling (Biliaderis et al., 1980). Russell and Juliano (1983) used DSC to characterize the gelatinization properties of eight rice starches, pretreated with sodium dodecyl benzenesulfonate to remove milled rice proteins. Marshall (1992) and Normand and Marshall (1989) employed DSC techniques to investigate the gelatinization behavior of whole grain milled rice. They found that the whole rice grain exhibited two endothermic transitions, whereas rice flour exhibited only one endotherm. Chang and Liu (1991) reported that the retrogradation enthalpy for high-amylose rice starch was higher than those for low-amylose or waxy starches. In a recent study of retrogradation kinetics of rice flours from different cultivars, Fan and Marks (1998) demonstrated that long-grain rice retrograded faster than did medium-grain rice and that the rate of retrogradation was directly related to the amount of amylose present in the rice materials. None of these studies, however, evaluated the effects of postharvest operations on gelatinization and retrogradation properties of rice.

Therefore, the objective of this project was to quantify, via differential scanning calorimetry (DSC), the effects of rough rice pre-drying, drying, and storage treatments on the gelatinization and retrogradation behaviors of rice flour.

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Materials and Methods

Drying and Storage

The rough rice used in this study was a long-grain cultivar, Cypress, which was harvested via a plot combine from the University of Arkansas Rice Research and Extension Center (Stuttgart, Ark.) at 20.5% moisture content (m.c.) in the fall of 1995. (All moisture contents are reported as wet basis.) The rice was transported to the University of Arkansas Rice Processing Laboratory (Fayetteville, Ark.) on the day of harvest, and cleaned in a Carter-Day dockage tester (Seedboro Equipment Co., Chicago, Ill.).

Subsequent postharvest variables included two pre-drying conditions (immediate or delayed by 86 h), two drying conditions (high or low temperature), and four storage treatments (no storage or storage at 4, 21, or 38°C for 20 wk). For the immediately dried samples, the rough rice was dried immediately after cleaning (on the same day as harvest); for delayed-dried samples, the rice was held in insulated containers for 86 h at the harvest moisture content, then subjected to the same drying procedures as were the immediately dried samples. This was done to simulate an extended period of wet holding prior to drying, as might occur during the peak drying season. During wet holding, the rice temperature did not increase more than 3°C above the ambient temperature of approximately 20°C.

Two drying treatments were applied to the rough rice: high-temperature (54.3°C, 21.9% rh, for 45 min) and low-temperature (33.0°C, 67.8% rh, for 45 min), which corresponded to equilibrium moisture contents of 6.4 and 12.5%, respectively, as calculated from the Chung equation (ASAE, 1995). Drying was conducted in a thin layer (<2 cm), with air supplied by relative humidity and temperature control units (Climate-Lab-AA, Parameter Generation and Control, Inc., Black Mountain, N.C.). After drying, all the rough rice was placed in thin layers in a chamber controlled at 33°C and 67.8% rh, and equilibrated to 12.5% m.c. over a period of approximately two weeks. Subsequently, each lot of rice was placed in a sealed plastic bucket and stored for 20 weeks in one of three storage chambers maintained at 4, 21, or 38°C. The total test consisted of 12 lots (i.e., 2 pre-drying conditions, 2 drying conditions, and 3 storage temperatures).

After 20 wk, one 300-g sample/lot was removed and equilibrated for 6 to 8 h at room temperature in sealed plastic bags. To assure representative samples, all buckets were rotated end-over-end five times prior to the sampling. Each 300-g sample was randomly divided in half. Each half was then dehulled in a McGill laboratory hulling machine (Seedboro Equipment Co., Chicago, Ill.), and the resulting brown rice was milled for 30 s in a McGill No. 2 laboratory mill (Seedboro Equipment Co., Chicago, Ill.) with a 1.5-kg mass positioned on the mill lever arm 15 cm from the centerline of the milling chamber. The head rice was collected via a double tray shaker table, with both trays having 4.76-mm indentations (Seedboro Equipment Co., Chicago, Ill.). Sixty grams of the head rice were ground to flour in a laboratory cyclone mill equipped with a 0.5-mm screen (UDY Co., Fort Collins, Colo.).

Differential Scanning Calorimetry

Gelatinization and retrogradation of rice flour were investigated via a differential scanning calorimeter (Pyris 1, Perkin-Elmer Co. Norwalk, Conn.). Rice flour (3.5 to 4.5 mg randomly pulled from the 60 g of ground head rice) was weighed into an aluminum DSC sample pan, and distilled water was added through a microsyringe to give a water:flour (dry solid) ratio of 2.5:1, after determining the flour moisture content (AOAC, 1990). The sealed sample pans were first heated in the DSC from 20 to 110°C, with a heating rate of 10°C/min, to determine the gelatinization enthalpy and temperature. A pan containing 15 μL of distilled water was used as a reference during scanning, and the DSC instrument was calibrated using indium.

The gelatinized samples were subsequently held at 4°C for seven days (in the sample pans) and then rescanned in the DSC from 20 to 90°C, at 10°C/min, to determine the retrogradation temperature and enthalpy. The temperatures and enthalpies for gelatinization and retrogradation were extracted from the DSC thermograms via Pyris data analysis software (Perkin-Elmer Co., Norfolk, Conn.). The enthalpy (ΔH) required to melt starch crystallites was measured from the area of endothermic peaks in the thermograms and expressed as Joules per gram rice flour on a dry weight basis. Onset temperature (T_o), peak temperature (T_p), and conclusion temperature (T_c) of thermal transitions were determined following the procedure outlined by Russell (1987). Three replicate measurements were conducted for each sample.

Statistical Analysis

The coefficients for orthogonal contrasts (table 1) of the effects of pre-drying, drying, and storage treatments were first computed (Montgomery, 1991). Then half-normal plots of contrasts (Draper and Smith, 1981) were created to determine which should be used as the mean square error (MSE). The regression analysis was performed, via the REG procedure (SAS Institute, 1987), on the contrasts that were not used in the MSE. The backward selection was used to determine which of the remaining contrasts were significant.

Results and Discussion

The gelatinization and retrogradation endotherms (fig. 1) were analyzed for comparison of the enthalpies and characteristic temperatures of transition. The standard deviations among replicates of the enthalpy and temperature measurements were below 0.6 J/g and 0.5°C, respectively, for all of the samples. The gelatinization of Cypress rice flours occurred at temperatures from 73 to 86°C, with a peak temperature of 78 to 79°C (table 2). The enthalpy for gelatinization varied from 7.8 to 9.7 J/g, depending on the postharvest treatment (table 2). These results were consistent with those of Normand and Marshall (1989), who reported gelatinization enthalpies of

Table 1. Coefficients for orthogonal contrasts of the effects

Treatments*	Orthogonal Contrast Coefficients			
PDT	–1 (immediate)	1 (delayed)		
DT	–1 (high)	1 (low)		
ST				
ST1 (linear)	0 (no storage)	1 (4°C)	0 (21°C)	–1 (38°C)
ST2 (quadratic)	0 (no storage)	1 (4°C)	–2 (21°C)	1 (38°C)
ST3 (control)	–3 (no storage)	1 (4°C)	1 (21°C)	1 (38°C)

* Pre-drying treatment (PDT): immediate or delayed drying; Drying treatment (DT): high or low temperature; Storage treatment (ST): no storage or storage at 4, 21, or 38°C for 20 wk.

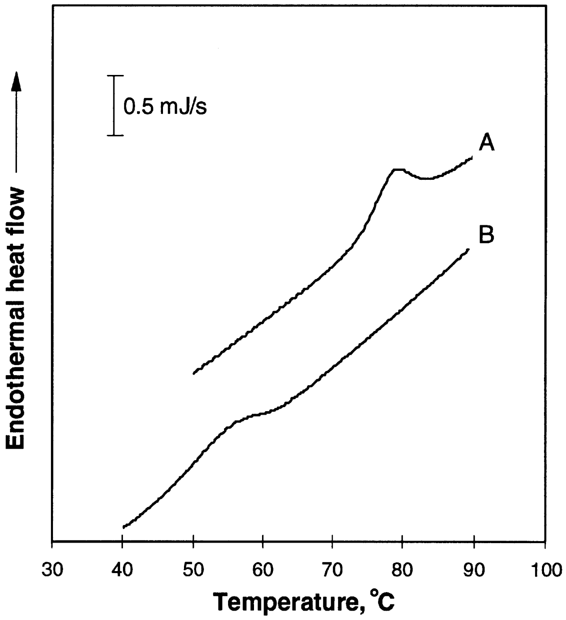

Figure 1–Example DSC thermograms for gelatinization (Curve A) and retrogradation (Curve B) of Cypress rice flour (immediate drying, high drying temperature, no storage). The heating rate was 10°C/min, and the mass of the dry rice flour sample was 3.7 mg.

Table 2. Gelatinization enthalpy (ΔH), onset (T_0), peak (T_p), and conclusion (T_c) temperatures after different drying and storage treatments

Pre-drying Condition	Drying Temp.	Storage Treat-ment*	Gelatinization Enthalpy and Temperatures†			
			ΔH (J/g)	T_o (°C)	T_p (°C)	T_c (°C)
Immediate	High	No storage	8.6	73.7	78.6	84.1
Immediate	High	4°C	7.8	73.5	78.5	85.1
Immediate	High	21°C	9.0	73.2	78.5	84.4
Immediate	High	38°C	9.7	74.0	79.8	86.0
Delayed	High	No storage	8.5	72.8	78.1	84.1
Delayed	High	4°C	8.6	73.0	78.1	83.6
Delayed	High	21°C	8.5	73.1	78.4	84.3
Delayed	High	38°C	9.0	73.9	79.6	85.4
Immediate	Low	No storage	9.0	74.0	79.4	85.4
Immediate	Low	4°C	8.3	73.1	78.4	85.4
Immediate	Low	21°C	8.6	73.3	78.6	84.5
Immediate	Low	38°C	9.3	74.3	79.5	85.3
Delayed	Low	No storage	8.7	73.2	78.1	83.9
Delayed	Low	4°C	8.1	73.4	78.3	85.3
Delayed	Low	21°C	8.3	72.7	78.2	84.9
Delayed	Low	38°C	9.0	74.1	79.6	84.8

* Storage treatment included no storage (zero week) or storage at 4, 21, or 38°C for 20 wk.
† Means of triplicate measurements.

rice flours from several long- and medium-grain varieties (Lemont, Mars, S-201, Calmochi) ranging from 9.5 to 11.2 J/g, with peak temperatures of 66.6 to 75.9°C. For pure rice starches, Russell et al. (1983) reported gelatinization enthalpies of 12.0 to 16.8 J/g, with peak temperatures of 63 to 78°C. Rice flour, as used in this study, contained a certain amount of non-starch components (e.g., protein, fiber, and lipids) and thus shows a lower gelatinization enthalpy than does pure rice starch.

The DSC data from scans of the retrograded samples, kept at 4°C for seven days, were considerably different from those of the first scans, in which the rice underwent gelatinization (fig. 1). The endothermic peaks of retrogradation were at lower temperatures, typically ranging from 46 to 63°C, with a peak temperature of approximately 55°C (table 3). The enthalpy for retrograded rice gels varied from 5.1 to 6.7 J/g, which was about 60 to 70% of the gelatinization enthalpy.

The regression analysis for the gelatinization results (table 4) showed that the gelatinization enthalpy was a function of storage treatment ($p<0.0001$). As storage temperature increased from 4 to 38°C, gelatinization enthalpy also increased. The mean gelatinization enthalpy for the samples stored at 38°C was 9.3 J/g, which was 1.1 J/g higher than that of samples stored at 4°C and 0.7 J/g higher than that of samples stored at 21°C.

The onset temperature of gelatinization was significantly affected by pre-drying treatment ($p<0.05$), and linear ($p<0.0007$) and quadratic ($p<0.005$) terms of storage treatment (table 4). Immediately dried rice showed significantly higher onset temperature than did the delayed dried rice. The rice stored at 21 and 38°C gave a higher onset temperature than did that stored at 4°C. Storage treatment had a significant linear effect on the peak temperature of gelatinization ($p<0.0005$). The regression model indicates that the peak temperature of gelatinization significantly increased as the storage temperature increased. The mean gelatinization peak temperature for rough rice stored at 38°C for 20 wk was 79.6°C, which was 1.1°C higher than that for the unstored control. However, the mean gelatinization peak temperatures for the rice stored at 4 or 21°C were close to that for the control sample. The results (table 4) also indicate

Table 3. Retrogradation enthalpy (ΔH), onset (T_0), peak (T_p), and conclusion (T_c) temperatures after different drying and storage treatments

Pre-drying Condition	Drying Temp.	Storage Treat-ment*	Retrogradation Enthalpy and Temperatures†			
			ΔH (J/g)	T_o (°C)	T_p (°C)	T_c (°C)
Immediate	High	No storage	5.4	46.0	55.2	62.8
Immediate	High	4°C	6.2	46.0	55.1	63.0
Immediate	High	21°C	6.3	45.2	54.8	62.3
Immediate	High	38°C	6.3	46.0	55.9	61.8
Delayed	High	No storage	5.2	47.4	55.4	63.2
Delayed	High	4°C	6.1	46.5	55.6	63.1
Delayed	High	21°C	6.3	46.6	54.9	64.1
Delayed	High	38°C	6.4	40.7	54.7	63.7
Immediate	Low	No storage	5.1	47.1	55.4	63.3
Immediate	Low	4°C	6.2	45.5	55.4	63.7
Immediate	Low	21°C	5.8	46.3	55.6	63.9
Immediate	Low	38°C	5.9	46.3	55.7	63.7
Delayed	Low	No storage	5.5	46.4	55.4	62.9
Delayed	Low	4°C	5.6	46.3	55.3	62.7
Delayed	Low	21°C	6.7	45.4	55.1	62.3
Delayed	Low	38°C	6.6	45.9	55.4	62.4

* Storage treatment included no storage (zero week) or storage at 4, 21, or 38°C for 20 wk.
† Means of triplicate measurements.

Table 4. Significant terms and associated coefficient estimates after performing backward elimination procedure for gelatinization data

Dependent Variables	Model Terms	Parameter Estimates	p-Value
Enthalpy (ΔH)	Intercept	8.6875	0.0001
	ST1	−0.5250	0.0001
Onset temperature (T_o)	Intercept	73.4562	0.0001
	PDT	−0.1813	0.0154
	ST1	0.1958	0.0007
	ST2	−0.4125	0.0029
Peak temperature (T_p)	Intercept	78.7312	0.0001
	ST1	−0.6500	0.0005
Conclusion temperature (T_c)	Intercept	84.7813	0.0001
	DT*ST1	0.4125	0.0834

* PDT and DT are the main effects of pre-drying and drying treatment, respectively; ST1 and ST2 are the linear and quadratic effects of storage treatment, respectively.

that gelatinization conclusion temperature can be a function of the drying × storage interaction ($p<0.10$).

Storage effects on rice quality and cooking properties have been previously reported by several investigators. For example, in a study of three typical North American rice varieties, Chrastil (1990b) observed that age-induced changes in swelling ratio, water uptake, and cooking time were greatly enhanced by higher storage temperature. Because starch is the major component of milled rice, increases in water uptake and cooking time after high temperature storage are probably due to structural and physicochemical changes of the starch. Other investigators (Villareal et al., 1976) have demonstrated that a higher storage temperature also resulted in a higher amylograph peak viscosity. These previous reports are consistent with the observed increase in gelatinization temperature and enthalpy, which are additional measures of starch functionality, after high temperature storage in this study.

Retrogradation properties of rice were also shown to be affected by pre-drying, drying, and storage treatments. There was a significant storage effect ($p<0.0001$) on the retrogradation enthalpy of rice flour (table 5). The mean retrogradation enthalpy for stored samples was 6.2 J/g, which was 0.9 J/g higher than that for the unstored samples. There were no significant differences in mean retrogradation enthalpies across the rice samples with different storage temperatures.

Table 5. Significant terms and associated coefficient estimates after performing backward elimination procedure for retrogradation data

Dependent Variables	Model Terms	Parameter Estimates	p-Value
Enthalpy (ΔH)	Intercept	5.9750	0.0001
	ST3	0.2250	0.0001
Onset temperature (T_o)	Intercept	55.3062	0.0001
	PDT*ST1	0.2375	0.0349
Peak temperature (T_p)			NS†
Conclusion temperature (T_c)	Intercept	63.0562	0.0001
	PDT*DT	–0.5312	0.0001

* PDT, DT and ST1 are the main effects of pre-drying, drying, and storage treatments, respectively. ST3 is the effect of storage comparing to no storage.

† No significant effects.

The pre-drying storage treatment interaction was found to affect ($p<0.05$) the onset temperature of retrogradation (table 5), but the temperature difference due to this effect was less than 0.5°C. The interaction between pre-drying and drying treatments also affected ($p<0.0001$) retrogradation conclusion temperature. However, for retrogradation peak temperature, none of the included postharvest variables had a significant effect.

Starch gelatinization and retrogradation are important physicochemical properties of rice materials which affect the quality of milled rice and value-added applications of rice flour (Juliano, 1990). Retrogradation, in which gelatinized starch tends to reassociate to form ordered molecular structure, results in the "staling" of cooked rice and starch gels (Perez et al., 1993). Rice cooking times appeared to be directly related to starch gelatinization temperatures (Juliano and Perez, 1981). The results reported herein demonstrated significant effects of some important postharvest operations, including pre-drying, drying, and storage treatments, on gelatinization and retrogradation properties of rice flours. Therefore, postharvest handling conditions should be carefully considered to achieve desirable functional properties of rice materials.

Conclusions

Storage temperature had a significant effect on the gelatinization characteristics of long-grain Cypress rice after 20 weeks of rough rice storage. Pre-drying treatments had no significant effect on the gelatinization enthalpy and peak temperature, but did on the onset temperature. The postharvest treatments also significantly affected the retrogradation enthalpy and temperatures of gelatinized rice flour. Unstored rice had a significantly lower retrogradation enthalpy than did the stored rice, even though their retrogradation temperatures were nearly the same. Pre-drying, drying and/or storage treatments significantly affected the retrogradation onset and conclusion temperature, but did not affect the peak temperature. These results suggest that postharvest operations on rough rice might be manipulated to achieve target end-use functionalities of rice flour.

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