Phase Transitions of Rice Starch and Flour Gels[1]

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ABSTRACT

The phase transition temperatures (glass transition temperature [T_g] and melting temperature [T_m]) of 10-50% (w/w) starch and flour gels from waxy, japonica, and indica varieties of rice were studied by differential scanning calorimetry. Effects of additives and storage conditions were also investigated. The T_g of all samples we examined ranged from −10 to −5°C. The T_m and the enthalpy (ΔH) of ice melting increased with increased moisture contents of the samples. When the concentration of starch used was higher than 40%, two endotherms of ice melting and one exotherm between T_g and T_m were observed. The T_g of starch and flour gels under the same concentration were similar, and there were no significant differences among the varieties. The addition of sucrose or sodium chloride decreased the T_g. T_g of 40% rice starch and flour gels decreased to −12 ~ −15°C and to −13 ~ −19°C with the addition of 20% sucrose and 5% sodium chloride, respectively. T_g of rice starch gels increased progressively during the storage. The extent of the increase was affected by the storage temperature (5> − 5> − 18°C).

Starch granule is a natural polymer and has amorphous, intercrystalline, and crystalline regions (Biliaderis et al 1986). During heating, two phase transitions occur: one is the glass transition (T_g) of the amorphous region and the other is melting (T_m) of the crystalline region (Maurice et al 1985, Biliaderis et al 1986).

The T_g is specific for each compound and depends on the free volume, degree of polymerization, molecular geometry, crystallinity, and average molecular weight of the polymer (Levine and Slade 1988, 1989; Slade and Levine 1989, 1991). Water, as a plasticizer, depresses the T_g of a polymer-water system (Levine and Slade 1988, 1989). Biliaderis et al (1986) reported that, below 30% water content, the T_g of rice starch-water systems decreased with the increase of water content. Similar results have been reported for wheat starch (LeMeste et al 1992). The glass transition behavior of the food plays a key role on the quality and the storage stability of the product. It may affect the texture and the staling rate of bakery goods (Ablett et al 1986, Levine and Slade 1989), the quality and shelf-life of the extrudate (Noel et al 1990). The reduction in the loss of flavor components by evaporation and the deterioration of the food product by oxidation may be controlled with adjustment of the glass transition (Tsourouflis et al 1976, Geji-Hansen and Flink 1977, To and Flink 1978, Noel et al 1990). It may also be applied to depress the browning reaction by reducing the activity of enzyme (Flink et al 1974) and to direct the rate of crystallization (Karel 1985, Levine and Slade 1986, Roos and Karel 1991).

Many studies have been reported on the T_g of starch-water systems by using differential scanning calorimetry (DSC) (Nakazawa et al 1984; Maurice et al 1985; Biliaderis et al 1986; Yost and Hoseney 1986; Slade and Levine 1988, 1989; Liu and Lelievre 1991, 1992). However, the technique has seldom been applied to the investigation of the T_g of the rice product. This study was initiated to examine the T_g of the starch and flour gels prepared from the different varieties of rice. The effects of the water content, additives, and storage on the T_g were observed. The result might be valuable for the preparation of rice and other cereal products.

MATERIALS AND METHODS

Rice, Rice Flours, and Rice Starches

The varieties of rice used were TNuS19 (indica rice), TNu70 (japonica rice), and TCW70 (waxy rice). All polished rices were the first season's crop of 1991 and were obtained from Taichung District Agricultural Improvement Station, Chunghua, Taiwan. The polished rice was ground into the rice flour with a two-stage procedure: 1) a cyclone sample mill (Udy Corp., Boulder, CO) to pass a 65-mesh sieve; 2) a laboratory centrifugal mill (Pulverisette 2, Fritsch GmbH Laborgeraetebau Industriestr, Germany) to pass a 100-mesh sieve. Rice starch was isolated and purified according to the method described by Yang et al (1984). The isolated starch was dried at 40°C and ground by the laboratory centrifugal mill to pass a 100-mesh sieve. All samples were stored at 5°C.

Chemical Analysis

The moisture, crude protein, crude lipid, and ash of the rice flour and starch were determined using AOAC methods (1984). The conversion factor of 5.95 was applied to convert nitrogen to crude protein contents. The amylose content of the rice flour was estimated by the colorimetric method of Juliano et al (1981) with slight modifications (Lii et al 1986). A mixture of amylose from potato starch and purified amylopectin from waxy rice (TCW46) solution was prepared as the reference.

Thermal Analysis

To study the gelatinization of the rice flour and starch, 40% (w/w) sample suspension was prepared by hand-mixing powdered sample with deionized water to a homogeneous slurry. The slurry (100-130 mg) was hermetically sealed in a stainless steel DSC crucible (Setaram 12732) with a stainless steel stopper (12735) and an aluminum O-ring (12733). The sample pan was heated from 20 to 140°C in a Setaram 121 DSC (Caluire Cedex, France) at a heating rate of 5°C/min. An empty pan was used as the reference.

For the determination of T_g, 5-10 mg (10-50%, w/w) of the rice flour or starch slurry was sealed into a coated aluminum pan (Du Pont, Wilmington, DE). The sample was heated in a calorimeter (DuPont 910 DSC) from room temperature to 120 ~ 150°C at the rate of 15°C/min, then cooled to −40°C immediately by using a mechanical cooling accessory (model 990476-905, Du Pont). The T_g was determined by reheating the gelatinized sample from −40 to 30°C at the rate of 5°C/min.

Sucrose (20%) or sodium chloride (5%) solutions were used to make the 40% (w/w) sample suspension for investigating the effect of the additive. The effect of sucrose, sodium chloride, and soybean oil combined together was observed by premixing

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the additives at the described ratio with the starch, and then examining the sample as described above.

For the storage study, the sample was prepared by heating the 40% (w/w) rice starch slurry in the glass jar (2.5 cm × 6 cm) at 100°C for 30 min. The gelatinized sample was cooled at room temperature for 30 min and then stored at −18, −5, and 5°C for 10 and 20 days, respectively. A portion (5–10 mg) of the gel was used for the measurement of T_g. The T_g was also examined from −40 to 30°C at a rate of 5°C/min.

RESULTS AND DISCUSSION

Chemical Compositions

The chemical compositions of the rice flour and starch are listed in Table I. The major difference among the three varieties of rice was the amylose content. The indica rice (TNuS19) contained the highest amount of amylose and the waxy rice (TCW70) the least. The low contents of crude protein (≤0.60%) and crude lipid (≤0.09%) of the isolated starch attested to the purity of the sample.

Gelatinization Temperatures and Thermal Properties

Among the three varieties measured, the waxy rice (TCW70) flour and starch showed higher gelatinization temperatures and enthalpies than those of other two varieties (Table II). The result were similar to those reported by Nakazawa et al (1984) with rice and those reported by Krueger et al (1987) with corn starches. Because waxy starch is almost entirely composed of amylopectin, the crystallinity of the starch granule is considered higher than that of a nonwaxy one (Slade and Levine 1989). Consequently, the thermal energy required for the gelatinization of the waxy starch is higher than that of the nonwaxy starch (Nakazawa et al 1984, Slade and Levine 1989).

The onset temperature (T_o) of the first endotherm during the gelatinization for the rice flour was higher than that of the corresponding rice starch (Table II). But the enthalpy of the endotherm for the flour was lower than that of the starch. This might be attributable to the existence of the higher amounts of protein and lipid in the rice flour that could inhibit the gelatinization of starch and reduce the amount of starch in the sample (Biliaderis et al 1985, Marshall et al 1990).

The second endotherm, corresponding to the dissociation of the amylose-lipid complex, was only detected in indica (TNuS19) and japonica (TNu70) rice starches, not with the waxy variety.

TABLE I
Chemical Compositions of Rice Starches and Flours

	Starches, %		Flours, %			
Rice Variety	Crude Protein	Crude Lipid	Crude Protein	Crude Lipid	Crude Ash	Amylose Content
TNuS19	0.60	0.09	8.02	0.39	0.41	24.17
TNu70	0.34	0.07	9.97	0.34	0.43	13.86
TCW70	0.14	0.08	6.33	0.60	0.53	0.25

The temperatures of the endotherms were 92–122°C, which is similar to other reports (Kugimiya et al 1980, Biliaderis et al 1985, Marshall et al 1990).

Glass Transition Temperature and Thermal Property

Gelatinized starch, instead of native starch, was used in the study of T_g because the T_g of native starch is not easily detected by DSC. Due to the low amount of free water inside the starch granule, there is little plasticizing effect, and the complex structure of the starch granule, in which the crystalline and amorphous regions are neither independent of each other nor homogeneous, leads to the change of heat capacity (ΔCp) because the glass transition is too low to be detected (Zeleznak and Hoseney 1987; Slade and Levine 1988; Liu and Lelievre 1991, 1992; Biliaderis 1992; LeMeste et al 1992). However, the T_g can be found from the DSC data by using the first derivative curve of heat flow to time (dCp/dt) (Fig. 1). Similar results were observed with wheat starch by Slade and Levine (1988, 1989) and Liu and Lelievre (1991, 1992).

Effects of Water Contents

The DSC thermograms obtained upon heating the gelatinized rice flour and starch gels are shown in Figures 1 and 2. The T_g of the starch and the flour gels ranged from −5 to −10°C, when the water contents were more than 50% (Tables III and IV). In general, an increase in the water content increased the T_g slightly. At lower water contents (<50%), the sample would not gelatinize completely, and the plasticizing effect of water would be less during the heating process. Consequently, the baseline shift was too small to be detected, and the T_g could not be located. Similar results were found on corn, wheat, and rice starches by

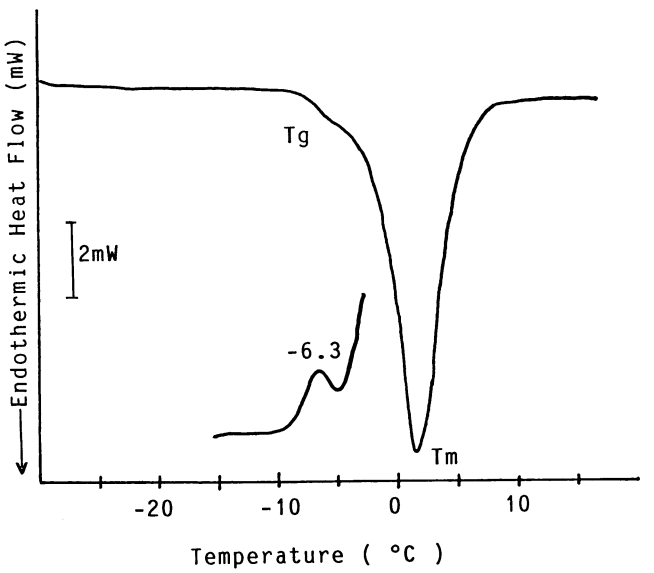

Fig. 1. Differential scanning calorimetry thermogram of glass transition (T_g) for TCW70 rice flour gel (40%). T_m = melting temperature.

TABLE II
Differential Scanning Calorimetry Characteristics of Rice Starches and Flours[a]

	Primary Endotherm				Secondary Endotherm			
Rice Variety	T_o °C	T_p °C	T_c °C	ΔH J/g	T_o °C	T_p °C	T_c °C	ΔH J/g
TNuS19								
Starch	57.31	64.37	81.37	3.69	93.14	102.18	110.44	0.94
Flour	58.66	67.19	83.80	2.64	92.92	103.87	121.89	1.42
TNu70								
Starch	59.08	65.98	84.31	4.37	94.36	105.26	115.65	0.77
Flour	60.86	69.23	83.43	2.77	93.47	103.90	119.00	1.13
TCW70								
Starch	62.46	72.29	90.07	6.07	nd[b]	nd	nd	nd
Flour	64.65	76.05	90.81	3.65	104.01	110.64	120.74	0.32

[a] T_o = onset temperature, T_p = peak temperature, T_c = completion temperature, ΔH = enthalpy.
[b] Not detectable.

Zeleznak and Hoseney (1987) and Liu and Lelievre (1991, 1992). Incidentally, the reproducibility of the measurement was found to be in the range of 1–3°C. Such variation may result from the complexity of the starch granule causing an uneven distribution of water during gelatinization. Hence, the different plasticizing effects occurred among samples (Slade and Levine 1989). The rate of cooling during the measurement could also cause the variation. Lower T_g would be observed with higher cooling rates (Simatos et al 1975).

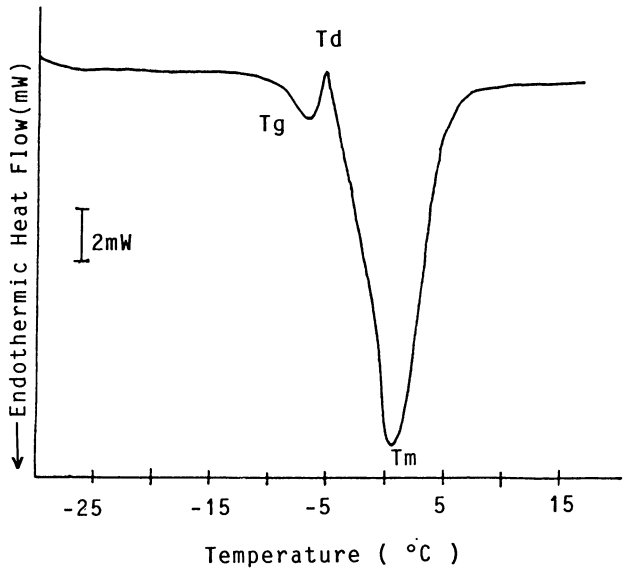

Fig. 2. Differential scanning calorimetry thermogram of glass transition (T_g) for TCW70 rice starch gel (40%). T_m = melting temperature. T_d = exothermic peak.

Previous reports (Maurice et al 1985; Zeleznak and Hoseney 1987; Slade and Levine 1988, 1989; LeMeste et al 1992) suggested that the increase of water content led to an increase in free volume and a decrease in local viscosity. The segmental mobility of the molecular chain in the amorphous region would then rise, resulting in a decrease of T_g. The influence of the water content below 10% on T_g would be more profound. The T_g decreased about 5–10°C with each increase in weight% of water (Slade and Levine 1989). However, under high levels, the change in water content did not affect the T_g drastically (Tables III and IV). This might be explained by the plasticizing effect of water reaching a plateau when the water content was higher than 50%.

The exothermic peak (T_d) following the glass transition was attributed to the crystallization of unfrozen water (Fig. 2). During the rapid decrease in temperature, the water did not freeze and was maintained in a glassy state during the cooling treatment on the gelatinized sample. When the sample was reheated from −40°C for the T_g determination, the temperature went over the T_g, and the devitrification occurred. The exothermic peak (T_d) was then detected when the devitrified water crystallized (Simatos et al 1975, Liu and Lelievre 1992). The occurrence and the intensity of the peak became greater as the water content decreased. At higher water content (>50%), the exothermic peak was not detectable. The size of the T_d peak for starch was larger than that for flour under the same water content. Such results might mean that the water absorption of flour was higher than that of starch, which caused less glassy water.

The melting peak (T_m) of ice for the flour is shown in Figure 3. The T_m and ΔH of ice decreased as the solid content of gel increased from 10 to 50% (Tables III, IV). The amount of freezable water was reduced with an increase in the solid content of the gel. This caused the depression of both T_m and ΔH (Liu and Lelievre 1992).

TABLE III
Effects of Water Contents on the Differential Scanning Calorimetry Characteristics of Rice Starch Gels[a]

Rice Variety	Starch, %	T_g °C	T_m °C	ΔH J/g
TNuS19	10	−6.43	1.21	284
	25	−6.87	0.54	211
	40	−8.01	0.14	147
	50	−9.55	−0.74	97
TNu70	10	−6.21	2.08	267
	25	−6.44	1.12	196
	40	−8.65	0.25	120
	50	−9.49	−0.51	79
TCW70	10	−6.57	1.08	219
	25	−6.85	0.83	202
	40	−8.41	−0.19	137
	50	−8.31	−1.26	66

[a] T_g = glass transition temperature, T_m = melting temperature, ΔH = enthalpy.

TABLE IV
Effects of Water Contents on the Differential Scanning Calorimetry Characteristics of Rice Flour Gels[a]

Rice Variety	Flour, %	T_g °C	T_m °C	ΔH J/g
TNuS19	10	−5.49	1.85	246
	25	−6.43	0.77	180
	40	−7.00	−0.18	101
	50	−7.89	−0.14	78
TNu70	10	−6.02	1.37	213
	25	−7.44	0.03	172
	40	−7.98	−0.25	118
	50	−7.98	0.07	89
TCW70	10	−6.09	1.97	254
	25	−6.79	1.36	195
	40	−6.31	1.03	184
	50	−8.32	0.13	49

[a] T_g = glass transition temperature, T_m = melting temperature, ΔH = enthalpy.

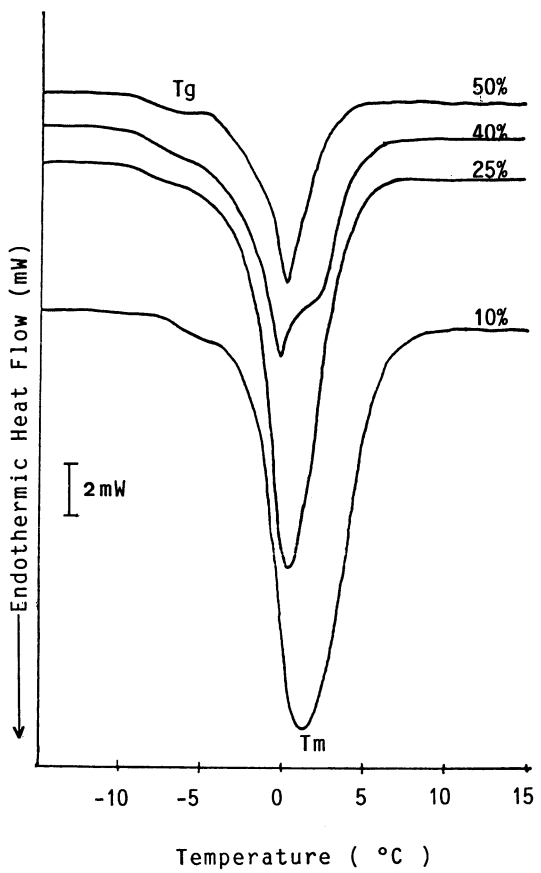

Fig. 3. Differential scanning calorimetry thermogram of glass transition (T_g) for TNuS19 rice flour gels with different water contents. Percentages designate weight fraction of rice flour.

Effects of Varieties

The influence of the water content on T_g, T_m, and ΔH of the rice flour and the starch gels were similar (Tables III and IV). No significant differences were observed among the three varieties of rice examined. This implied that the starch component also played the key role in the glass transition of the flour gel. The result was different from the report of LeMeste et al (1992) on glass transition of bread that was controlled by the gluten constituent.

Effect of Additives

The T_g of rice starch and flour gels (40%, w/w) decreased with the addition of sucrose or sodium chloride (Tables V and VI). The depression effect on T_g could be explained by free volume theory. The capability of a diluent to depress T_g is inversely proportional to its molecular weight (Slade and Levine 1989). The additive also showed a lowering effect on T_m and ΔH (Tables V and VI).

In a native starch-water system, water acts as the plasticizer and the mobility enhancer, which not only increases the free volume of the system but also affects both the T_g and T_m of the partially crystalline polymer (Levine and Slade 1989). The presence of solute with low molecular weight, such as sugar and polyols, changes the system into starch-solute-water system. The solute-water solution, which can be treated as a cosolvent, has higher average molecular weight but lower molecular mobility and free volume than that of pure water. Hence, the cosolvent shows less plasticizing effect than does water alone (Slade and Levine 1989). Furthermore, sugar-solute may form a sugar bridge with starch that can stabilize the amorphous region of starch and restrict the flexibility of molecular chain (Spies and Hoseney 1982). It also causes the rise of T_g in the system (Ghiasi et al 1983, Slade and Levine 1989, Kim and Walker 1992). However, in our study, the sample was fully gelatinized, and the addition of sucrose and sodium chloride decreased the T_g. This might be explained by the high water-binding capabilities of sucrose and sodium chloride that could increase the amount of bound (unfreezable) water (Spies and Hoseney 1982). Consequently, the amount of water that acted as a plasticizer was increased (Biliaderis 1990). Also, the dissolution of sucrose in water might increase the volume of solvent (Ghiasi et al 1983, Kim and Walker 1992), which would increase the free volume. Therefore, the T_g decreased.

TABLE V
Effects of 20% Sucrose Solution on the Differential Scanning Calorimetry Characteristics of 40% (w/w) Rice Starch and Rice Flour Gels[a]

Rice Variety	Gel	T_g °C	T_m °C	ΔH J/g
TNuS19	Starch	−13.57	−2.08	79
	Flour	−12.03	−2.03	72
TNu70	Starch	−14.40	−1.77	84
	Flour	−13.15	−1.88	80
TCW70	Starch	−14.35	−1.24	87
	Flour	−13.48	−1.44	84

[a] T_g = glass transition temperature, T_m = melting temperature, ΔH = enthalpy.

TABLE VI
Effects of 5% NaCl Solution on the Differential Scanning Calorimetry Characteristics of 40% (w/w) Rice Starch and Rice Flour Gels[a]

Rice Variety	Gel	T_g °C	T_m °C	ΔH J/g
TNuS19	Starch	−13.12	−0.95	132
	Flour	−15.86	−4.08	82
TNu70	Starch	−18.17	−2.79	140
	Flour	−17.23	−3.49	109
TCW70	Starch	−18.43	−3.27	122
	Flour	−16.56	−3.52	105

[a] T_g = glass transition temperature, T_m = melting temperature, ΔH = enthalpy.

The endotherm of the ice melting often split into two peaks (Fig. 4). This might be because of condensate water on the lid of the sample pan during the examination, which could melt at the different temperature (Liu and Lelievre 1992). This peak was reduced with the addition of sucrose and sodium chloride. The high water-binding capacity of the additive limited the amount of water vaporized, and thereby the condensate on the lid of the pan. Hence, the second endothermic peak became smaller.

The combined effect of the additives (sucrose, sodium chloride, and oil) on the T_g was similar to that of the one-additive system (Table VII).

Effect of Storage Conditions

Rice starch gels (40%, w/w) from TCW70 and TNuS19 varieties were used to study the storage effect on T_g (Table VIII). Storage temperatures were 5, −5, and −18°C. The ΔT at zero day of storage (i.e., the difference between storage and temperature and T_g) were about 13, 3, and −10°C for TCW70 and they were 10, 0, −13°C for TNuS19. Table VIII showed that the T_g of starch gels increased with the storage time. The degree of influence on T_g was proportional to ΔT. The ΔT of the sample stored at 5°C was the highest. It was also close to $(T_g + T_m)/2$, the temperature of the fastest rate of crystallization. T_m here is designated for the melting of crystallite (Levine and Slade 1988, Morris 1990, Shukla 1991). So it was in an unsteady, rubbery state that caused a greater extent of reassociation and retrogradation. Therefore, the higher crystallinity would form and the T_g would increase (Zeleznak and Hoseney 1987, Levine and Slade 1989, Roos and Karel 1991). Furthermore, the syneresis caused by starch retrogradation might decrease the amount of effective plasticizing water and increase the T_g (Biliaderis 1992).

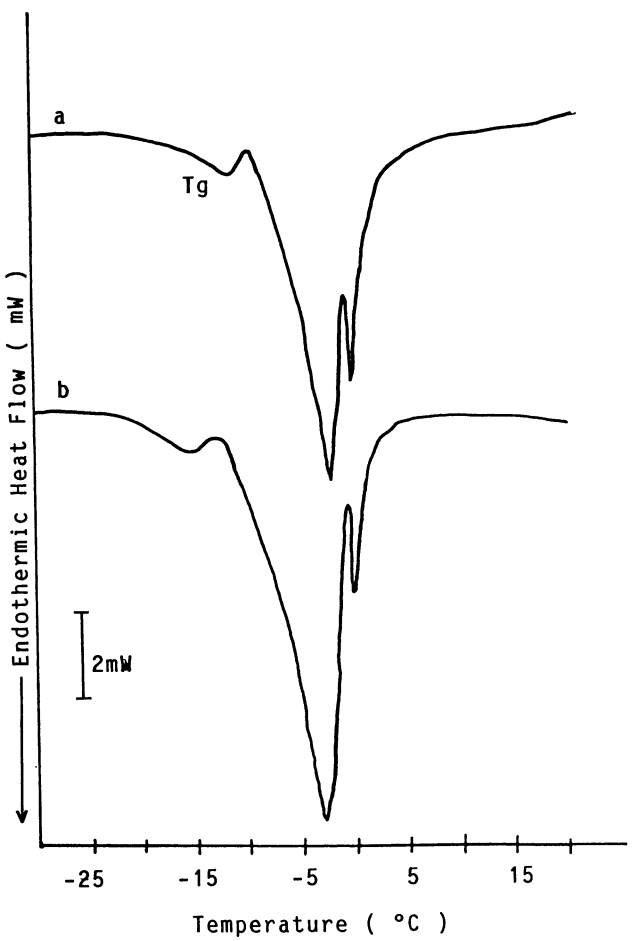

Fig. 4. Effects of sucrose and sodium chloride on differential scanning calorimetry thermograms of rice starch gels. a, TNu70 rice starch with sucrose. b, TCW70 rice starch with sodium chloride. T_m = melting temperature.

TABLE VII
Effects of Additives on the Differential Scanning Calorimetry Characteristics of 40% (w/w) Rice Starch Gels[a]

Rice Starch	Starch-Sucrose-NaCl-Oil-Water Ratio	T_g °C	T_m °C	ΔH J/g
TNuS19	1 : 1 : 0.02 : 0.15 : 2	−28.08	−5.04	81
TNu70	1 : 1 : 0.02 : 0.15 : 2	−27.28	−5.15	79
TCW70	1 : 1 : 0.02 : 0.15 : 2	−25.90	−4.59	89
TNuS19	1 : 0.1 : 0.02 : 0.15 : 2	−15.13	−1.46	115
TNu70	1 : 0.1 : 0.02 : 0.15 : 2	−15.81	−1.49	131
TCW70	1 : 0.1 : 0.02 : 0.15 : 2	−15.28	−1.03	129
TNuS19	1 : 0.6 : 0 : 0 : 0.9	−21.57	−7.09	35
TCW70	1 : 0.9 : 0 : 0 : 0.7	−35.16	−13.85	33

[a] T_g = glass transition temperature, T_m = melting temperature, ΔH = enthalpy.

TABLE VIII
Effects of Storage Conditions on the Glass Transition Temperature (T_g) of 40% (w/w) Rice Starch Gels

	TCW70			TNuS19		
Temp. °C	0 Days	10 Days	20 Days	0 Days	10 Days	20 Days
−18	−7.56	−8.08	−6.48	−5.30	−4.82	−3.37
−5	−7.56	−3.53	−4.62	−5.30	−3.67	−3.19
5	−7.56	−4.31	−3.88	−5.30	−3.95	−3.13

CONCLUSION

T_g is one of the major factors for the quality and the storage stability of the rice product. The formulation and the processing condition may influence the T_g of the rice food. The T_g of rice was controlled by the starch component. The amount of water and the different varieties of rice did not show significant effects on T_g under the moisture contents studied. Nevertheless, additives could affect the T_g. Further studies are required to understand how to formulate rice product to control the T_g.

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