Effect of Sucrose on Glass Transition, Gelatinization, and Retrogradation of Wheat Starch

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ABSTRACT

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Differential scanning calorimetry (DSC) was used to study the effect of sucrose on wheat starch glass transition, gelatinization, and retrogradation. As the ratio of sucrose to starch increased from 0.25:1 to 1:1, the glass transition temperature (T_g, T_g') and ice melting enthalpy (ΔH_{ice}) of wheat starch-sucrose mixtures (with total moistures of 40–60%) were decreased to a range of –7 to –20°C and increased to a range of 29.4 to 413.4 J/g of starch, respectively, in comparison with wheat starch with no sucrose. The T_g' of the wheat starch-sucrose mixtures was sensitive to the amount of added sucrose, and detection was possible only under conditions of excess total moisture of >40%. The peak temperature (T_m) and enthalpy value (ΔH_G) for gelatinization of starch-sucrose systems within the total moisture range of 40–60% were increased with increasing sucrose and were greater at lower total moisture levels. The T_g' of the starch-sucrose system increased during storage. In particular, the significant shift in T_g' ranged between 15 and 18°C for a 1:1 starch-sucrose system (total moisture 50%) after one week of storage at various temperatures (4, 32, and 40°C). At 40% total moisture, samples with sucrose stored at 4, 32, and 40°C for four weeks had higher retrogradation enthalpy (ΔH) values than a sample with no sucrose. At 50 and 60% total moisture, there were small increases in ΔH values at storage temperature of 4°C, whereas recrystallization of samples with sucrose stored at 32 and 40°C decreased. The peak temperature (T_p), peak width (δT), and enthalpy (ΔH) for the retrogradation endotherm of wheat starch-sucrose systems (1:0.25, 1:0.5, and 1:1) at the same total moisture and storage temperature showed notable differences with the ratio of added sucrose. In addition, T_p increased at the higher storage temperature, while δT increased at the lower storage temperature. This suggests that the recrystallization of the wheat starch-sucrose system at various storage temperatures can be interpreted in terms of δT and T_p.

The phase transitions associated with ordering and disordering, such as glass transition, gelatinization, and retrogradation in starch systems have been intensively investigated with differential scanning calorimetry (DSC) (Donovan 1979; Zeleznak and Hoseney 1987a,b; Slade and Levine 1988; Kalichevsky et al 1992).

The glass transition temperature (T_g, T_g') in starch decreases with increasing water content due to the plasticization effect of water (Slade and Levine 1987). The T_g of starch systems with water contents >20% drops below room temperature (Zeleznak and Hoseney 1987a) and below 0°C for water contents ≥50% (Slade and Levine 1988). At temperatures below T_g, the material becomes a glassy solid and molecular motion is so slow that crystallization does not occur in a realistic period of time. At temperatures above T_g, however, the material is a rubbery liquid and sufficient motion of the polymer can occur, allowing a retrogradative recrystallization process to occur (Morris 1990).

Sucrose, which is a less effective plasticizer than water, is arguably the most important single sugar for food manufacture (Slade et al 1993). Sucrose increases the gelatinization temperature (T_m) of starches, and the effect increases with increasing sucrose concentration (D'Appolonia 1972; Johnson et al 1990; Chinachoti et al 1991). With the addition of sucrose, the T_g of a sample (or product) changes and, thus, its storage stability also changes (Levine and Slade 1986). Several researchers reported sugar enhanced starch gel retrogradation (Maxwell and Zobel 1978; Chang and Liu 1991). Kohyama and Nishinari (1991), however, reported that sugars prevented retrogradation of sweet potato starch paste and they proposed that sugar molecules interacted with starch molecular chains to stabilize the starch matrix, thus inhibiting retrogradation. The antiplasticizing effect of sugar relative to water has been used to explain the decreased rate of starch retrogradation with increasing T_g of the water-plasticized amorphous regions (Levine and Slade 1988). Thus, there is still controversy over the effect of sugar on starch retrogradation. It has been suggested that the overall rate of starch crystallization is related to the T_g, T_m, and storage temperature (Zeleznak and Hoseney 1987b). Nevertheless, there has been limited study of the effect of sucrose on starch retrogradation as related to the principles of starch crystallization.

The purpose of this study was to use DSC to investigate the effect of sucrose on T_g, T_m, and retrogradation of starch at various storage temperatures, and to interpret starch retrogradation in relation to the addition of sucrose in terms of polymer crystallization principles.

MATERIALS AND METHODS

Materials

Wheat starch was obtained from Sigma-Aldrich (St. Louis, MO) and sucrose was obtained from Junsei Chemical Corp. (Tokyo, Japan). Because of differences in particle size between starch granules and granular sucrose granules, the individual materials were first finely ground to uniform powders using a multicrusher (Samsung Co., Korea) and then mixed together. The wheat starch and sucrose contained ≈10 and 8% moisture, respectively.

Differential Scanning Calorimetry

For DSC investigations, a Perkin-Elmer DSC7 instrument was used. Melting points (mp) and enthalpies for indium (mp 156.6°C, ΔH_m 28.5 J/g) and *n*-dodecane (mp 9.65°C, ΔH_m 218.73 J/g) were used for temperature and heat capacity calibrations. Samples of wheat starch and wheat starch-sucrose mixtures were prepared in aluminum pans and the sample pans and lids were weighed. An empty pan was used as a reference to balance the heat capacity of the sample pan.

Samples of wheat starch and wheat starch-sucrose mixtures (1:0.25, 1:0.5, and 1:1) were prepared. An excess amount of deionized water was added to the sample with a microsyringe, until all of the sample was wetted and uniformly distributed across the bottom of the pan. Pans were left exposed to the atmosphere until they reached the desired moisture level. Pans were then sealed and left to equilibrate for one day at 25°C before DSC analysis. Samples (2–6 mg) containing larger amounts of water were generally heated at a rate of 10°C/min from –30 to 130°C. Pan failure commonly occurred at ≈130°C. After the first heating, the samples were immediately cooled to –30°C and then rescanned over the same temperature range (Zeleznak and Hoseney 1987a). The temper-

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ature of glass transition (T_g') was taken from the second scan of each sample by measuring the midpoint of the baseline shift in heat flow before the ice melting endotherm (Slade and Levine 1988). The standard deviation for T_g' values was generally ±1°C. The enthalpy (ΔH_{ice}) of the ice melting endotherm was determined from the area of the peak from T_g' to the conclusion temperature of the endotherm (T_c) (Hatley et al 1991).

The wheat starch and wheat starch-sucrose gels were aged for one or four weeks at either 4, 32, or 40°C, then reheated from 10 to 100°C at 10°C/min. Pans were reweighed at the end of the experiment to determine whether water had been lost during heating. The endotherm peak areas for gelatinization and retrogradation, representing the energy required to melt the crystalline material, were converted to enthalpy values (ΔH_G and ΔH J/g of starch, respectively). These enthalpy values were used as indices of the initial gelatinization and of the recrystallization that had occurred during aging. Peak temperatures for the gelatinization and retrogradation endotherms (T_m and T_p, respectively) were obtained easily using Perkin-Elmer DSC software. The width of the retrogradation endotherm (δT) was taken as the difference between the baseline points of the peak. Each sample was analyzed in triplicate. Standard deviations were ±0.6 for T_m, ±0.5 for T_p, ±0.2 for δT, and ±3 for ΔH_{ice}, ±0.2 for ΔH_G, and ±0.2 for ΔH.

RESULTS

T_g' and ΔH_{ice} of Starch-Sucrose Mixtures

At water contents <30% (w/w) of total weight, T_g transitions for wheat starch-sucrose samples were not observed by DSC between –20 and 0°C. The T_g (T_g') of wheat starch-sucrose mixtures with >40% water content could be easily observed at temperatures <0°C. The changes in T_g' with sucrose-starch ratio at 50% (w/w) total water content are shown in Fig. 1. There was a progressive decrease in T_g' from –6 to –26°C as the ratio of sucrose to starch increased from 0 to 1.

For total water contents of 40–60% (w/w), T_g' and ΔH_{ice} values for starch-sucrose systems are shown in Fig. 2 and Table I, respectively. With increasing sucrose-starch ratio at the same water content, the T_g' of starch-sucrose gels fell –4 to –21°C lower than that of samples with no sucrose. This range of depressed T_g' values was within the T_g' range (–10 to –43°C) for amylopectin-sucrose (10:1) systems reported by Kalichevsky et al (1993). The difference in ΔH_{ice} values for starch-sucrose samples at the same water content increased from 29.3 to 413.4 J/g as the ratio of sucrose to starch increased (Table I). The plasticizing effect of sucrose on the T_g of wheat starch can be explained by free volume theory (Ferry 1980): the lower molecular weight sugar increases the mobility of starch, thus decreasing the T_g of the amorphous mixture (Levine and Slade 1988).

Effect of Sucrose on T_m and ΔH_G of Wheat Starch

The gelatinization T_m (first transition, G or G + M1 endotherm) (Jang and Pyun 1996) and ΔH_G for wheat starch with various sucrose-starch ratios are shown in Figs. 3 and 4, respectively. In accord with starch crystallization principles (Morris 1990), increasing the sucrose to starch ratio from 0.25 to 1 increased the T_m and ΔH_G by 1.5–16.4°C and by 1.5–7.1 J/g, respectively, compared with the values for a starch sample with no sucrose at the same total water content. At 40 % (w/w) water content, T_m increased by 3.6–16.4°C; at 50% (w/w) water content, T_m increased by 1.5–10.5°C; and at 60% (w/w) water content, T_m increased by <4°C. Accordingly, the increase in T_m of wheat starch due to the presence of sucrose was greater the lower the water content. Thus, the sugar increases the gelatinization temperature of starch, which can be explained on the basis of the reduced plasticizing effectiveness of sugar-water cosolvents containing more sugar and less water (Slade and Levine 1987). The same explanation relating to plasticization and antiplasticization by sugars (Slade and Levine 1995) can be used to interpret the increase in ΔH_{ice} (Table I).

Effect of Sucrose on T_g' of Wheat Starch During Storage

It has been reported that the T_g' of starch-sugar mixtures varied with storage (Wang and Jane 1994) and that the stability of starch-based foods is controlled by the temperature difference (ΔT) between the storage temperature (T) and T_g' of a matrix ($\Delta T = T - T_g'$ (Levine and Slade 1986). The higher the T_g' of mixtures, the greater the storage stability of frozen foods.

TABLE I
Ice Melting Enthalpy on Rescanning of Wheat Starch-Sucrose Gels[a]

	g of Sucrose/g of Starch			
Total Moisture (%)	**0**	**0.25**	**0.5**	**1**
40	74.7	104.0	126.2	136.4
50	166.2	209.4	255.5	306.9
60	203.1	408.5	480.7	616.5

[a] After cooling to freezing, J/g of starch.

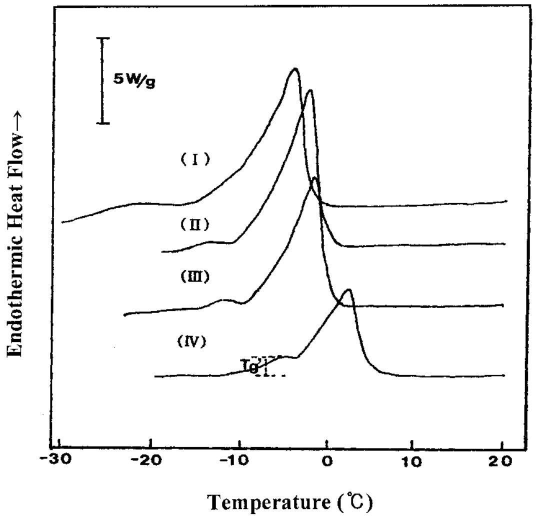

Fig. 1. Typical differential scanning calorimetry thermograms for wheat starch-sucrose gel systems with 50% total water content. Sucrose-starch ratios: (I) 1:1, (II) 0.5:1, (III) 0.25:1, and (IV) 0:1. T_g' = glass transition temperature.

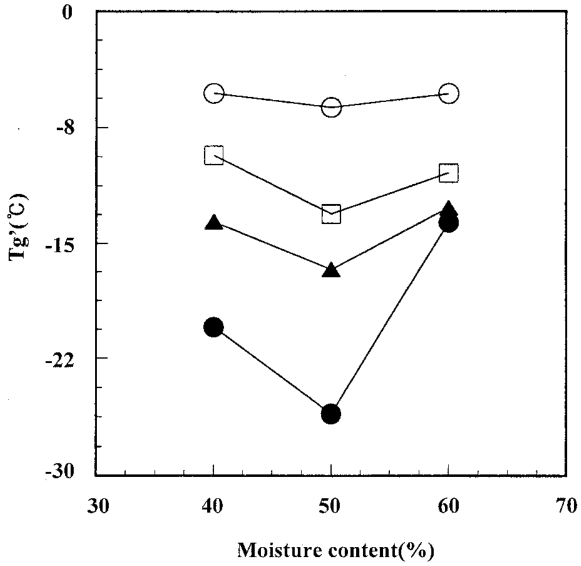

Fig. 2. Effect of sucrose on glass transition temperature (T_g') for wheat starch-sucrose gel systems with total water contents of 40–60%. Sucrose-starch ratios: 1:1 (●), 0.5:1 (▲), 0.25:1 (□), and 0:1 (○).

The changes in T_g' for wheat starch-sucrose systems with total water content of 40, 50, and 60% (w/w) were measured during storage for one or four weeks at 4, 32, or 40°C. The typical changes for the samples with 50% total water content are shown in Fig. 5. For sucrose-starch ratios of 0.25:1 and 0.5:1, the T_g' of the samples stored for one week at 4°C varied by 0.6 to 5°C. However, no significant changes in T_g' were found at storage temperatures of 32 and 40°C. For 1:1 sucrose-starch systems, T_g' of the samples at all storage temperatures greatly increased by 15–18°C. The T_g' for the wheat starch-sucrose systems with 40 and 60% (w/w) total water content stored for four weeks only increased by a maximum of 3°C for storage temperatures of 4, 32, or 40°C, even for increasing sucrose-wheat starch ratios. Wang and Jane (1994) reported that T_g' for corn starch containing sucrose varied by 2.6–3.3°C during storage at 2°C, and that individual components and storage conditions might have an effect on T_g' of such a mixture with sugar.

Effect of Sucrose on Wheat Starch Retrogradation (Recrystallization)

The extent of retrogradation of starch in the presence of sucrose is sometimes expressed as a retrogradation percentage (Wang and Jane 1994; Ward et al 1994), which is defined as the ratio of retrogradation enthalpy to gelatinization enthalpy. This approach applies best to samples containing >60% total moisture. When our samples of wheat starch-sucrose at 1:0 and 1:0.25 with 50% water contents were stored for four weeks at 4°C, the gelatinization enthalpies were 4.5 and 5.4 J/g, while the retrogradation enthalpies were 7.3 and 6.8 J/g, respectively. Thus, the calculated retrogradation percentages were >100% because the retrogradation enthalpies were higher than the gelatinization enthalpies. Therefore, the extent of retrogradation for wheat starch-sucrose gels with 40–60% water content was simply expressed in terms of recrystallization enthalpy (ΔH). The results for storage at 4, 32, or 40°C for one or four weeks are shown in Fig. 6. For a storage temperature of 4°C, ΔH values of samples after one week of storage were higher than ΔH values for a sample with no sucrose. The ΔH values of starch-sucrose samples with 40% total water content increased continuously over four weeks but the increase occurred within one week for samples with 50 and 60% total water contents, except for the no-sucrose sample. For storage temperatures of 32 or 40°C, the recrystallization of starch-sucrose samples with total water contents of 40% increased to a greater extent than the recrystallization for a sample with no sucrose as the sucrose-starch ratio increased. However, the recrystallization of starch-sucrose samples with total water contents of 50 and 60% decreased more than that of a sample with no sucrose and the 1:1 starch-sucrose system did not show recrystallization in four weeks.

DISCUSSION

According to one view, sucrose increases the recrystallization of starch. Maxwell and Zobel (1978) and Chang and Liu (1991) reported that the rate of retrogradation in wheat and corn starch gels was increased by sucrose, glucose, fructose, and maltose, but

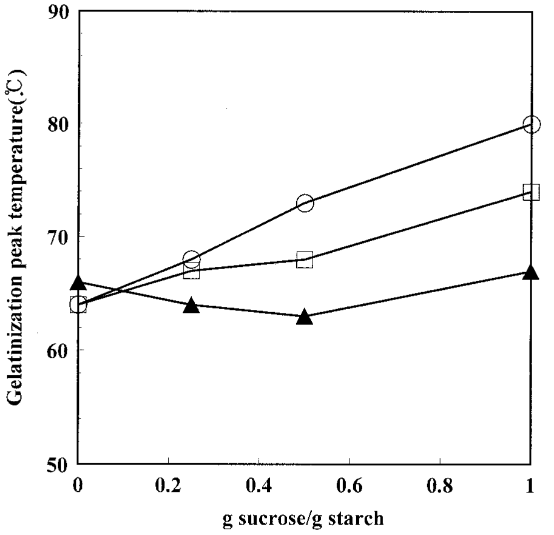

Fig. 3. Effect of sucrose on gelatinization peak temperature for wheat starch at total water contents of 40 (○), 50 (□), and 60% (▲).

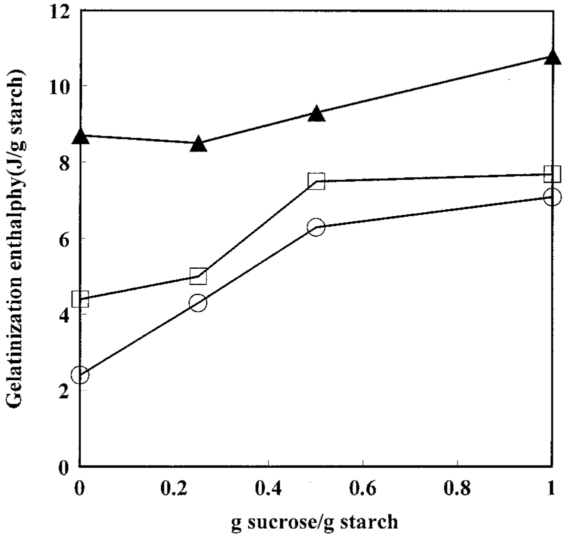

Fig. 4. Effect of sucrose on gelatinization enthalpy for wheat starch at total water contents of 40 (○), 50 (□), and 60% (▲).

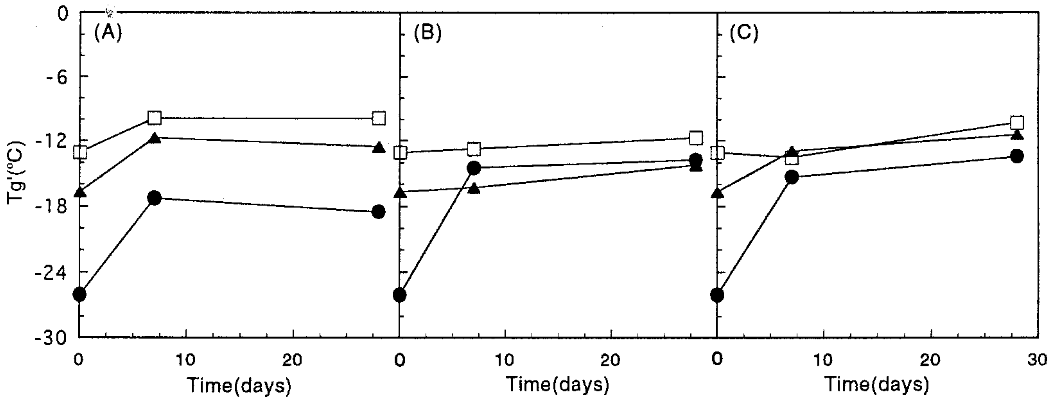

Fig. 5. Changes in glass transition temperature (T_g') for wheat starch-sucrose gels with 50% total water content during storage at 4 (A), 32 (B), and 40°C (C). Sucrose-wheat starch ratios: 1:1 (●), 0.5:1 (▲), and 0.25:1 (□).

gel rigidity was reduced by glucose and sucrose. Ward et al (1994) reported that corn starch or wheat starch with glucose or fructose showed greater retrogradation than did the starch with no sugar after storage at 23°C. According to another view, sucrose inhibits the recrystallization of starch. Levine and Slade (1987) suggested that sugar-water has an antiplasticizing effect relative to water alone, thus increasing the T_g of the water-plasticized amorphous amylopectin regions, which would explain the decreased rate of starch retrogradation for a 1:1:1 starch-sugar-water system, in comparison to 1:1 starch-water systems. Kalichevsky et al (1993) suggested that sucrose prevents the retrogradation of amylopectin by reducing viscosity and structurally changing the water-sucrose phase of the starch-sucrose-water system.

These different views may have resulted from differences in the proportion of starch to sucrose and in storage conditions such as temperature (Wang and Jane 1994). The relationship between storage temperature and the size of the endotherm can be interpreted in terms of principles of polymer crystallization (Slade and Levine 1987). The overall rate of crystallization depends on the rates of nucleation and of crystal growth, both of which are affected by temperature (Slade and Levine 1987; Zeleznak and Hoseney 1987b). It is difficult to differentiate the nucleation rate from the growth rate within the starch retrogradation endotherm. In a previous study (Jang and Pyun 1997), the nucleation rate and propagation rate for starch recrystallization were represented in terms of the enthalpy and peak temperature of starch retrogradation endotherms. This representation showed a similarity with the net rate of recrystallization. The peak temperatures of endotherms increase, whereas the peak width and enthalpy values decrease with increasing storage temperature due to the formation of a more perfect crystal structure (Slade and Levine 1987; Zeleznak and Hoseney 1987b).

In the present study, the T_p, δT, and ΔH of the retrogradation endotherms differed markedly with sucrose-starch ratios from 0.25 to 1:1, even for the same storage temperature and total moisture. Thus, to evaluate the effect of sucrose on the retrogradation of starch, ΔH, δT, and T_p of the retrogradation endotherms for samples stored for four weeks at various temperatures were determined

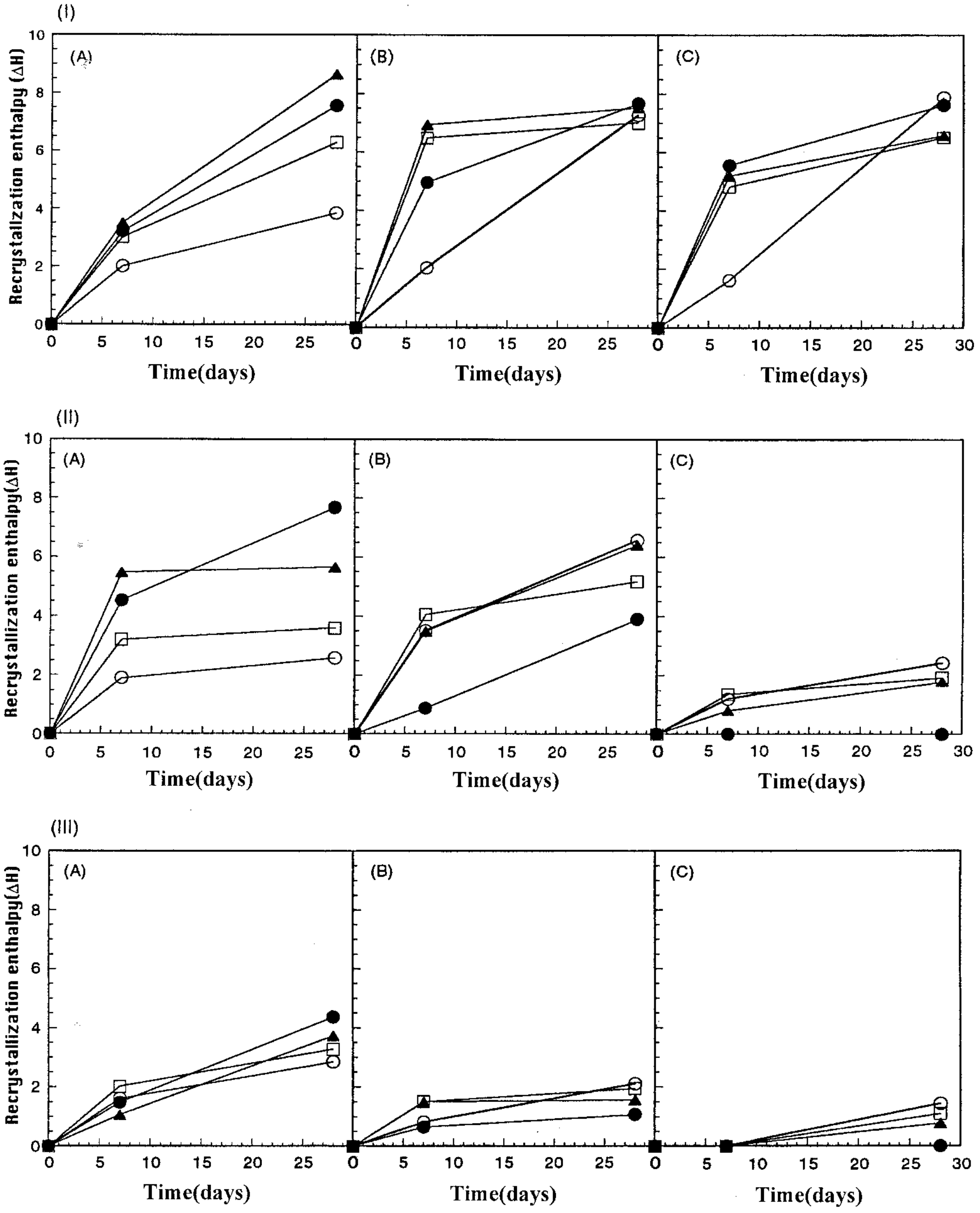

Fig. 6. Effect of sucrose on rate of starch recrystallization in wheat starch-sucrose gels containing various total water contents during storage at various temperatures: (I) 4, (II) 32, and (III) 40°C. Total moisture contents: 40 (A), 50 (B), and 60% (C). Sucrose-wheat starch ratios: 1:1 (●), 0.5:1 (▲), 0.25:1 (□), and 0:1 (○).

(Fig. 7). At 40% total moisture, the sample with sucrose stored at 4°C showed greater δT and lower T_p than for a sample with no sucrose. This may indicate that the former sample had a higher ΔH due to a higher nucleation rate (δT), but its propagation rate (T_p) was lower. Based on this interpretation, it can be construed that the effect of nucleation rate on the recrystallization of starch is greater than the effect of propagation rate. The effect of propagation rate, however, was observed in terms of a smaller decrease in the slope of ΔH than in δT as the storage temperature was increased from 4 to 32°C. At a total moisture of 50%, ΔH was only slightly different for samples with or without sucrose stored at 4°C, but when the storage temperature was 32 or 40°C, the samples with sucrose showed lower δT and ΔH than the samples with no sucrose. At a total moisture of 60%, the samples with sucrose stored at 4, 32, or 40°C showed lower ΔH due to lower δT. Recrystallization in samples containing sucrose at total moistures of 50 or 60% stored at 32 or 40°C may have been inhibited to some extent because T_p was a little higher, whereas δT decreased with increasing ratio of added sucrose. In particular, the 1:1 starch-sucrose system with 60% total moisture stored at 32 or 40°C showed no recrystallization after four weeks of storage (Figs. 6 and 7).

These trends with storage temperature and sucrose addition (versus no sucrose) were likely due to differences in crystallization kinetics (Slade and Levine 1987; Morris 1990) between T_g' and T_m because the T_g and T_m for starch with sucrose were changed (Slade and Levine 1987). The values for increasing sucrose to wheat starch from 0 to 0.25, 0.5, and 1 for 40% total moisture were –5.3, –9.3, –13.6, and –20.6 for T_g' and 64.1, 67.7, 75.0, and 80.5 for T_m, respectively. The values for increasing sucrose to wheat starch from 0 to 0.25, 0.5, and 1 for 50% total moisture were –6.2, –13.1, –16.7, and –26.1 for T_g' and 64.2, 65.7, 68.9, and 74.7°C for T_m, respectively. The values for increasing sucrose to wheat starch from 0 to 0.25, 0.5 and 1 for 60% total moisture were –5.3, –10.4,

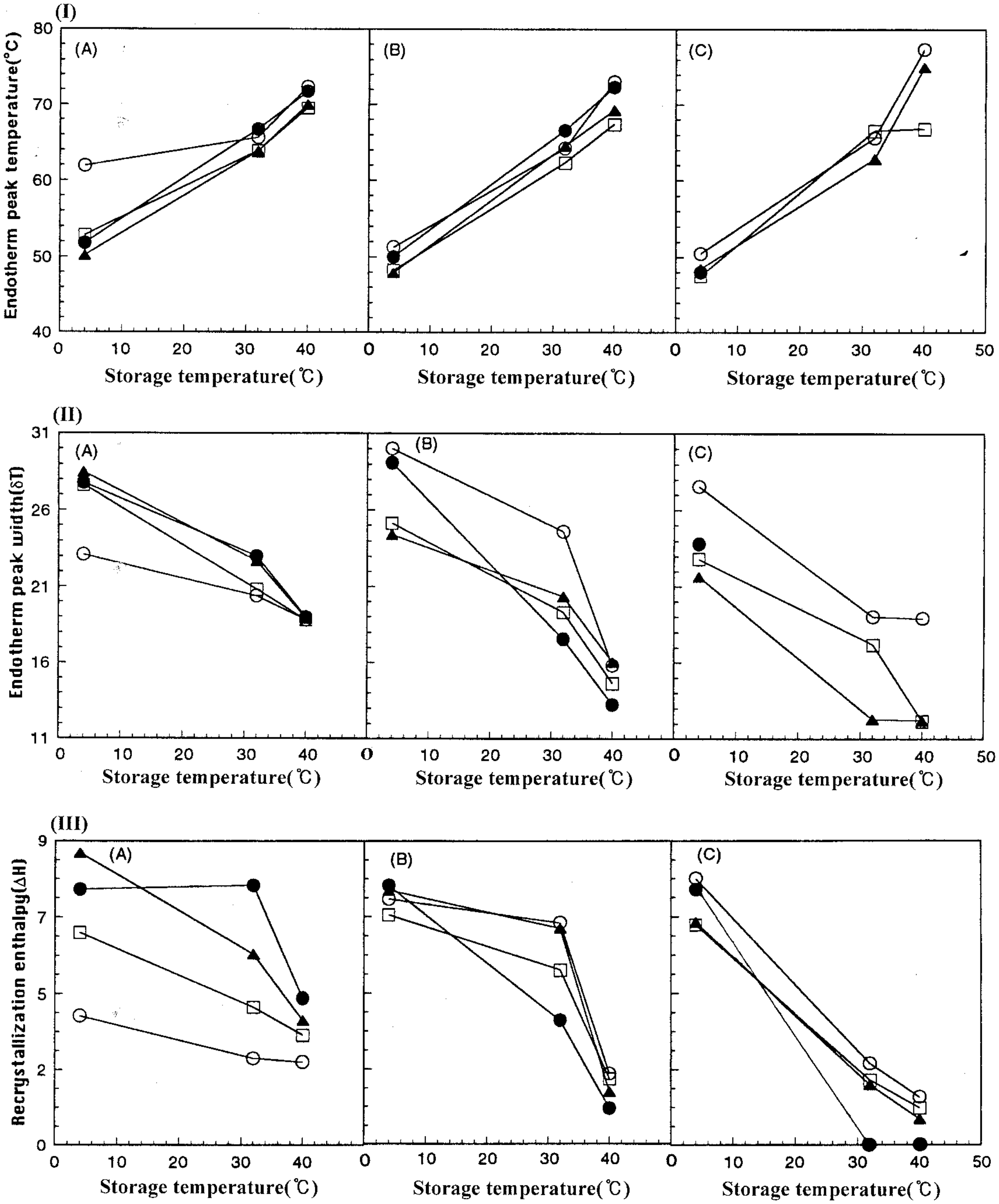

Fig. 7. Effect of sucrose on retrogradation of wheat starch-sucrose gels containing various total water contents stored for four weeks at various temperatures. Total moisture contents: 40 (A), 50 (B), and 60% (C). Sucrose-wheat starch ratios: 1:1 (●), 0.5:1 (▲), 0.25:1 (□), and 0:1 (○).

–12.7, and –13.7 for T_g' and 66.7, 64.4, 66.8, and 68.6 for T_m, respectively. Samples with sucrose showed lower T_g' and higher T_m than samples with no sucrose. Depending on the extent of the T_g decrease and the T_m increase, compared with samples with no sucrose, the crystallization kinetics of starch could have been changed (Fig. 8).

Because the difference in T_g' between samples with and without sucrose was greater than the difference in T_m (for the starch-sucrose systems at total moistures of 50 and 60%), the T_g and T_m of samples containing sucrose became $T_g{}^2$ and $T_m{}^2$ (maximum recrystallization temperature $\approx T^2$). For the 1:0.5 and 1:1 samples at 40% total moisture, the T_g and T_m became $T_g{}^1$ and $T_m{}^1$ (maximum recrystallization temperature $\approx T^1$). Also, the T_g and T_m became $T_g{}^1$ and $T_m{}^2$ (the 1:0.25 sample at 40% total moisture) or $T_g{}^2$ and $T_m{}^1$. Accordingly, samples with sucrose at each storage temperature showed different results than samples with no sucrose for nucleation and propagation rates. Also, when sucrose was added, the change in starch recrystallization with changing T_g' during storage needed to be considered, but we failed to detect this under our experimental conditions because the significant change in T_g' had already occurred during the first week of storage, which was the shortest storage period.

Based on these results, we made several conclusions for the influence of sugar on recrystallization in wheat starch. Based on the polymer crystallization principles that describe the overall rate of recrystallization between T_g and T_m (Slade and Levine 1987; Morris 1990), samples with sucrose with decreased T_g and increased T_m exhibited an altered maximum recrystallization temperature for wheat starch. Accordingly, samples with sucrose showed different extents of recrystallization at each storage temperature than did samples with no sucrose, because the temperature range between T_g and T_m became wider to a varied extent. Therefore, to enhance the storage stability of starch-based, multicomponent food systems containing added sucrose or other sugar, a full understanding of the pattern of recrystallization produced by changes in T_g and T_m with the amount of plasticizer at a given storage temperature is required (Slade and Levine 1987).

CONCLUSIONS

The T_g' was decreased by the addition of sucrose to wheat starch, whereas the T_m and ΔH_G for the gelatinization endotherms were increased (Slade and Levine 1995). When wheat starch with sucrose was stored under the same conditions for four weeks, differences in the T_p, δT, and ΔH for the retrogradation endotherms of wheat starch with sucrose were found. Also, T_p of wheat starch with and without sucrose at various storage temperatures (4, 32, or 40°C) increased at the higher storage temperature, while δT increased at the lower storage temperature. These results also confirmed that recrystallization in the starch-sucrose system can be interpreted in terms of δT and T_p substitution for the nucleation and propagation rates of the overall rate of recrystallization, respectively. Additional studies on the effects of other sugars are needed to further understand the effect of T_g' and T_m on starch recrystallization.

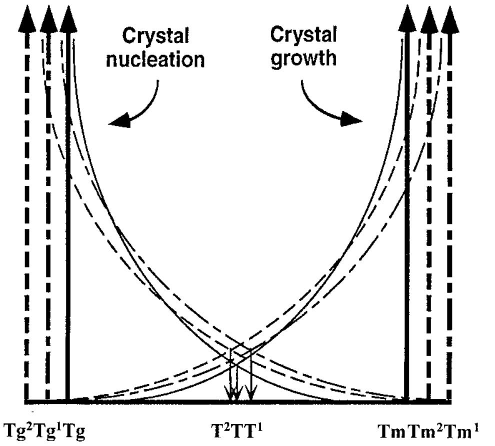

Fig. 8. Crystallization kinetics of wheat starch (extent of glass transition temperature [T_g'] decrease and peak temperature [T_m] increase) with sucrose. Curves (solid, uneven dashed line, dashed line) show dependence of overall rate of crystallization on rates of nucleation and propagation. Maximum recrystallization temperature: $T \approx 1/2(|T_g| + T_m)$, $T^1 \approx 1/2(|T_g{}^1| + T_m{}^1)$, $T^2 \approx 1/2(|T_g{}^2| + T_m{}^2)$. Figure modified from Slade and Levine (1987).

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