

Impact of Melting Conditions of Sucrose on Its Glass Transition Temperature

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The impact of the melting conditions of sucrose crystals on the glass transition temperature (T_g) of the sucrose melt was studied. Final temperature, heating rate, and the residence time at the final temperature were the experimental conditions considered. The glass transition temperature of the different glasses was measured by differential scanning calorimetry, and the degradation of sucrose during the thermal treatments was studied by high-performance liquid chromatography. The results showed that the T_g is sensitive to the degradation of sucrose: T_g decreases with the appearance of small molecules and then increases with the appearance of polymerization products. Thus, the choice of thermal treatment is of the utmost importance for the determination of the T_g of pure sucrose.

Keywords: Sucrose; glass transition; degradation

INTRODUCTION

Over the past few decades, there has been a growing interest in the glassy state in food science and technology. In food products, a glassy state is frequently achieved, for example, in cooked sugar, milk, or coffee powders. The temperature at which this phenomenon occurs has been proposed to explain and predict functional properties and stability during processing and storage of frozen or low-moisture foods (White and Cakebread, 1966; Slade and Levine, 1993; Le Meste and Simatos, 1990; Simatos and Blond, 1991; Nelson and Labuza, 1994).

The glass transition is a universal property of all liquids. It is a kinetic and relaxation event. It occurs at a temperature below which some molecular motions become so slow that a liquid can be considered to take on the properties of a solid. In fact, when liquids are rapidly cooled, molecular motions sharply decrease and crystallization becomes impossible; the liquid is quenched into the glassy state. Glass transition is therefore defined as the intersection between the supercooled liquid state and the glassy state (Zarzycki, 1982; Perez et al., 1990).

Due to the great importance attributed to the storage of fructose-, glucose-, or sucrose-containing foods in the glassy state, there has been an upsurge of interest especially in the study of the calorimetric behavior of carbohydrate monomers and polymers (Luyet and Rasmussen, 1968; Blond, 1989; Orford et al., 1990; Roos and Karel, 1991a; Arvanitoyannis and Blanshard, 1993).

One of the main objectives of scientific researchers is to build a library of state diagrams for different compounds. For this purpose, exact glass transition temperature (T_g) determinations are required; unfortunately, they are generally not available. For example, the T_g values published by different authors vary from

52 to 70 °C (Boneless et al., 1997) for melted sucrose, but Saleki-Gerhardt and Zograf (1994) measure 74 °C for freeze-dried sucrose.

A first explanation can refer to the kinetic character of the glass transition process; the value of T_g depends on the measurement time and on the thermal history of the material. It also depends on which temperature on the DSC curve is chosen as T_g ; the midpoint of the heat capacity jump is usually considered. Simatos et al. (1996) showed that the T_g for sorbitol could vary within a 6 °C range when cooling and heating rates were varied from 1.25 to 20 °C/min.

Roos (1993) noted that differences between reported T_g values for anhydrous common sugar glasses were probably due to residual water. Bonelli et al. (1997) also investigated the impact of residual moisture content of freeze-dried sugar matrices on T_g ; they concluded that T_g was increasingly sensitive to the water content of sugars at low water content, and they claimed that a consequence of this extremely high sensitivity could be the variability in published T_g values. They noted, however, that variations in T_g values could also be due to different thermal histories of samples.

Fan and Angell (1995) studied the relaxation transitions within the fluid state for fructose and galactose melts. They attributed part of the complexity of these molten states to the nonequilibrium state (tautomerization) obtained on fusion. Another part derives from the consequence of the decomposition process that begins around the melting point. The T_g decrease observed might be caused by the many small-molecule fragmentation products.

The thermal degradation of sucrose has been extensively studied. Richards and Shafizadeh (1978) studied the thermal degradation mechanism for sucrose held at 190 and 194 °C for different times; the resulting products were analyzed by gas–liquid chromatography. They concluded that the major reaction is oligomer and polymer formation. Richards (1986) confirmed those results and asserted that an *amorphous* sucrose melt, obtained by rapid water evaporation at the beginning of heating, is decomposed more rapidly than crystalline

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material. The degradation of sucrose during the caramel-making process was analyzed by Defaye and Fernandez (1994, 1995). According to those authors, the chemical composition of the nonaromatic fraction is a mixture of five dianhydrides of D-fructose with α,β -1,2':2,1'-difuranose dianhydride (20% of the total) and branched gluco-oligosaccharides.

Thus, the differences in sucrose T_g values found in the literature could be a consequence, in part, of different thermal treatments of a sucrose melt. To understand this possibility, we compared the T_g of different "sucrose" glasses with their compositions. To identify the potential chemical changes and to optimize melting conditions, we prepared melts with different time-temperature histories. Three procedures for obtaining a sucrose glass have been investigated; they consisted of changes in the final heating temperature, the heating rate, and the residence time at the final temperature.

Two techniques were used for the analysis: differential scanning calorimetry (DSC) for measuring the T_g and high-performance liquid chromatography (HPLC) for characterizing the degradation products.

MATERIALS AND METHODS

Materials. Sucrose was used as obtained from Merck (Darmstadt, Germany). Crystals were melted in DSC pans, according to different thermal treatments. The heated samples were dissolved in distilled water with shaking and then filtered before HPLC analysis.

Methods. *DSC.* DSC measurements were carried out using a Perkin-Elmer DSC-7. The temperature calibration was made using the melting points of indium (156.6 °C) and azobenzol (68 °C). Sealed 20 μ L aluminum pans were used for measurements; an empty pan was used as the reference. Approximately 4 mg of sucrose crystals was accurately weighed in DSC pans and then melted according to the different conditions of time and temperature defined below. After the melting treatments, the samples were quickly cooled (at 40 °C/min) to 100 °C and then cooled slowly (at 10 °C/min) to 25 °C. Finally, the samples were heated at 10 °C/min to 100 °C to measure the T_g . Perkin-Elmer TADS software calculated three temperatures values ($T_{g,onset}$, $T_{g,midpoint}$, and $T_{g,end}$) for the glass transition plus the enthalpy jump (ΔC_p) observed during the transition.

Melting Experiments. The sucrose samples were subjected to different thermal treatments in the DSC equipment. In the first series of thermal treatments, sucrose crystals were heated to different final temperatures (from 190 to 235 °C), using the same heating rate of 10 °C/min. For the second series, sucrose crystals were heated to 200 °C, using different heating rates (5, 10, 20, 30, and 40 °C/min); 200 °C was chosen to allow the melting of the whole sample at 40 °C/min. Finally, for the third series of treatments, the sucrose crystals were melted by heating to 190 °C at 10 °C/min and then maintained at this temperature for two different residence times (5 and 10 min) before cooling.

HPLC Conditions. Heated sucrose was analyzed in a Hitachi Merck liquid chromatograph equipped with an RI detector (Bischoff 8110 refractometer) and a D-2500 chromatointegrator. Separations of compounds were made on an Aminex ion-exclusion HPX-87H column from Bio-Rad Laboratories (Richmond, CA); the length and diameter were, respectively, 300 and 7.8 mm. The mobile phase used for the elution consisted of a filtered 5 mM solution of sulfuric acid; the flow rate was 0.6 mL/min. Measurements were made at ambient temperature. The HPLC was calibrated with sucrose, glucose, and fructose solutions of known concentrations.

RESULTS

A typical DSC heating trace, for a final temperature of 200 °C, is shown in Figure 1. The solid-line curve

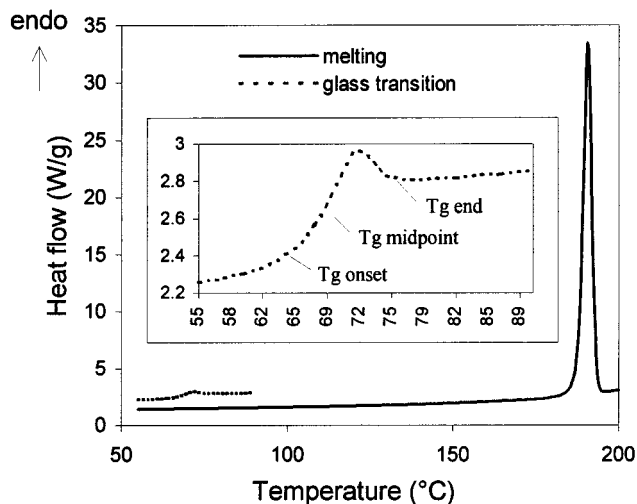


Figure 1. DSC thermogram for dry sucrose: the first heating scan (solid line) shows the crystal melting; the second heating scan shows the glass transition (dotted line).

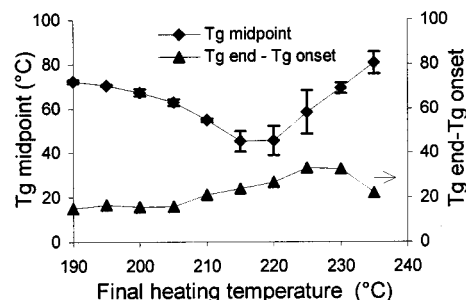


Figure 2. Changes in the different characteristic temperatures of the glass transition, as a function of the final heating temperature.

corresponds to the melting of crystalline sucrose; the dotted-line curve shows the glass-liquid transition for the molten sucrose.

Influence of Final Heating Temperature. The final heating temperatures were chosen according to the melting temperature of sucrose, as observed by DSC. The temperature of 190 °C corresponds to the melting peak maximum for anhydrous sucrose obtained by DSC, using a heating rate of 10 °C/min (Figure 1). Lower temperatures (185 and 187.5 °C) were also tested; they correspond to the onset and the midpoint between the onset and maximum of the DSC melting peak. In fact, with the chosen heating rate, these two temperatures were too low and the sucrose was not completely melted. Thus, during cooling, crystallization occurred, and the formation of sucrose glass was avoided. Higher temperatures up to 235 °C were also tested.

The variations in the value of the T_g with the maximum temperature reached during crystal melting are shown in Figure 2. T_g values of the different melts decreased when the final heating temperature increased from 190 to 215 °C; the T_g then increased for the higher temperatures. Changes in the width of the glass transition, corresponding to the interval between $T_{g,end}$ and $T_{g,onset}$, can also be observed in Figure 2. The interval is twice as large for 225 °C as for 200 °C. We also note the larger dispersion of the measurements around 215–225 °C, possibly due to more heterogeneous material or to a changing sample.

The variation in the enthalpy change corresponding to ΔC_p , was relatively small at 190 °C, increased at 195

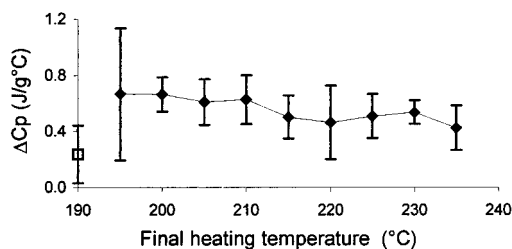


Figure 3. Variations in ΔC_p with the final heating temperature; the sample \square is incompletely melted.

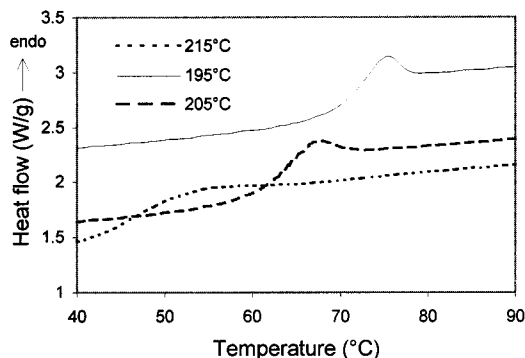


Figure 4. DSC thermograms for glasses obtained after melting to final heating temperatures of 195, 205, and 215 °C.

°C, and then decreased slowly as shown in Figure 3. The small enthalpy change observed at 190 °C probably corresponded to incomplete sucrose melting.

The change in the shape of the DSC thermogram according to the final heating temperature parameter was also considered. The curves in Figure 4 show not only the prominence of the glass transition feature but also the absence of a relaxation peak for the highest final temperature.

The composition of the glasses obtained after the different thermal treatments was investigated by HPLC analysis. Figure 5 shows the chromatograms for two samples, corresponding to heating to 190 and 215 °C. After heating to 190 °C, only the sucrose peak was visible; no evidence of a significant degradation reaction was observed. After heating to 215 °C, the sucrose peak had disappeared; the peaks for glucose and fructose were still evident, and polymerization products appeared; their retention times were shorter than that for sucrose.

Figure 6 summarizes all of the HPLC results; the T_g values are also represented. The sucrose content decreased sharply as the final temperature increased, and it disappeared completely above 215 °C. The fructose and glucose contents increased; their maximum concentration was obtained at 215 °C. The other compounds (polymerization products, calculated by difference) were prominently shown for temperatures >215 °C.

Influence of Heating Rate. Different heating rates were applied, for a final temperature fixed at 200 °C. Figure 7 depicts the increase in T_g with heating rate. Because a lower heating rate corresponds to a more severe thermal treatment, these results confirm those obtained for different final heating temperatures. The concentration of sucrose changed in the same way as T_g ; both increased with increasing heating rate. Moreover, glucose and fructose concentrations decreased. In other words, a higher T_g value corresponds to better preservation of sucrose.

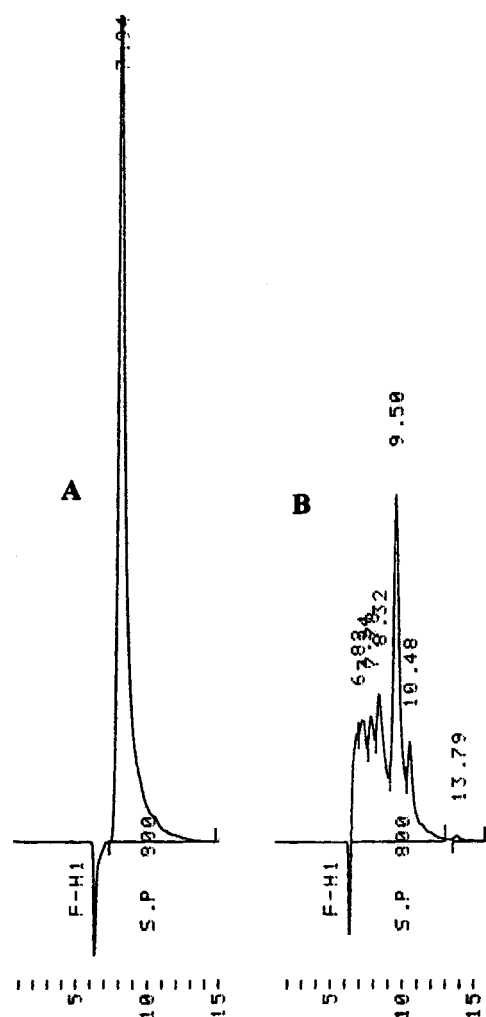


Figure 5. HPLC chromatograms for sugar solutions, corresponding to two heating temperatures: 190 °C (A) and 215 °C (B). The retention times were 7.95 min for glucose, and 10.50 min for sucrose.

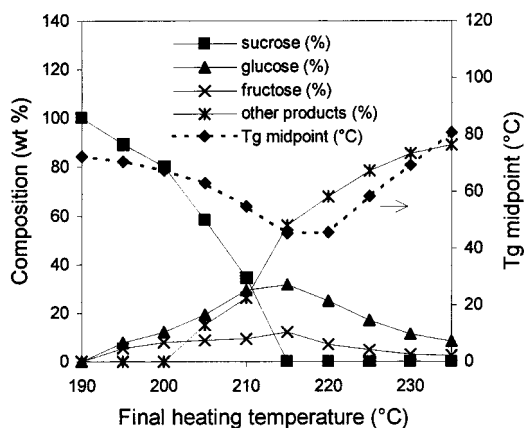


Figure 6. Changes in sucrose, degradation product concentrations, and T_g with the maximum temperature reached during crystal melting.

The change in concentration of the products resulting from sucrose degradation was surprising for heating rates of 30 and 40 °C/min; apparently, sucrose was preserved, but a small quantity of fructose was present. Our analytical technique was not precise enough to obtain a good separation between sucrose and glucose peaks when the former was still present in large amount. However, the presence of fructose indicated

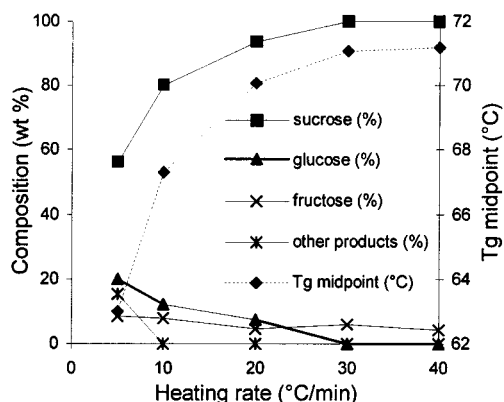


Figure 7. Changes in sucrose, degradation products, and T_g with the heating rate.

Table 1. Sucrose, Glucose, and Fructose Compositions as Functions of the Residence Time at 190 °C

residence time (min)	sucrose (wt %)	glucose (wt %)	fructose (wt %)	other products (wt %)
0	100	0	0	0
5	36.1	27.1	9.2	27.7
10	0	29.2	10.7	60.1

Table 2. Change in the Temperature and Width of the Glass Transition with Residence Time at 190 °C (σ = Standard Deviation, $n = 3$)

residence time (min)	$T_{g, \text{midpoint}} (\sigma)$	$T_{g, \text{end}} - T_{g, \text{onset}} (\sigma)$
0	72.2 (0.2)	14.9 (0.8)
5	57.0 (0.3)	18.4 (1.4)
10	45.6 (0.6)	19.5 (1.46)

that sucrose degradation had started, although it was limited. A high heating rate was associated with a gentle thermal treatment.

Influence of Residence Time at High Temperature. Sucrose crystals were heated to 190 °C, and the melt was maintained for a specified time at this temperature before cooling. The composition and the T_g of the resulting glasses are reported in Tables 1 and 2. The results of this experimental series confirmed the ones previously described: sucrose degradation increased with the intensity of the thermal treatment.

The sugar composition, as determined by HPLC (Table 1), shows that the sucrose fraction decreased, whereas the fructose and glucose contents increased, and rapidly increasing MW products appeared as the residence time increased. However, T_g remained low (Table 2). The width of the glass transition range was an indicator of the complexity of the composition. The residual sucrose content was similar to values obtained by Richards and Shafizadeh (1978) for a similar thermal treatment.

DISCUSSION

All of the HPLC results confirmed the hypothesis about thermal degradation of sucrose during crystal melting. This degradation increased when the time spent at high temperatures was long. Initially, the thermal decomposition of sucrose proceeded via scission of the glucosidic linkage, producing glucose and fructose, but the fructose content rapidly become lower than that of glucose (Figure 6); this disappearance was compensated by the appearance of larger molecules.

The work of Richards and Shafizadeh (1978) allows us to interpret these results. They investigated the

initial products of thermal degradation of sucrose and claimed that at 194 °C, sucrose undergoes a complex series of nonspecific reactions that are very difficult to analyze. Thus, they reported that the main products are glucose and disaccharides, with smaller amounts of fructose and 1,6-anhydroglucoses. They concluded that the predominant initial reactions are as follows. First, anhydrous fructose is formed; its reactivity allows its condensation with itself to form difructose dianhydrides. It can also react quickly with an available alcohol group. During the initial stage of the reaction, undegraded sucrose hydroxyls can play this role and produce, in this way, trisaccharides.

Richards (1986) was interested in the degradation of anhydrous sucrose melt at 135 °C. Fructose was again found in lower concentration than glucose, because it was more susceptible to consecutive thermal degradation. Trisaccharides (kestose) were formed in smaller quantities; traces of anhydrofructoses and disaccharides, except sucrose, were also found. These experiments showed that when molecular mobility is possible, that is, when a noncrystalline state is created, sucrose rapidly degrades.

After a short time at high temperature or a longer time at lower temperature, the products obtained would correspond to the beginning of degradation, that is, to the formation of small molecules arising from the scission of sucrose, with little condensation. For severe thermal treatments, a polycondensation resulting in different high molecular weight molecules could occur.

The observed T_g shifts can be explained by the appearance of molecules with different molecular weights and different T_g values. T_g increases with increasing molecular weight (Fox and Flory, 1950). For a homogeneous blend, T_g is a function of the T_g of each compound and its mass fraction (Gordon and Taylor, 1952). Indeed, the T_g of a monomer is lower than that of its polymer. The reported T_g values for glucose and fructose are, respectively, around 31 to 39 and -4 to 13 °C (Finegold et al., 1989; Orford et al., 1990; Ablett et al., 1992; Roos, 1993). For maltodextrines, Roos and Karel (1991b) reported T_g values of 141, 160, and 188 °C, respectively, for molecular weights of 900, 1800, and 3600.

Our HPLC results confirmed that the melts formed by different thermal treatments were complex blends of several components with different molecular weights, so changes in their T_g values could be expected. T_g decreased when small molecules were present in large proportion in the blend, and, in the opposite way, increased when polymers with high molecular weights were major constituents.

The change in width of the glass transition, observed for the highest temperature reached during the melting process, corresponded to the appearance of both smaller molecules (glucose, fructose, and other isomers) and larger molecules via polymerization. The breadth of the glass transition range, as observed in DSC scans, should be credited to the nonmiscibility of the compounds resulting from degradation with each component contributing its own T_g .

Finally, the disappearance of the T_g relaxation phenomenon, as observed by DSC, when the final temperature was high could also be explained by the presence of higher molecular weight compounds, for which the relaxation rate would be lower. With small molecules, the cooling time was long enough to induce aging in the glass (Simatos et al., 1995); an enthalpy relaxation peak

was observed both for sucrose and for monosaccharide plus disaccharide blends (Figure 4).

The degradation state for sugars depends on time-temperature factors; with the thermal conditions used in our experiments, we can say that the more important factor was temperature. At 190 °C, scission was the most important stage of degradation; polymerization was limited, because the T_g values were always lower than that for sucrose. More precise chemical analyses could confirm this conclusion. The identification of all the degradation products was not complete or precise; other techniques such as gas chromatography would be necessary to improve our analyses.

Our analysis concentrated on the reasons for the variability observed in published T_g values for sucrose. For that purpose, we investigated the effect of the melting conditions for sucrose on its T_g , but an ideal melting condition to form sucrose glass could not be determined. Up to now, we typically measured the T_g of sucrose obtained from crystals melted at 200 °C, because this temperature corresponds to the end of the melt of this compound, as observed by DSC analysis (Blond et al., 1997). The present results show the sucrose degradation would have begun. At 190 °C, the temperature at which the sucrose concentration was at its maximum, the enthalpy change was low. The entire sample may not have been completely glassy, because the crystals initially present in the DSC pan may not have had enough time to melt. For these reasons, a better method than crystal melting to prepare a pure sucrose glass should be used, for instance, dehydration of a sucrose solution by a freeze-drying process. The T_g value given by Saleki-Gerhardt (74 °C) is very close to the maximum value (72 °C) found in these experiments. The problem with freeze-dried products is their very high hygroscopicity, so they should be handled with great care in a dry atmosphere.

The determination of the T_g of other sugars from the crystal melting is also questionable; it is a study subject yet.

CONCLUSION

The aim of this study was to explain the differences among the sucrose T_g values found in the literature; therefore, the impact of the thermal treatment (final heating temperature, heating rate, residence time) undergone by samples before the T_g measurement was investigated.

The main conclusions of this study are as follows:

T_g decreases with increasing heating temperature or residence time and with decreasing heating rate, that is, when the proportion of small molecules (glucose, fructose) increases.

T_g increases after a severe thermal treatment, that is, when the proportion of molecules with high molecular weight (polymerization products) in the melt increases.

Perhaps most importantly, the analyzed glass is often not a pure sucrose glass; a reduced water quantity can be present.

Thus, T_g depends strongly on the melting conditions for sucrose crystals, but an ideal method to form pure sucrose glass by melting was not found; the product obtained is a complex mixture of molecules the sizes of which are a function of the intensity of a given thermal treatment.

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