

# Effects of Sugars and Emulsifiers on Starch Gelatinization Evaluated by Differential Scanning Calorimetry<sup>1</sup>

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## ABSTRACT

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Differential scanning calorimetry (DSC) was used to evaluate the effects of sugar and emulsifier interactions on gelatinization temperatures of three different starches. A constant weight ratio of starch to sugar to water (1:1.5:1.5), typical in high-ratio cake batters, was used in the DSC. Sucrose exhibited a greater effect than glucose on raising gelatinization temperatures. However, with lactose, typical complete gelatinization endo-

therms could not be observed because of the peak for large lactose crystal melting. Therefore, the solubility of the sugars may have an important effect on starch gelatinization. Low emulsifier concentrations (0.6%) did not appear to change DSC starch gelatinization temperatures. Sugars and emulsifiers may interact and affect the gelatinization temperature ranges.

The control of starch gelatinization in cake systems has been emphasized as important in obtaining a noncollapsing, porous cake structure (Bean and Yamazaki 1973, Glover et al 1986). When various sources of commercial starches replaced flour, variations occurred in cake-baking performance (Howard et al 1968, Kulp and Lorenz 1981, Glover et al 1986).

It has long been known that various sugars raise gelatinization temperature (Bean and Yamazaki 1978, Spies and Hosney 1982, Slade and Levine 1987, Buck and Walker 1988). Generally, monosaccharides delay gelatinization less than disaccharides, except for maltose, which acts like a monosaccharide. The delay of starch gelatinization in a sugar solution has been attributed to the abilities of sugar to 1) limit water availability to the starch granule, 2) lower the water activity, 3) form sugar bridges between starch chains, and 4) exert an antiplasticizing effect, relative to water. The exact reaction mechanism by which sugar delays starch gelatinization still remains unclear, however.

Aside from the obvious emulsifying action, emulsifiers may interact with other components during baking. When emulsifiers

were added to starch, the gelatinization temperature changed (Eliasson 1986, Buck and Walker 1988). This might be attributed to an amylose-lipid complex formation. The amount of complex formed was affected by the starch (Eliasson 1985, 1986) and lipid sources (Krog 1971, 1977; Larsson 1980; Riisom et al 1984; Eliasson and Krog 1985).

Cakes containing sucrose esters tended to collapse immediately upon removal from the oven (Ebeler and Walker 1984). Later, verification of this phenomenon was supported by observing increased gelatinization temperatures with the addition of sucrose ester at 1 and 3% to wheat starch-sucrose mixtures (Buck and Walker 1988). When sucrose ester F-160 (SE) or polysorbate 60 (PS) emulsifiers were added to the starch-sugar mixtures (at 0.5-1.5%), differences in paste consistency were clearly detected at all concentrations of SE in a Rapid Visco Analyser (Newport Scientific, Narrabeen, Australia) pasting study in our laboratory (unpublished data).

The objective of this study was to evaluate the effects of sugars, emulsifiers, and their interactions on gelatinization temperatures for different starches.

## MATERIALS AND METHODS

Wheat starch was provided by Manildra Milling Corp., Minneapolis, MN; corn (maize) starch was obtained from National Starch and Chemical Corp., Bridgewater, NJ; potato starch was purchased from Raisio Inc., Berwick, PA.

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Food-grade sugars were used in all studies. Fine granulated sucrose was donated by the American Crystal Sugar Co., Moorhead, MN. Dextrose monohydrate (glucose) was obtained from Corn Products, CPC International Inc., Englewood Cliffs, NJ. Hydrated lactose was purchased from Land O'Lakes Inc., Arden Hills, MN.

PS, with a hydrophile-lipophile balance of 14.7, was provided by Humko Chemical Division, Witco Corp., Memphis, TN. SE was obtained from Dai-Ichi Kogyo Seiyaku Co., Kyoto, Japan. This emulsifier is a mixture of the stearic and palmitic acid esters of sucrose (Walker 1984). It contains approximately 70% mono-ester and 30% di-tri-poly ester, and the hydrophile-lipophile balance is approximately 15.

### Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) was done with a Perkin Elmer DSC-4 (Norwalk, CT). Indium was used to calibrate the instrument.

Before the samples were weighed, DSC sample pans were pretreated in an annealing process. The pans were placed in distilled water in glass dishes for about 1 hr, the water was drained, and the pans heated in an oven for 3 min at 300°C. They then were cooled to room temperature before use.

To examine the effects of sugars and emulsifiers on gelatinization, a constant starch to sugar to water weight ratio (1:1.5:1.5) was used in the DSC. This is approximately the ratio present in a typical high-ratio cake batter. Starch (2 mg) and sugar (3 mg, on a moisture-free basis) were weighed to within 0.1 mg directly into DSC aluminum sample pans on a microbalance, and then 2 mg of SE or PS solution containing 0.6% emulsifier, based on the dry weight of the starch, was added to the starch-sugar mixture with a Hamilton microsyringe. About 1 mg of distilled water was added, the pan was placed back on the microbalance, and any excess water was allowed to evaporate to make the final, total sample weight 8 mg. The pans were hermetically sealed. The pan with sample was weighed once more to record the final weight. Samples were allowed to equilibrate for at least 2 hr before analysis.

One minute was allowed for temperature equilibration of the sample chamber before starting to increase its temperature from 20 to 150°C. Samples were heated in the DSC at a rate of 10°C/min, with a sensitivity of 0.5 mcal/sec and a recorder chart speed of 10 mm/min. Another aluminum pan, containing an appropriate amount of aluminum to balance the heat capacity of the sample, was used as the reference. The starch gelatinization characteristics in a DSC thermogram can be indicated by various temperatures. The onset temperature ( $T_o$ ) and the conclusion temperature ( $T_c$ ) (points where the extrapolated endotherm intersects with the baseline) and the peak temperature (temperature of maximum differential heat flow) were determined. The gelatinization temperature range ( $T_c - T_o$ ) was obtained by subtracting the  $T_o$  from the  $T_c$ . The amounts of energy required for gelatinization ( $\Delta H$ ) were obtained by converting the areas under the peaks, measured with a planimeter, to calories by using a calibration coefficient determined from the thermograms of weighed amounts of indium with a known enthalpy of 6.8 cal/gm, expressed relative to the dry weight of the starch. To obtain the area, a baseline was constructed as a smooth line from the beginning to the end of the transitions.

The effect of water content on gelatinization was examined using mixtures of water and starch at 1:1, 1:1.5, and 1:3 starch-water ratios. Data obtained were based on a minimum of two replications. A personal computer statistical program, Number Cruncher Statistical Systems (Hintze 1987), was used for the analysis of variance and regression analysis.

## RESULTS AND DISCUSSION

### Gelatinization Temperatures of Commercial Native Starch

As seen in Table I, in the absence of sugar, commercial starches were characterized by the transition temperatures of their DSC endotherms and  $\Delta H$ . The  $T_o$  and peak ( $T_p$ ) temperatures were

lowest for wheat starch, higher for potato starch, and highest for corn starch. For  $T_c - T_o$ , potato was lowest, followed by corn and wheat. The  $\Delta H$  was much larger for potato starch (6.4 cal/g, 26.8 J/g) than it was for wheat (4.6 cal/g, 19.2 J/g) and corn (4.7 cal/g, 19.7 J/g) starches.

### Effects of Sugars

The effects on the gelatinization temperature and enthalpy of the addition of various sugars to the starch-water mixture may be observed by comparison with the starch-water results (Table I).

For the dextrose-starch mixtures, a peak appeared before the major gelatinization endotherm, but this was not the case with sucrose (Figs. 1-3). This endotherm may have been caused by melting of dextrose crystals, which have lower solubility than sucrose crystals, as clearly shown in Figure 4. Dextrose melting started at 30°C and was completed by 65°C.

With sucrose or dextrose addition to the starch-water mixture, the major starch gelatinization endotherms shifted to a higher temperature to different extents (Figs. 1-3). Even though they contained only 1.5 parts of water, their thermograms were very similar in appearance to those of the 1:3 starch-water ratio mixture (Fig. 5). More water appeared to be available to the starch with sugar addition. Many workers have observed that the addition of sucrose, which dissolves in the water, has the functional effect of increasing the solvent volume (Spies and Hosney 1982, Ghiasi et al 1983).

This phenomenon also was observed in the DSC thermograms of annealed starch, as reported in Slade and Levine's review (1987). According to French (1984), heating at a slow rate facilitates the annealing process, which may include realignment of the starch chains in their amorphous areas and some additional crystallization.

The behavior of the starch-sugar-water mixtures during heating in the DSC may be explained in the same way as the changes in the annealed starch granule structure (Slade and Levine 1987). Stabilization of the amorphous regions by interaction of the starch chains with the sugar would restrict the chain's flexibility (Spies and Hosney 1982), producing in turn a primary structural relaxation transition (glass transition of amorphous regions) at increased temperatures. An increased glass transition temperature may occur immediately, followed by and superimposed on the crystal's melting transition (Slade and Levine 1987). Thus, the temperature range becomes narrow.

More current work with corn starch (Johnson et al 1990) shows similar patterns for dextrose (glucose) and sucrose. They used slightly different starch-sugar-water ratios, however, so the  $T_o$  and  $T_c$  temperatures are shifted slightly in comparison.

TABLE I  
Comparison of Differential Scanning Calorimetry-Measured Gelatinization Temperatures and Enthalpies for Commercial Starches in the Presence of Sugar (Starch-Sugar-Water Ratio, 1:1.5:1.5) or for Starch-Water Mixture (1:1.5)

Sugar	Starch	Transition Temperature <sup>a</sup> (°C)				$\Delta H$ (cal/g) <sup>b</sup>
		$T_o$	$T_p$	$T_c$	$T_c - T_o$	
None	Wheat	55.0	62.0	82.5	27.5	4.6
	Corn	65.0	72.5	86.0	21.0	4.7
	Potato	59.0	63.8	78.3	19.3	6.4
Sucrose	Wheat	80.0	90.0	95.5	16.5	3.3
	Corn	88.8	97.3	104.0	15.2	3.8
	Potato	82.8	89.5	97.0	14.3	4.2
Dextrose <sup>c</sup>	Wheat	71.3	80.5	88.0	16.8	3.1
	Corn	81.3	89.7	95.8	14.5	3.9
	Potato	75.0	82.0	90.0	15.0	4.4

<sup>a</sup>  $T_o$  = Transition onset temperature,  $T_p$  = transition peak temperature,  $T_c$  = transition conclusion temperature,  $T_c - T_o$  = gelatinization temperature range. Two or more replications were averaged for each value.

<sup>b</sup> Based on dry starch weight.

<sup>c</sup> Dextrose = glucose monohydrate derived from corn (maize) starch.

This tightened amorphous structure would be expected to result in an increase in the  $\Delta H$  by DSC; however, a lower  $\Delta H$  was obtained from this study (Table I). It is difficult to explain this situation. The low  $\Delta H$  of the starch-sugar-water mixtures may have been due in part to the low water content of the starch-sugar-water ratio, 1:1.5:1.5, as compared with that in the starch-water mixture ratio, 1:1.5 (no sugars), used in this study. Other researchers have reported that the gelatinization enthalpy increased with increasing moisture content (Wootton and Bamunuarachchi 1979, Abboud and Hosney 1984).

With the addition of sucrose and dextrose, the  $T_o$ ,  $T_p$ , and  $T_c$  temperatures increased, and the  $T_c - T_o$  decreased for all three starches (Table I). Sucrose delayed gelatinization more than dextrose. The above results confirm the data reported in other studies (Evans and Haisman 1982, Spies and Hosney 1982, Buck and Walker 1988).

The  $\Delta H$  of sucrose or dextrose-starch combinations was not significantly different ( $P > 0.05$ ) for wheat or corn starches. However, the  $\Delta H$  of the dextrose-potato starch combination was significantly higher ( $P < 0.05$ ) than that of the sucrose-potato starch. This increased enthalpy might have been caused by some additional crystallization by specific interaction between dextrose and amylopectin molecules, which attach to the negatively charged phosphate. This hypothesis might explain why the potato starch-dextrose mixture produced a lower paste consistency by limited starch swelling, as seen in our Rapid Visco Analyser study (unpublished data).

When the lactose-starch mixtures were heated in the DSC, three small endotherm peaks (Fig. 4, curve E) appeared. The larger

peak is presumably for lactose, the result of an extremely low solubility. As the water-to-sugar ratio decreased from 1.7 to 1.0, the melting temperature of lactose crystal increased from 78 to 87°C (Fig. 4, curves C and D). With the addition of one part starch to the water-lactose mixture (1.5:1.5) (Table I), all of the third peaks were at or above 87°C. The large peak was assumed to be a melting peak of lactose crystal. With starch present, the lactose crystal melting was delayed to a higher temperature, competing with starch granules for water. Consequently, we assumed that the first and second peaks were the melting peaks of starch crystals. This may have been because less dissolved sugar solution was available to produce a single, sharp starch gelatinization endotherm. Thus, a typical gelatinization endotherm could not be observed.

The slow dissolving of lactose during the DSC heating may have given the starch granules more chance to absorb water compared with starch with sucrose and dextrose. More plasticizer (water) may have exerted a greater destabilization effect on the amorphous regions, and thus starch crystal melting occurred earlier.

Lactose increased the gelatinization temperature less than dextrose or sucrose. Therefore, the sugar's solubility should be

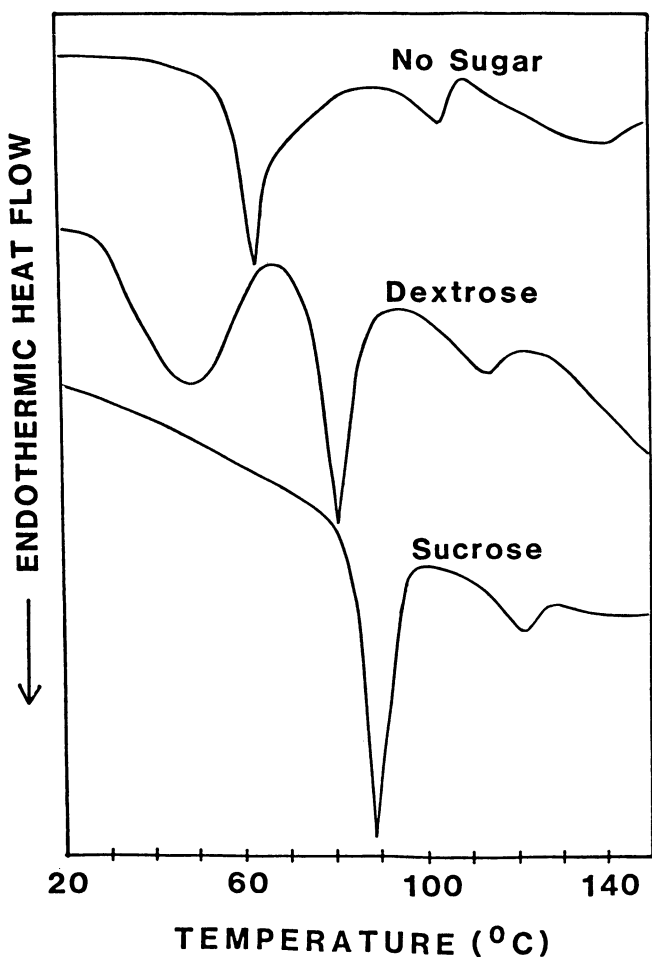


Fig. 1. Differential scanning calorimetry thermograms of wheat starch (2.0 mg) at a starch-water ratio of 1:1.5 (top line) and at a starch-sugar-water ratio of 1:1.5:1.5 for dextrose and sucrose (middle and bottom lines).

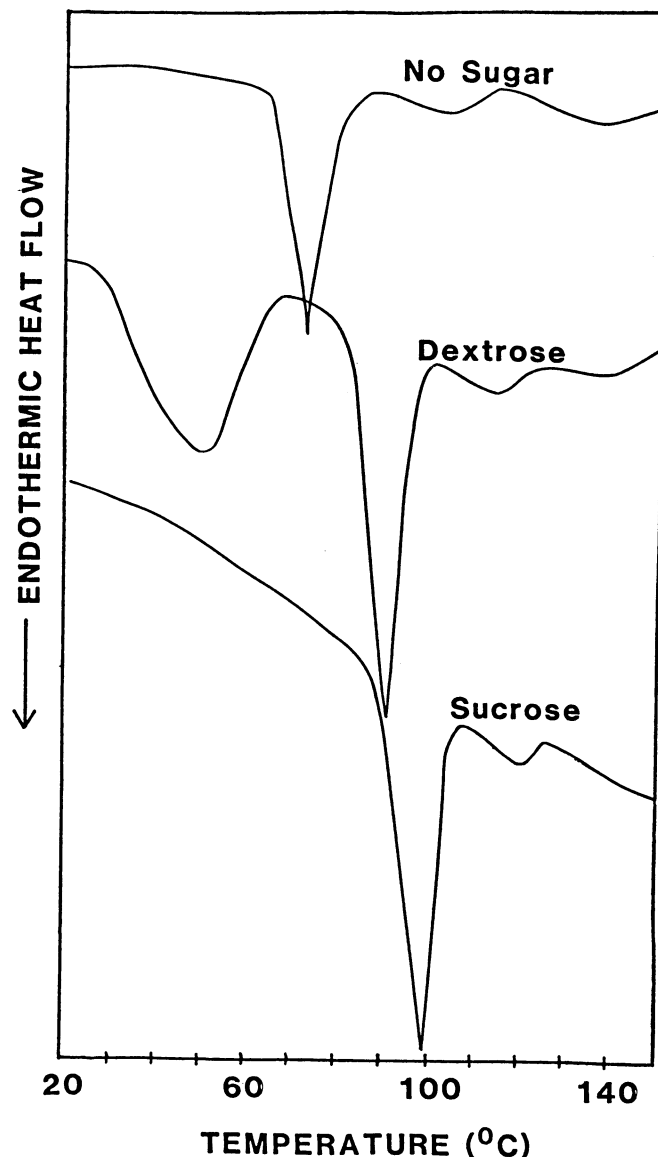


Fig. 2. Differential scanning calorimetry thermograms of corn starch (2.0 mg) at a starch-water ratio of 1:1.5 (top line) and at a starch-sugar-water ratio of 1:1.5:1.5 for dextrose and sucrose (middle and bottom lines).

considered an important factor affecting starch gelatinization temperatures.

#### Emulsifier's Effects

No significant differences ( $P > 0.05$ ) occurred in the gelatinization temperature and enthalpy of most treatments when SE or PS were added to starch-sucrose or starch-dextrose mixtures (Tables II and III).

Buck and Walker (1988) reported that, when SE at 1 or 3% was added to starch (corn or wheat) and/or starch-sugar (sucrose, dextrose, and fructose) combinations, SE had no significant effects on gelatinization temperatures, except for that of the wheat starch-sucrose combination. In that case, the peak temperature increased with increasing SE concentration.

When PS was added to mixtures of either the wheat or potato starch and lactose, a second starch melting peak did not appear. It might have been hidden by the lactose crystal melting endotherm, however. Therefore, the emulsifiers' effects on gelatinization temperatures could not be satisfactorily observed on the lactose DSC thermograms.

Although emulsifiers themselves did not affect the DSC gelatinization temperatures of most treatments (Table III), some interactions between the emulsifier and sugar were observed. No

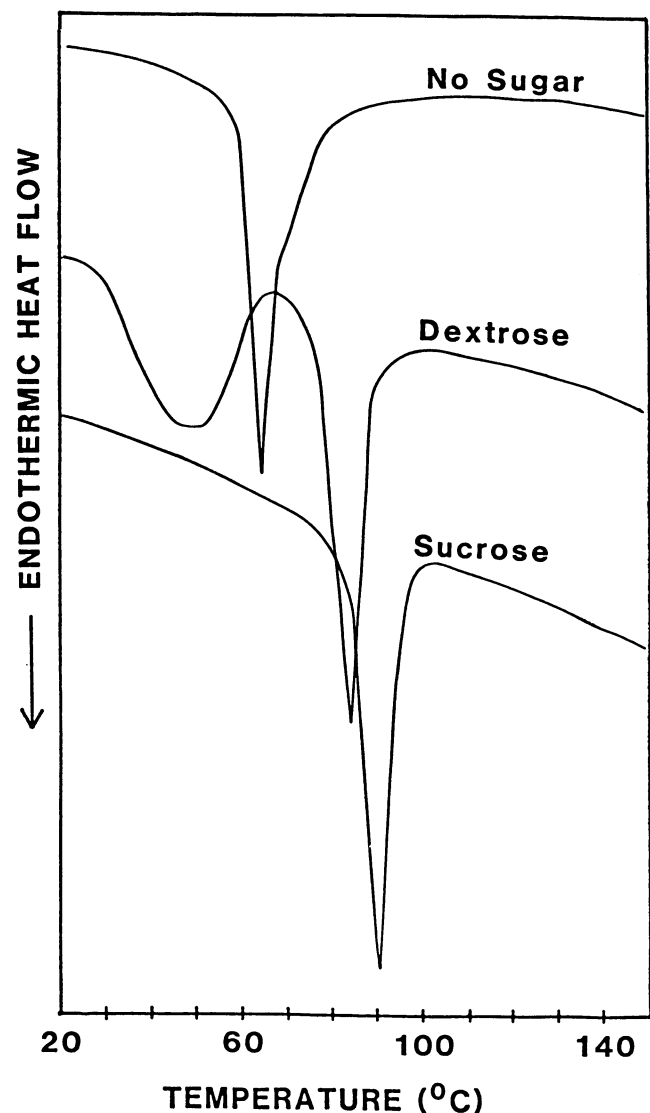


Fig. 3. Differential scanning calorimetry thermograms of potato starch (2.0 mg) at a starch-water ratio of 1:1.5 (top line) and at a starch-sugar-water ratio of 1:1.5:1.5 for dextrose and sucrose (middle and bottom lines).

significant differences in the endothermic  $T_c$  and the transition ranges ( $T_c - T_o$ ) between the starch-sucrose and starch-dextrose combinations were seen individually (Fig. 6). However, their overall trends were significantly different ( $P < 0.05$ ) in the presence of emulsifiers, indicating that sugar-emulsifier interactions might have occurred.

#### CONCLUSIONS

As a result of our study, we have drawn the following conclusions. The disaccharides (sucrose and lactose) had greater effects than the monosaccharide (dextrose) on starch gelatinization. A sugar's solubility may be an important factor affecting gelatinization temperature in a limited water system. At a very low concentration (0.6%), neither SE nor PS emulsifiers

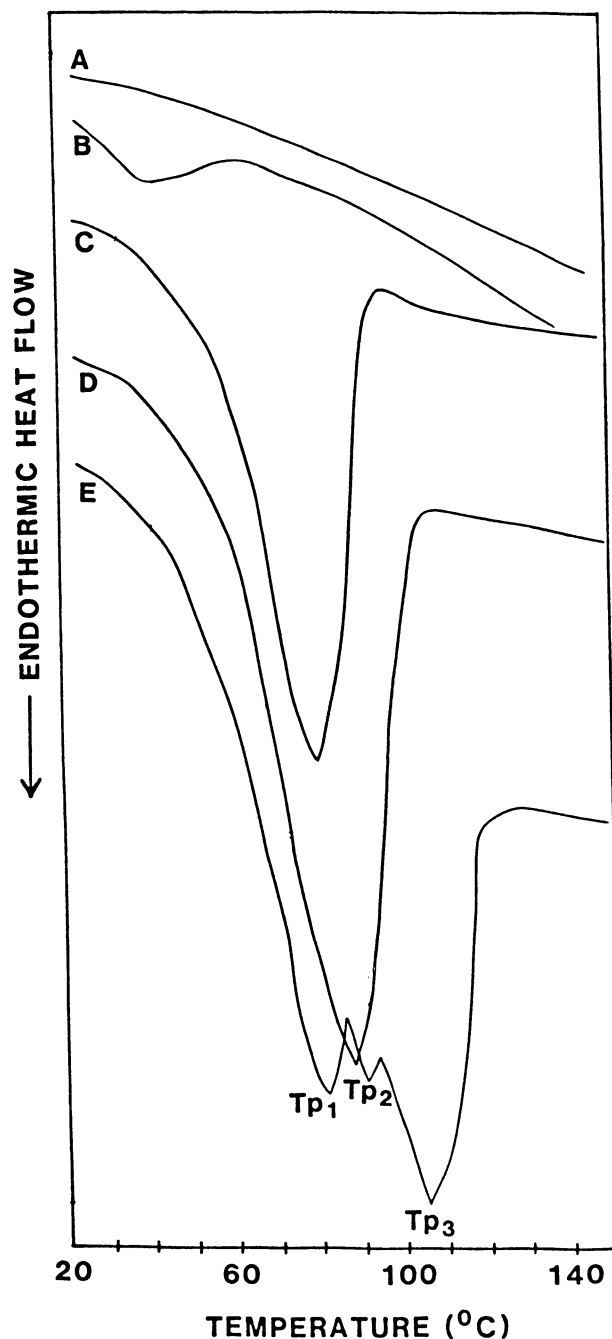


Fig. 4. Differential scanning calorimetry thermograms of sugars. A, Sucrose, and B, dextrose, at a water-sugar ratio of 1:1. C and D, Lactose at water-sugar ratios of 1.7:1 and 1:1. E, Corn starch-lactose-water mixture (1:1.5:1.5).  $T_{p1}$ ,  $T_{p2}$ ,  $T_{p3}$  = peak temperatures.

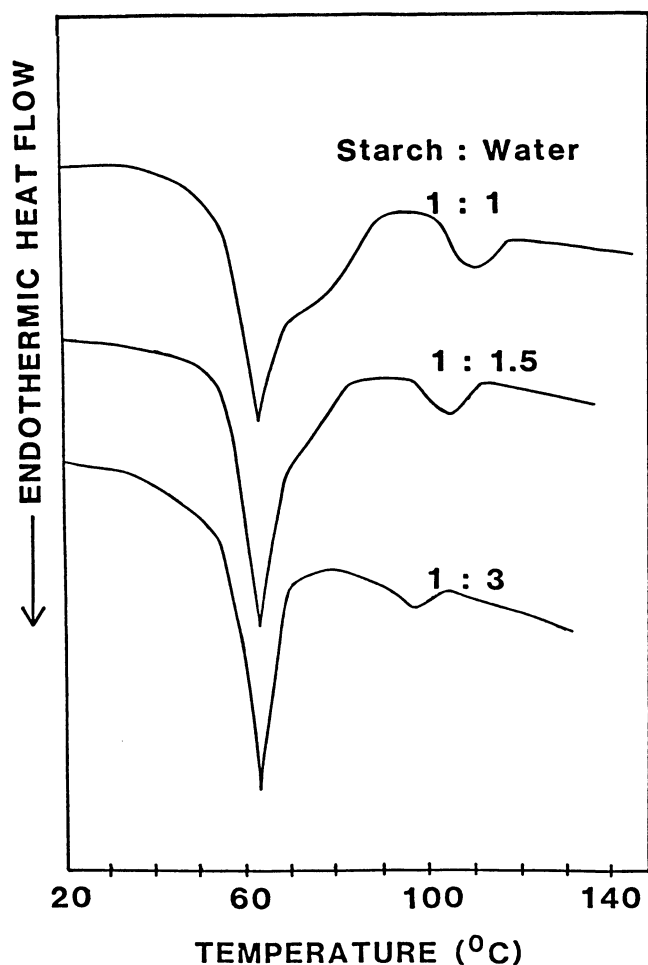
**TABLE II**  
Gelatinization Temperatures and Enthalpies of Commercial Starches in the Presence of Sugar and Emulsifier (Starch-Sugar-Water Ratio, 1:1.5:1.5), as Measured by Differential Scanning Calorimetry

Starch	Sugar	Emulsifier <sup>b</sup>	Transition Temperature <sup>a</sup> (°C)				$\Delta H$ (cal/g) <sup>c</sup>
			$T_o$	$T_p$	$T_c$	$T_c - T_o$	
Wheat	Sucrose	None	80.0	90.0	96.5	16.5	3.3
		SE	80.5	90.3	99.0	18.5	3.3
		PS	81.0	91.3	100.0	19.0	3.0
	Dextrose	None	71.3	80.5	88.0	16.8	3.1
		SE	71.3	80.3	87.3	16.0	2.8
		PS	71.5	80.5	86.3	14.8	2.9
Corn	Sucrose	None	88.8	97.3	104.0	15.2	3.8
		SE	91.5	100.5	106.8	15.3	3.9
		PS	89.9	98.8	105.2	15.3	3.8
	Dextrose	None	81.3	89.7	95.8	14.5	3.9
		SE	81.0	89.1	94.5	14.5	3.9
		PS	81.5	89.5	95.5	13.8	3.7
Potato	Sucrose	None	82.8	89.5	97.0	14.3	4.2
		SE	83.0	90.5	98.0	15.0	4.2
		PS	82.0	89.0	96.7	14.7	4.1
	Dextrose	None	75.0	82.0	90.0	15.0	4.4
		SE	76.8	83.5	91.0	14.3	4.5
		PS	77.0	83.0	90.5	13.5	4.7

<sup>a</sup> $T_o$  = Transition onset temperature,  $T_p$  = transition peak temperature,  $T_c$  = transition conclusion temperature,  $T_c - T_o$  = gelatinization temperature range. Two or more replications were averaged for each value.

<sup>b</sup>0.6% based on dry starch weight. SE = Sucrose ester, PS = polysorbate.

<sup>c</sup>Based on dry starch weight.



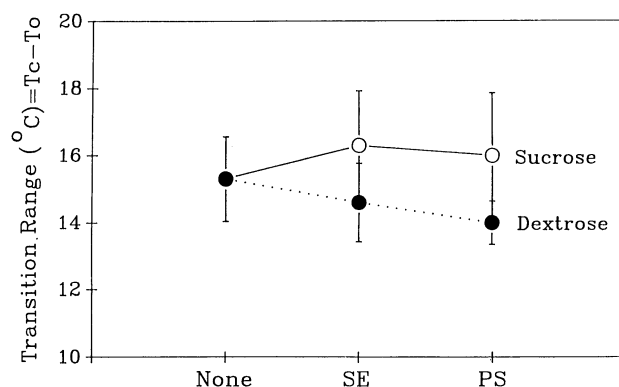
**Fig. 5.** Differential scanning calorimetry thermograms of wheat starch at different starch-water ratios, using 4.0, 3.2, and 2 mg of starch from top to bottom, respectively.

**TABLE III**  
Interactions of Variables in Gelatinization Thermogram of Differential Scanning Calorimetry

Source	Significance of $F$ -Value from Analysis of Variance <sup>a</sup>				$\Delta H$ (cal/g)
	$T_o$	$T_p$	$T_c$	$T_c - T_o$	
Starch	S	S	S	S	S
Sugar <sup>b</sup>	S	S	S	S	NS
Emulsifier	NS	NS	NS	NS	NS
Starch $\times$ sugar	S	S	S	S	S
Starch $\times$ emulsifier	NS	NS	NS	NS	NS
Sugar $\times$ emulsifier	NS	NS	S	S	NS
Starch $\times$ sugar $\times$ emulsifier	NS	NS	NS	NS	NS

<sup>a</sup> $T_o$  = Transition onset temperature,  $T_p$  = transition peak temperature,  $T_c$  = transition conclusion temperature,  $T_c - T_o$  = gelatinization temperature range. S = Significant at the 5% level if probability less than 0.05. NS = Not significant at the 5% level if probability larger than 0.05.

<sup>b</sup>Sucrose and dextrose were used.



**Fig. 6.** Interactions between sugars and emulsifiers, sucrose ester F-160 (SE) and polysorbate 60 (PS), on gelatinization temperature range through differential scanning calorimetry thermograms. Values are averages for all replications across all starches.

appeared to change the DSC starch gelatinization temperature, with the possible exception of the corn starch-SE-sucrose system. Sugars and emulsifiers may interact to increase or decrease the  $T_c - T_o$ , depending on the sugars. More work will be done in actual high-ratio cake systems with these starches, sugars, and emulsifiers to see how they affect the quality of the finished product.

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