

Effect of sucrose on the structure, mechanical strength and thermal properties of corn extrudates

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The effects of sucrose on extrusion process parameters and the structural, mechanical and thermal characteristics of the resultant extrudates have been investigated. To this end, sucrose in concentrations between 0 and 10% by weight was added to corn meal prior to extrusion at two levels of moisture (15 and 20%). The resulting data revealed the following significant features. (i) At the higher moisture level, sucrose progressively reduced the specific mechanical energy, while exhibiting little effect at the lower moisture level. (ii) At both moisture levels, sucrose increased the bulk density and reduced the cell size; this effect was progressive for high extrusion moisture samples and evident only at high sucrose contents for low extrusion moisture samples. (iii) In extrudate samples equilibrated to moisture contents between 12 and 17% wt, sucrose progressively plasticized the structures, as assessed by compression, dynamic mechanical spectrometry, and differential scanning calorimetry. In the aggregate, these results showed that the addition of sucrose requires a modification of the extrusion operating conditions to produce extrudates with optimal textural and storage properties.

INTRODUCTION

Sucrose is a common additive in commercially extruded foods, particularly breakfast cereals, and is incorporated into these products in concentrations up to 50% by weight (Hsieh *et al.*, 1990). In general, sugar concentration in dough ranges from 6 to 25% wt on a final dry product basis (Walker, 1990). Godshall (1988) reports that sugar contributes binding, flavor, and browning characteristics, is critical to controlling texture and mouthfeel, and acts as a carrier and potentiator of other flavors. Sucrose has been found to affect significantly the structure and texture of extrudates, although the reported direction and magnitude of such effects (as well as the specific experimental parameters used) have varied.

In the presence of adequate moisture during extrusion (above ~16% wt) the effect of sucrose on extrudate structure manifests itself as an increase in product density and a reduction in expansion with increasing sucrose concentration (Moore *et al.*, 1990; Sopade & Le Grys, 1991a; Ryu *et al.*, 1993; Jin *et al.*, 1994). In corn meal extrudates, very high concentrations of sucrose

(20–50% by weight) together with 25% wt processing moisture were found to reduce expansion progressively and to facilitate product collapse (Sopade & Le Grys, 1991a). Later, Jin *et al.* (1994) found that even low concentrations of sucrose (2–12% wt) in the presence of 20% (wt) moisture in feed resulted in an increase in bulk density and a reduction in expansion of corn meal extrudates. In wheat flour extrudates, Moore *et al.* (1990) noted an increase in apparent density with sucrose concentration (3–18% wt). Ryu *et al.* (1993) also reported that increasing sucrose concentration from 5 to 10% by weight significantly affected the expansion and cell structure by reducing expansion, increasing bulk density, increasing the number of cells per unit area, and also resulted in an increase in mechanical strength. In contrast to the results at high extrusion moisture content, when the feed moisture is kept low at 13% by weight, Hsieh *et al.* (1990) reported reduced bulk density and enhanced expansion for corn meal extrudates with added sucrose (2–8% wt). The structural changes in extrudates with the addition of sucrose have been attributed to competition for moisture (Hsieh *et al.*, 1990) and inhibition of gelatinization (Sopade & Le Grys, 1991a) during expansion. The influence of physical structure of extrudates on strength

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of these materials was reported by various investigators (Barrett & Peleg, 1992; Barrett *et al.*, 1994).

The modification of extrudate texture by addition of sucrose is due to plasticization of these systems by sucrose, as well as to structural effects. Plasticization of starch systems by small molecular weight constituents is a widely reported phenomenon (Shogren *et al.*, 1992; Slade & Levine, 1995). Plasticization results in an increase in the molecular mobility of polymers, a feature which is reflected in a number of observables. Specifically, a decrease in glass transition temperature (T_g) measured by differential scanning calorimetry (DSC) is observed with the addition of a plasticizer. Similarly, the value of $\tan \delta$, a measure of structural relaxation which can be detected by DMA (or DMTA), shifts to lower temperatures upon plasticization. In addition to these two commonly used techniques, NMR and the Instron Universal Testing Machine also have been used to detect this thermally induced event which relates to a change in state of a material under investigation. Slade and Levine (1995) recently have extensively reviewed studies involving various measuring techniques which are sensitive to changes in segmental mobility of biopolymers.

For corn starch extrudates, Shogren *et al.* (1992) observed that mixtures of urea and glycols increase the maximum elongation of the samples during tensile testing. Similarly, with increasing levels of added glucose, Ollette *et al.* (1991) observed progressive reduction in the stress-strain functions of extruded wheat starch during bending tests. Furthermore, moisture-induced textural plasticity in extrudates is evident from results of a number of studies. Kaletunç and Breslauer (1993) reported a rapid decrease in T_g of corn flour extrudates with increases in moisture content. Halek *et al.* (1989) found changes in texture profile parameters with moisture content. Sauvageot and Blond (1992), identified a 'critical' moisture content at which commercial breakfast cereals lost sensory crispness. Barrett *et al.* (1992), Rhode *et al.* (1993), and Wollney and Peleg (1995) showed losses in fracturability indices, such as the fractal dimension and the power spectrum of stress-strain functions, due to equilibration of samples at high relative humidity. In addition, sugars have been found to reduce the glass transition of (non-extruded) amylopectin in fructose-containing systems (Kalichevsky & Blanshard, 1993). A similar result was found by Kalichevsky *et al.* (1993), who compared the effects of sucrose, fructose, glucose and xylose. In the latter study, reductions in T_g were observed for each additive, although the magnitude of this effect was found to be ingredient-specific and to be influenced by moisture content.

Addition of sucrose alters the extrudate properties due to interactions between sucrose and components of cereal flours and due to its effects on the extrusion processing conditions. The results reported here build

on and expand our understanding of the influences of sucrose, a common additive in extruded foods. Specifically, the progressive effects of sucrose content (0, 2, 4, 6, 8 and 10% by weight) on extrudate cell structure, mechanical strength, and thermal properties have been determined and reported here. In addition, to determine interactive effects of sucrose level and processing moisture on extruder parameters and product characteristics, samples also were produced at two levels of extrusion moisture (15 and 20% wt). Finally, to assess the influence of storage conditions, the extrudate moisture contents were adjusted by equilibrating samples at different relative humidity levels prior to thermal or mechanical testing.

EXPERIMENTAL

Extrusion

Corn meal based extrudates (corn meal from Lincoln Grain Co.) containing 0, 2, 4, 6, 8 and 10% wt sucrose were produced on a twin screw extruder (Werner & Pfliederer, ZSK-30) using low (15% wt, 'Batch 1') and high (20% wt, 'Batch 2') initial moisture contents. Corn meal, sucrose, and water were each metered independently into the extruder using the following fixed parameters: 400 rpm screw speed; 27.2 kg/h solids feed rate; and a barrel temperature profile of 38-38-116-116-138-138°C. After extrusion, the products were freeze-dried and vacuum-canned until analysis. All percentages reported are by weight and on a wet basis.

Equilibration

The extrudates were held in desiccators containing saturated solutions of sodium chloride or potassium nitrate, which at 25°C produce relative humidities of 75% and 93%, respectively (Greenspan, 1976). The initial moisture contents of the freeze-dried samples were measured using a Computrac moisture analyzer and found to fall between 1 and 2%. The moisture contents after equilibration were determined by measuring increases in sample weight. Before analysis, the samples all were equilibrated to specific moisture contents. However, since the high extrusion moisture samples absorbed moisture less readily than did the low extrusion moisture samples, the precise humidity condition or equilibration time (between 2 and 6 days) varied between the extrusion batches. Actual moisture contents are reported for each analytical procedure.

Structural characterization

The area sizes of cells in cut sections of the (dry) extrudates were measured using an Olympus CUE 2 image analyzer and the procedure reported by Barrett and

Ross (1990). Three transverse and three longitudinal sections of each sample were measured and the data imported into a Minitab statistical program which calculated the mean cell size for the combined distributions (total number of cells was approximately 100–170).

Bulk density was calculated by cutting and weighing 10 cm lengths of the extrudates, averaging four caliper measurements of the diameter of each specimen, and calculating volume and density based upon geometric formulas for a cylinder. Density measurements for four sample replicates were averaged.

Dynamic mechanical spectrometry

A dynamic mechanical spectrometer (Seiko, DMS-110) was used to determine thermal transitions in the extrudates. Samples equilibrated at 17% moisture were compressed flat (1–2 mm thick) on a Carver press operated at a maximum force of 500 kg and carved into approximately 3 cm × 1 cm rectangles. Exact dimensions were measured before analysis. The samples were then subjected to oscillating three-point bending over a temperature range of –50 to 100°C, with a heating rate of 2°C/min. In each case, the peak temperature of the $\tan \delta$ curve for the 1 Hz frequency oscillation was recorded. The peak temperatures of between two and three replicates were averaged.

Differential scanning calorimetry

Thermograms of each corn extrudate were determined using a computer-controlled, pressure-variable, temperature-scanning calorimeter (Setaram DSC 111, France). The instrument was calibrated by the manufacturers with a joule effect calibration device. Calibrations for enthalpy and temperature were checked periodically using indium. All extruded samples were ground with a mortar and pestle and then equilibrated to 13% moisture content. DSC runs of the moist samples were carried out at 25 atm, thereby shifting the water peak outside the range in which the thermal transitions of the extrudates are observed. All DSC measurements were conducted using fluid-tight stainless steel crucibles with controlled pressure environments. An empty crucible was used as the reference. The sample and reference were heated at 5°C/min, with the upper temperature limit of the runs being dictated by the thermal decomposition of the samples. The resulting heat flow vs temperature data were analyzed to construct heat capacity curves. T_g values were determined from the inflection points of the specific heat capacity vs temperature curves. To eliminate differences due the thermal history of samples, as well as an overlapping irreversible endothermic transition present at approximately 40–50°C in the first heating scan, T_g always was determined from the second heating scan after cooling at 10°C/min.

Compression

Samples were sliced into 12 mm thick disks and compressed to 50% strain on a TA.XT2 Texture Analyzer (Texture Technologies, Scarsdale, NY, USA) interfaced with a Zenith computer. The compression speed was 0.2 mm/s and the force–dimension data were obtained at a rate of 12.5 points/s. Three caliper measurements were taken of each specimen diameter and averaged so that the force readings could subsequently be converted to stress units. The average level of stress in the central portion of the stress–strain functions (between 1 and 5 mm deformation) was computed by averaging all the data points in this region. The resulting average was taken as a measure of compressive resistance, a treatment previously employed by Barrett and Peleg (1992) and Barrett *et al.* (1994). Dry samples and samples containing 12% moisture were compressed.

RESULTS AND DISCUSSION

Effects on sucrose and extrusion variables

At each extrusion moisture level studied, increasing sucrose content reduced both specific mechanical energy (SME) and die pressure (Fig. 1). This effect was most pronounced in the high extrusion moisture extrudates, in which the highest sucrose content reduced SME by almost 30%. The negative effect of increasing moisture on SME and die pressure is well documented (Tayeb *et al.*, 1992; Donald *et al.*, 1993). In our experiments, the SME curve for the 20% moisture batch is substantially lower than that for the 15% moisture batch (by approximately 40%). The negative effect of sucrose on

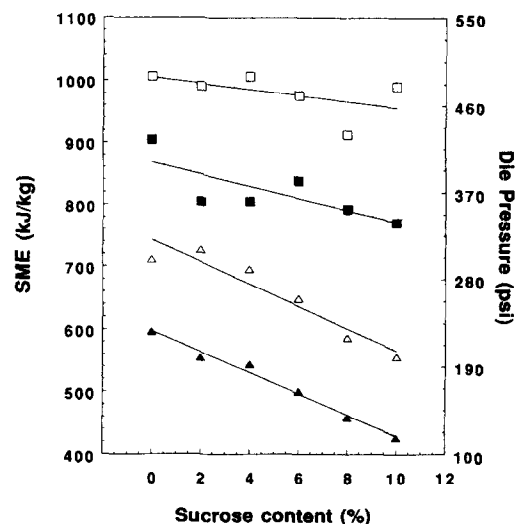


Fig. 1. Effect of sucrose content on specific mechanical energy (■ 15%, ▲ 20% extrusion moisture) and die pressure (□ 15%, △ 20% extrusion moisture).

SME, particularly in the high moisture samples, could possibly be ascribed to hindered starch-starch interactions due to sucrose, as well as to the replacement of a fraction of the starch-containing material by sucrose. These two effects predictably would lower the melt viscosity, which in turn would lead to lower SME and die pressure values. A decrease in die pressure is reported to reduce the driving force for expansion (Donald *et al.*, 1993). The influence of these two competing effects on the structure of extrudates is discussed below.

Effects of sucrose on structure

For extrudates produced at either moisture level studied, high levels of sucrose reduce the mean cell area size and increase the bulk density (Fig. 2). However, in samples produced at 15% moisture content, low levels of sucrose cause only negligible structural changes. In these products, increases in density and reductions in

mean cell size occur at sucrose contents of 8% or higher. By contrast, in samples produced at 20% moisture content, sucrose progressively reduces cell size and increases density, starting at 2% concentration. In both batches, sucrose eventually inhibits the ability of the samples to expand and/or facilitates collapse of the structure, possibly by reducing melt viscosity and elasticity by previously described mechanisms (i.e., Launay & Lisch, 1983). However, for the lower extrusion moisture (higher melt viscosity) products, this effect was moderate and evident only at the highest sucrose levels. By contrast, in the high extrusion moisture samples in which viscosity was already reduced by increased water, the effect was comparatively pronounced and evident even at 2% sucrose levels. Similar effects of sucrose on the structure of wheat flour extrudates have been reported by Ryu *et al.* (1993) at 5 and 10% sucrose levels. Ryu and coworkers observed that sucrose significantly reduced the volumetric expansion of extrudates at a 10% sucrose concentration level, while the extrudate bulk density and the number of cells per unit area increased with increasing sucrose. An increase in the number of cells per unit area, accompanied by a decrease in the sectional expansion index, correlates well with our observation of a reduction in cell size as the sucrose content increases.

Effects of sucrose on thermal properties

Dynamic mechanical spectrometry (DMS)

DMS spectra of 17% moisture content samples reveal pronounced $\tan \delta$ peaks and 1–2 decade reductions in modulus values. A representative DMS scan is shown in Fig. 3. The dependence of the average $\tan \delta$ peak temperature at 1 Hz on sucrose content for low and high extrusion moisture samples is shown in Fig. 4. Note that for both extrudate batches, the $\tan \delta$ peak temperature decreases by at least 20°C between 0 and 10% sucrose, indicating significant plasticization by this additive. Further note that, the reduction in the $\tan \delta$ peak temperature is slightly less pronounced for samples extruded at higher moisture content. However, the plasticizing effects of sucrose are even more notable here given that in the high extrusion moisture system sucrose progressively reduces SME, an effect that (neglecting plasticization) could predictably increase T_g .

Separate DMS tests of pure corn meal samples from either batch showed negligible changes in sample moisture content throughout the temperature range reported for the $\tan \delta$ peak temperatures. However, when subjected to higher temperatures (> 50°C) with the necessary longer hold times, the test samples did dry significantly. Consequently, any high temperature DMS transitions of water-containing products have inherent uncertainty about the exact moisture content of the sample at the transition temperature. For this reason, the thermal properties of lower moisture products, which would have

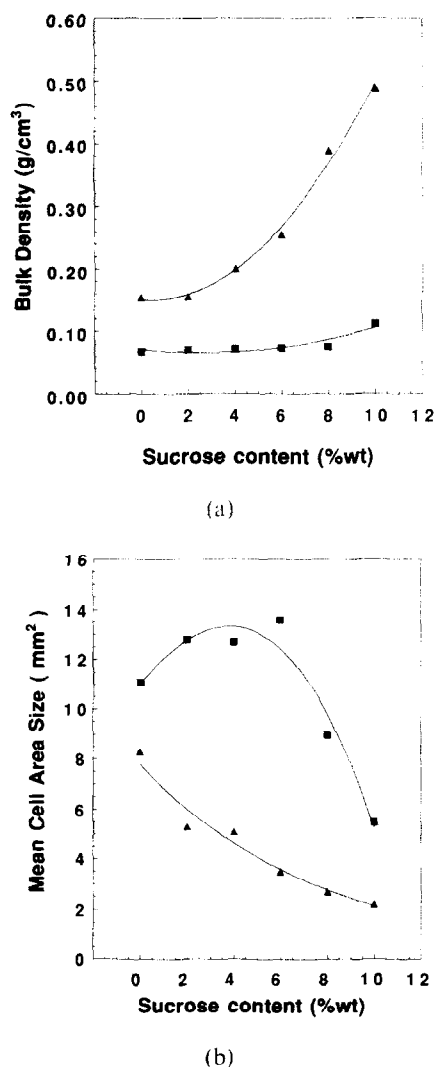


Fig. 2. Effect of sucrose content on extrudate (a) bulk density; and (b) mean cell area (■ 15%, ▲ 20% extrusion moisture). All samples are dried.

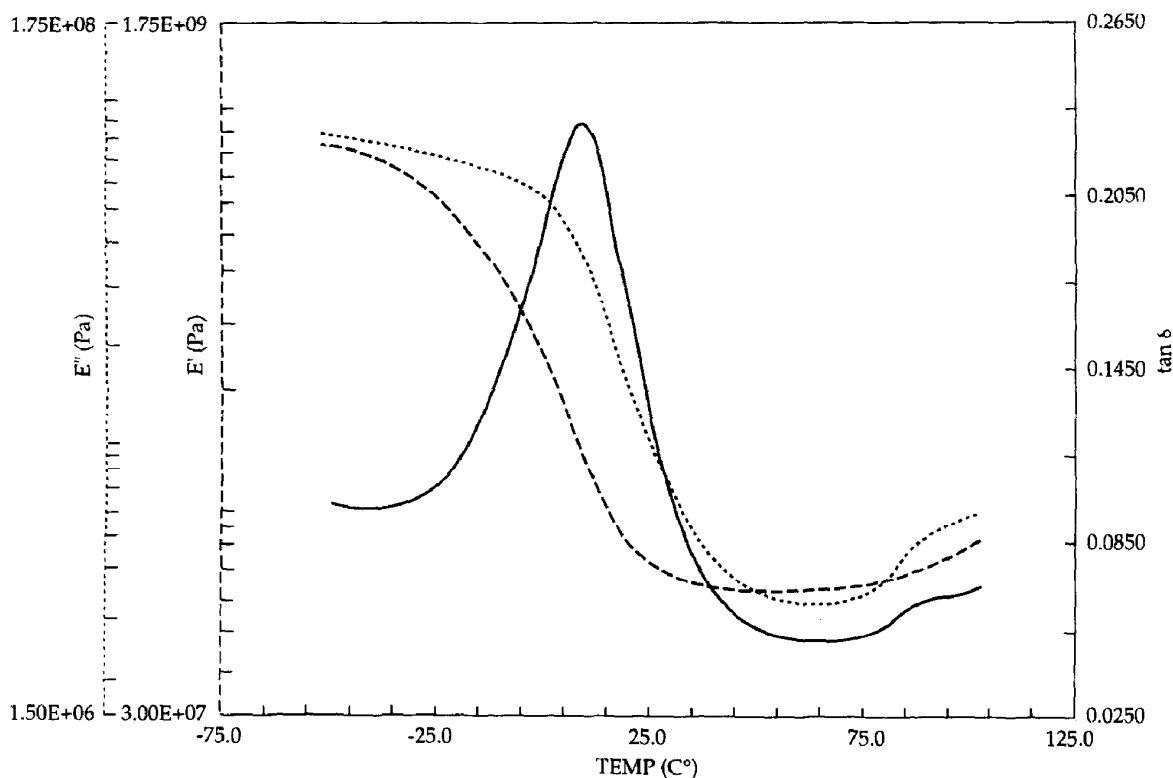


Fig. 3. Representative DMS scan showing $\tan \delta$ peak (17% moisture content sample).

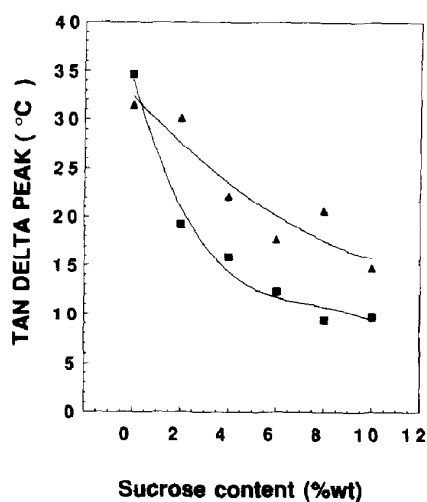


Fig. 4. Effect of sucrose content on DMS $\tan \delta$ peak temperature at 1 Hz for 17% moisture content samples (■ 15%, ▲ 20% extrusion moisture).

higher glass transition temperatures, were analyzed in sealed cells under constant pressure by DSC.

Differential scanning calorimetry

Pressure-variable DSC allowed us to run samples adiabatically at a predetermined, controlled pressure over a selected temperature range. The capability to conduct runs at a defined and controlled pressure is important and contrasts with conventional DSC instruments, in

which the sample is sealed in a cell, thereby experiencing an undefined increase in pressure with increasing temperature (a pressure-cooker effect).

DSC thermograms of dry samples exhibit no discernible effect of sucrose on the thermal properties of dry corn extrudates over the temperature range (-10 to 210°C) examined. However, DSC measurements on 13% moisture samples reveal distinct reductions in the glass transition temperatures with increasing sugar content. As shown in Fig. 5, in the first scan, a typical DSC curve for a moist, pure corn extrudate displays both an irreversible endothermic peak and a glass transition over approximately the same temperature range (30 – 50°C). Upon immediate rescanning after cooling, only the glass-to-rubber transition is observed. Therefore, data are reported for the second scan of each sample, in which glass transitions are unobscured. The low temperature endothermic event ($\sim 50^{\circ}\text{C}$) noted here also has been observed by Kalichevsky *et al.* (1992) for amorphous amylopectin containing 15% water during first scans and after storage. A similar first scan low temperature endotherm also has been reported as a common feature for polysaccharide by Gidley *et al.* (1993) and in extruded starch by Donald *et al.* (1993).

It is important to note that the reduction in T_g values induced by sucrose, as detected by DSC, parallels that observed by DMS measurements (Fig. 6). Furthermore, in both cases, plasticization was most pronounced for the higher SME samples and at sucrose contents less than 6% wt.

Figure 7 shows the effect of sucrose on the specific heat capacity of dry and moist corn extrudates at 30°C. Note that the specific heat capacity of extrudates increases as a function of the sucrose content. This effect is more pronounced in the moist samples. The discontinuity in the specific heat capacity data above 6% sucrose content for moist samples is consistent with a glass transition occurring for corn extrudate (10% sugar and 13% moisture) at about 30°C (Fig. 6). Similar discontinuities have been observed for the specific heat capacity of starch as a function of sucrose in the presence of water at 25°C (Sopade & Le Grys, 1991*b*). In the absence of sucrose, Noel and Ring (1992) reported about 20% moisture is required to observe a discontinuity in the heat capacity at 25°C.

Effects of sucrose on compressibility

Figure 8 in which average compressive stress is plotted vs sucrose content, shows the influence of sucrose on the compression of dry and equilibrated samples. In each figure, samples containing 0–6% sucrose are shown. These samples were selected from the low extrusion moisture batch since they are structurally similar, with fairly equivalent densities and mean cell sizes. In the high extrusion moisture batch, the 8 and 10% sucrose samples were only slightly expanded, so that the dried specimens cracked abruptly during compression and a complete test to 50% strain was not possible. For all the extrudates, the stress–strain data from compression tests were consistent with deformation behavior for cellular solids in general. Specifically, these stress–strain relationships contained linear elastic and subsequent non-increasing stress regions, as described by Gibson and Ashby (1988).

For each sample in either batch, the compressive resistance of equilibrated samples was higher than that of dry samples. We suggest that this phenomenon most likely is due to the dry and equilibrated samples failing by entirely different mechanisms. Compression of dry extrudates caused failure by brittle fracture, which involves the progressive breakage of cell wall components, while the most 'plastic' samples probably failed by bending instead of fracturing of cell walls. In fact, differences in compression behavior were manifest in the appearance of the stress–strain curves, which were extremely jagged for dry extrudates but fairly smooth for moist samples. However, further increases in the moisture content of the already plastic (and non-fracturable) extrudates could be expected to reduce the compressive resistance of samples since water would progressively plasticize the structures. Similar observations for the influence of moisture content on the mechanical properties of extrudates have been reported by Barrett (1991), in measurements of compressive resistance; Attenburrow and Davies (1993), in measurements of fracture stress and fracture strain; by Loh and

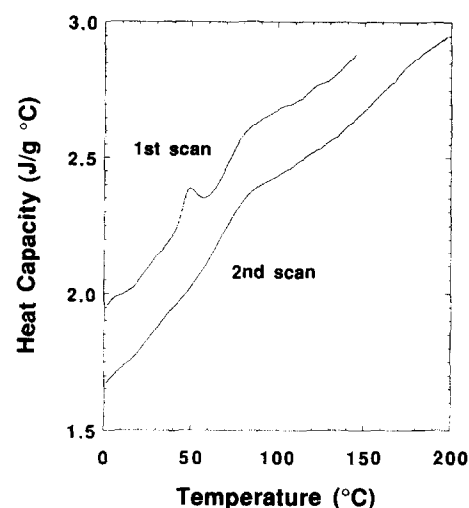


Fig. 5. Representative DSC scans of corn extrudate 3 (Batch 2, 13% moisture content, 0% sucrose). For the sake of clarity, the first scan is displaced by 0.3 J/g °C.

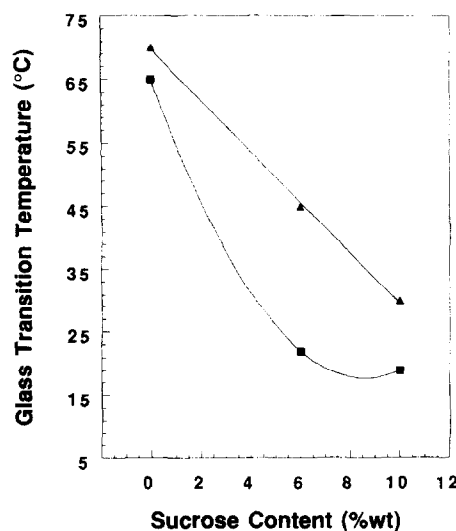


Fig. 6. Effect of sucrose content on glass transition temperature for 13% moisture content samples determined by DSC (■ 15%, ▲ 20% extrusion moisture).

Mannell (1990), in measurements of maximum shear force; and by Halek *et al.* (1988), in measurements of compressive strength and stiffness.

To assess the effects of sucrose on compressive resistance, relationships between physical structure and strength also must be considered. Such relationships have been reported by Barret and Peleg (1992) and Barrett *et al.* (1994), who observed increasing compressive resistance with either increasing bulk density or decreasing cell size. Similarly, for low moisture extrusion samples containing 0, 2, 4 and 6% sucrose, neither bulk density nor mean cell size varied significantly (Fig. 2) and the average compressive stress for dry specimens of these samples was correspondingly similar (Fig. 8a). However, the average compressive stress

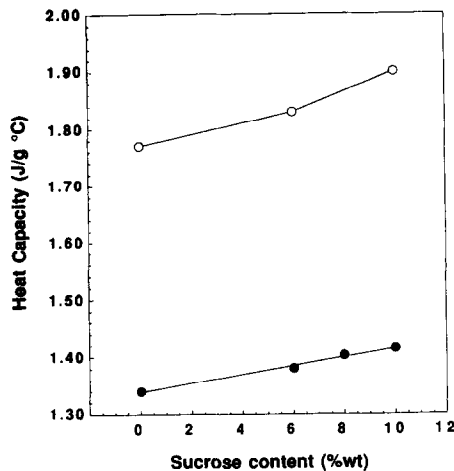


Fig. 7. Effect of sucrose content on the specific heat at 30°C of dry (●) and 13% moisture content (○) samples. Batch 2.

declined progressively and markedly with sucrose content after equilibration to 12% moisture. An analogous comparison can be made for high extrusion moisture samples in which the structure varied significantly with sucrose content. In these samples, increasing sucrose content increases the density and reduces the mean cell size (Fig. 2), therefore, resulting in a progressive increase in compressive resistance for the dry specimens (Fig. 8b). However, equilibration to 12% moisture causes the average compressive stress to become almost equal despite these structural differences. In both sample batches, sucrose, in the presence of moderate amounts of moisture, reduces compressive resistance, an effect that is consistent with plasticization as determined by thermal analysis.

CONCLUSIONS

Sucrose has pronounced effects on the processability and structure of corn extrudates. Sucrose-induced reductions in extrusion SME and in product expansion probably reflect the reductions in melt viscosity caused by replacement of starch-based material with sucrose. Such an effect would reduce shear conditions in the extruder and inhibit product expansion. These consequences are more pronounced in high moisture content formulations in which melt viscosity is relatively low, even prior to addition of sucrose. Combinations of sucrose and extrusion moisture should, therefore, be optimized in order to adjust product structure, physical properties, and consequently, sensory properties. For example, extrudate cell structure has been found to influence strongly fracturability and sensory texture (Barrett *et al.*, 1994).

In the studies reported here, it was found that sucrose, in the presence of moderate levels of moisture, significantly changes the thermal properties of the

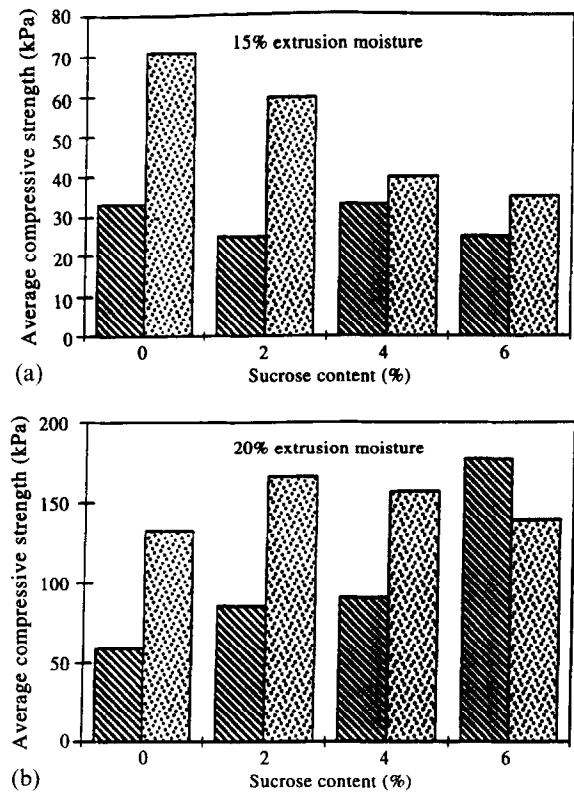


Fig. 8. Effect of sucrose content on average compressive stress: (a) 15% extrusion moisture; and (b) 20% extrusion moisture. □ dry, ▨ 12% moisture content samples.

extrudates; plasticization is evidenced by DMS-measured reductions in $\tan \delta$ peak temperature and by DSC-measured reductions in the glass transition temperature. Further, the data reveals that sucrose correspondingly produces significant textural plasticization in the samples, as determined by lowered resistance to compression. However, significant differences were observed in the moisture sorption characteristics of the two batches, regardless of sucrose content. In general, Batch 2 samples, produced under higher moisture and lower SME conditions, tended to absorb moisture much more slowly, perhaps due to thickened cell walls in the more dense structures or to differences in the degree of starch breakdown. For a given batch, sucrose content had no significant effect on moisture sorption. While the purpose of this study was to equilibrate samples to equivalent moisture contents to test physical properties, the dynamics of moisture sorption by themselves represent important functional behavior, with possible relationship to structure and/or shear conditions, and therefore, merits further study.

While sucrose acts as a plasticizer at moderate moisture levels, obvious plasticization was not observed in very dry systems. Average compressive stress in dried samples primarily varied with structural properties such as density and mean cell size, in accordance with recently published work (Barrett & Peleg, 1992; Barrett *et al.*, 1994). Furthermore, both DMS and DSC analy-

ses of dry samples revealed indistinct transitions occurring over a wide temperature range. However, the fact that plasticization is pronounced in the presence of moisture has practical significance: commercially available extruded breakfast cereals contain high levels of sucrose and also typically are served in the presence of milk. The concentration of sucrose in such products may well influence the ultimate texture of the cereal as it is consumed. Other analogous systems exist in which sucrose-containing extrudates potentially could be exposed to moisture; for example, the interfacing of extruded cracker-type products with higher moisture items such as cheese, jam, or savory spreads. Consideration should be given to such effects, particularly since sucrose can significantly alter thermal and mechanical properties at concentrations as low as 2%.

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