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RHEOLOGY AND THERMAL PROPERTIES OF SYRUPY COMPOSITES OF SACCHARIDES AND MILKFAT

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ABSTRACT

Viscous syrups with between 20 and 34% moisture were made from homogenized slurries of sugars, nonfat dry milk, anhydrous butteroil and emulsifiers. Homogenized mixtures were condensed under vacuum at 45C for 35 min, to form ordered structures with a syrup-like consistency. Complex viscosity determined through dynamic spectrometry increased with increasing fat content. At low frequency, high fructose corn syrup and maltose-based syrups showed more solid-like behavior ($G' > G''$) while the sucrose-based syrup was more liquid ($G'' > G'$). Thermal analyses showed all composites to have a uniform melting range (95–140C), whereas the original components melted in the range 20–200C. The rheological and thermal properties suggest that these syrups could be useful confectionery ingredients.

INTRODUCTION

Milk components are important ingredients in confectionery products where their functional properties are employed to enhance flavor, texture, color and nutritional qualities (Edwards 1984; Gingrich and Dimicki 1979). Sugar syrups are routinely employed as ingredients in products such as toffees, caramels, fondants and fudges, as well as in sweet baked products and other foods. Milkfat is also a significant functional component in candy and confectionery foods, im-

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proving smoothness and consistency (Campbell and Pavlasek 1987). With growing world-wide surpluses of milkfat (USDA 1991), new uses for this valuable commodity need to be sought; for example, a composite of milkfat and saccharides may be an intermediate product with unique functional properties in microwave foods. Such a product could find wide applications in the pastry industry since confectionery syrups are routinely made from hydrolyzed sugars and hard fats-butter (Chiu and Kosikowski 1985; Fullbrook 1983; Babayan 1978).

Rheological properties of confectionery products chiefly determine their acceptance. Sugar based confectionery products (candies, fondants, caramels, etc.) routinely contain large amounts of fats and sugars. The fat acts as a lubricant that improves viscous flow, texture and flavor (Minifie 1989). Emulsified milkfat has been reported to enhance gelation and network formation through crosslinking between the fat globules and casein (Xiong *et al.* 1991). Flow behavior and consistency of starch-fatty acid systems are known to decrease with increasing triacylglycerol chainlength and concentration (Raphaelides 1992). The physical state of carbohydrates is altered in the vicinity of triacylglycerols, affecting the rheological and chemical properties, resulting in measurable changes in associated gelatinization energy (Kim and Walker 1992; Buck and Walker 1988; Niediek 1988). Specific properties of sugar syrup composites containing large amounts of hard fats have been reviewed (Jeffery 1993). Traditionally, models describing flow properties of syrups are of the "power law" type (McMaster *et al.* 1988). In this study, we describe the rheological and thermal properties of saccharoglyceride (SG) composites, the objective being to develop food grade syrups from milk fat and sugars for microwave applications, and as cross-linked syrups for extruded snacks.

MATERIALS AND METHODS

Viscous syrups were made by vacuum evaporation from homogenized formulations containing sugars (high fructose corn syrup, maltose or sucrose), anhydrous butteroil and emulsifiers. Slurries were formulated (17 MPa, 63C, 40% total solids) to contain 20, 30 or 40% butteroil (Land O'Lakes, Minneapolis, MN); emulsifying agent (mono- and di-glycerides, American Ingredients Co., Kansas City, MO), added at 5% of the total weight of butteroil; nonfat dry milk (Maryland and Virginia Milk Producers Association, Laurel, MD), added at 4% of the total solids; and the remainder, sucrose (Domino Sugar Corp, NY), maltose (Penta Manufacturing, Livingston, NJ) or high fructose corn syrup (CornSweet™ 42) (ADM Corn Processing, Decatur, IL). These formulations are similar to those that were spray dried to produce encapsulated powders (Onwulata *et al.* 1994).

Preparation Method

The selected sugar and nonfat dry milk were dry blended, then dissolved in water at 50C to about 45% total solids. This mixture was combined with milkfat and the emulsifier which had previously been blended and heated to 50C. The final mixture (40% total solids) was slowly brought to 63C with constant stirring and homogenized at 17.2 MPa (Manton-Gaulin Triplex homogenizer Model 100 DJF3 855X, APV Gaulin, Inc., Everett, MA). Syrup composites were made by heating and evaporating the slurry at 45C under vacuum (5.2 MPa) in a rotary evaporator (Brinkmann Instruments, Inc., Westbury, NY) for 35 min within 2 days of homogenization. Viscosities of the SG composites were measured within 4 h of completion of evaporation at ambient temperatures. Samples were replicated three times in a completely randomized order.

Moisture analysis was carried out by an AOAC procedure (AOAC 1984).

Dynamic Rheometry

A frequency test was made to evaluate behavior of the SG composites in terms of viscous stability and dynamic viscous response to shear and temperature at oscillation frequency (ω) ranges from 0.01 to 100 rad/s with fixed amplitude. A strain test was made both to evaluate product behavior at increasing strain and to identify the range of linear viscoelasticity. The tests were performed at 25C with a Rheometrics RDA-700 Dynamic Analyzer (Rheometrics Inc, Piscataway, NJ) using a 0–200 g-cm torque transducer equipped with 2.0 cm radius parallel plates with a height of 0.2 cm. Temperature sweeps of the materials ranged from 25 to 70C, ramped at 5C steps with frequency set at 1.0 rad/s. The mechanics of following change in viscoelastic properties as a function of time and temperature were determined through dynamic shear measurement that gives the shear storage modulus (G') and loss modulus (G''). Complex viscosity (η^*) was determined from the frequency sweep at 1.0 rad/s. The relationship to dynamic moduli is as follows:

$$(G^*)^2 = (G')^2 + (G'')^2 \quad (1)$$

with $\eta^* = G^*/\omega$.

The ratio of energy loss to energy saved is given by the loss tangent (δ) which represents the relative elasticity within a network and the dynamics of network formation and breakdown (Aguilera *et al.* 1993):

$$\text{Tan } \delta = G''/G' \quad (2)$$

Three replicates of each syrup were each analyzed twice.

Thermal Analysis

Thermal properties of the SG components were analyzed with a Perkin-Elmer Differential Scanning Calorimeter, Model DSC-7 (Perkin-Elmer Corp., Norwalk, CT), flushed with liquid nitrogen at 20 psi. Ten mg \pm 1 mg, were weighed into aluminum pans and hermetically sealed. The heating rate was 10C/min from -90°C to 210°C after holding at -110°C for one min. Melting patterns, melting regions and peak melting points were recorded. Glass transition points were determined from inflection points on the thermograms. Each syrup, replicated 3 times, was run twice.

Microscopy

Samples were prepared for microscopy as follows: aliquots (0.2 g) of diluted slurries of the SG composites were mixed rapidly with 0.5 mL volumes of liquified agarose (2%) at 40°C in 1.5 mL plastic tubes, gelled on ice and stored at 4°C . Mixed samples were excised from the tubes, diced, and immersed in 2 mL volumes of 2% osmium tetroxide in 0.1 M imidazole hydrochloride (pH 6.8) for 2 h. Following fixation, samples were washed in 0.1 M imidazole buffer, and dehydrated in a graded series of ethanol solutions before embedding in an epoxy resin mixture, cured by heating at 55°C for 48 h. Embedded samples were sectioned at $0.25\ \mu\text{m}$ thickness and stained with methylene blue for optical microscopy. Optical magnification was calibrated with a slide micrometer and photomicrographs prepared (magnification $1300\times$) with an Olympus BH2 phase contrast microscope (Olympus Corp., Lake Success, NY).

For transmission electron microscopy (TEM), embedded samples were sectioned at 50 nm thickness, stained with a solution of 2% uranyl acetate and a lead citrate solution. TEM was done using a Philips CM12 electron microscope (Philips Electronics Instruments Co., Mahwah, NJ) in the transmission mode. Photographic images were made at an instrumental magnification of $72,000\times$.

RESULTS AND DISCUSSION

Optical (Fig. 1A and 1B) and TEM (Fig. 1C) photomicrographs of the SG composite containing 20% butteroil are presented in Fig. 1; this figure is representative of all the composites studied. The emulsion at 40% total solids prior to evaporation shows unstructured fat/protein/saccharose bodies dispersed in the water phase (Fig. 1A). As the moisture is evaporated during the cooking pro-

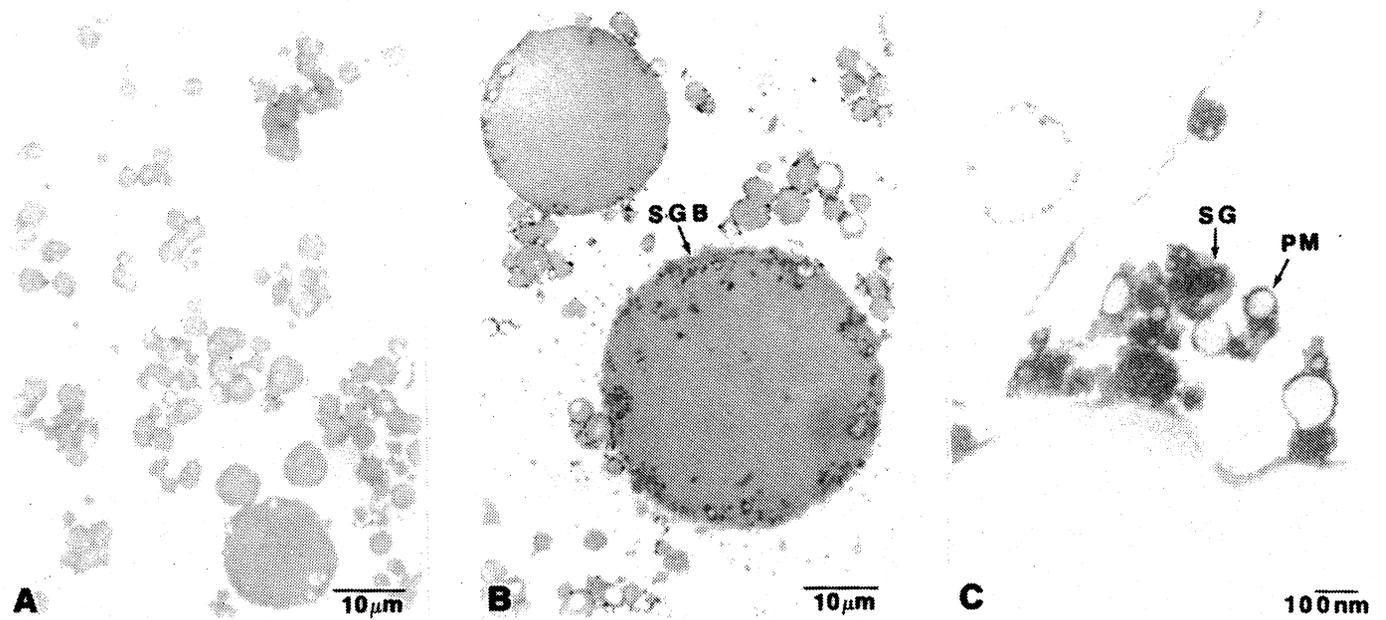


FIG. 1. OPTICAL AND TRANSMISSION ELECTRON PHOTOMICROGRAPHS OF 20% MILKFAT-CONTAINING HIGH FRUCTOSE CORN SYRUP COMPOSITE

1A. 40% total solids slurry before evaporation, showing unassociated protein/saccharose/fat bodies. (Magnification: 1300×).
 1B. Composite after evaporation; Arrow: Protein/saccharide matrix. (Magnification 1300×). 1C. Transmission electron microscope image of sugar-butteroil network; Pm: protein film material, and SGB: Small SB bodies. (Magnification: 72,000×).

cess, the components coalesce and form an ordered structure (Fig. 1B). Structures within the matrix of the SG composite (Fig. 1C) show the encasement of both fat globules and saccharides by a thin protein film. Matrix formation and growth occur through the coalescence of smaller SG bodies (SGB) that are engulfed by the larger particles. The process of structure formation was similar for all composites. Structure formation in confectionery, particularly caramels, occurs during the cooking process, when breakdown of protein membranes and deformation of fat globules takes place (McMaster *et al.* 1988). It is believed that in the presence of emulsifying agents (including protein from nonfat dry milk), an ordered structure is formed with polysaccharides. The formation of an ordered structure is improved by Maillard-type reactions leading to a lowering of product viscosity at low temperatures (Yost and Kinsella 1993; Kato *et al.* 1993). Our SG composites had low viscosities, possibly from the formation of an ordered structure (Fig. 1B). Further removal of moisture increases linking of structures that are separate at higher moisture and viscosity; this phenomenon is characteristic of high temperature boiled caramels (McMaster *et al.* 1988).

The sucrose/buteroil composites were similar rheologically to high fructose corn syrup/buteroil at the three fat levels studied in terms of complex viscosity (Fig. 2). Complex viscosity (η^*) increased with increased fat content with a sharp increase occurring between 30 and 40% fat. Maltose/buteroil composites were

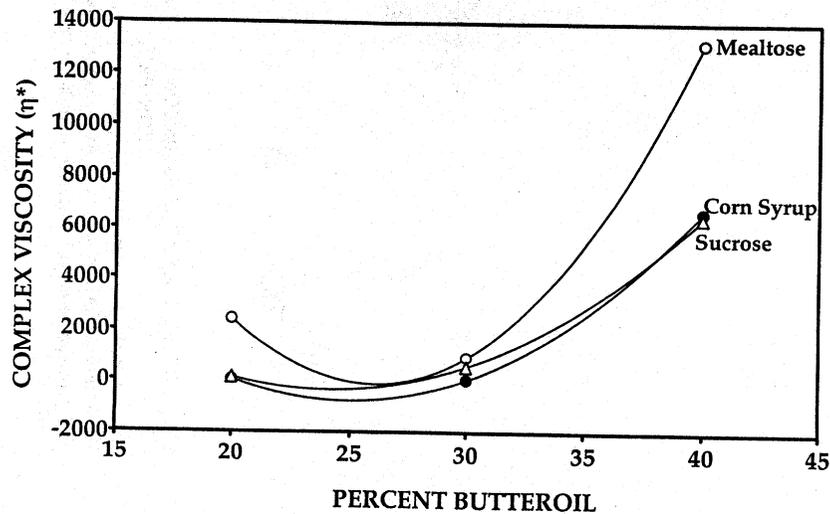


FIG. 2. PLOT OF COMPLEX VISCOSITY (η^*) VERSUS PERCENT BUTTEROIL OF SACCHARO-GLYCERIDE COMPOSITES

● = sucrose; ○ = maltose; ▽ = high fructose corn syrup.

more viscous than sucrose and high fructose corn syrup based composites, especially at 40% fat.

The viscous responses of the three SG composites with 30% milkfat to constrained oscillatory frequency are presented in Fig. 3; similar low shear rate response patterns were observed for other composites. High fructose corn syrup and maltose composites (C30 and M30) exhibited more solid-like behavior ($G' > G''$) at low frequency ($\omega < 1$ rad/s), while the sucrose composite (S30) was more liquid-like ($G'' > G'$) in behavior. Ultimately, all composites reached an elastic plateau where ($G' = G''$) at higher frequency ($\omega > 10$ rad/s), which is characteristic of an entangled network (Lopes da Silva *et al.* 1993). Complex viscosity (η^*) decreased logarithmically with increasing frequency, suggesting power law behavior; this behavior also agrees with that of a viscoelastic solid such as a lightly crosslinked amorphous polymer (Ferry 1980) and confirms the pseudoplasticity of our composites. These flow patterns are consistent with flow behavior reported for starchy gels with an ordered structure (Lopes da Silva *et al.* 1993).

When some of the SG composites are adjusted to the same moisture content (30%), the patterns of flow change (Fig. 4). In contrast to the previous figure, the diluted samples of high fructose corn syrup and maltose composites became more fluid, with the loss modulus being greater than the storage modulus ($G'' > G'$); in contrast, the sucrose composite became more viscous with the storage modulus being greater than the loss modulus ($G' > G''$). Complex viscosity again decreased with increasing frequency but not as rapidly as with the undiluted samples. The diluted samples were an order of magnitude lower than the undiluted samples in terms of complex viscosity. The dilution suggests loss of ordered structure (G') for the high fructose corn syrup and maltose composites.

Dynamic changes in phase angle with temperature for the composites are presented in Fig. 5. The ratio of the loss (G'') and storage (G') moduli gives the relative contribution of each to the viscoelasticity (stress/strain). Since phase angle changes from $\delta = 0$ for a completely elastic component to $\delta = 90$ for a completely viscous phase (Ferry 1980), the composites are mostly elastic fluids with low ($\delta < 2$) phase angles. In contrast to the other samples, the high fructose corn syrup composite with 20% milkfat showed a sharply increased $\tan \delta$ with increasing temperature, reflecting a large increase in the loss modulus, while the 20% maltose composite maintained a more elastic behavior. The behavior observed may be due to the composition of the 42 DE high fructose corn syrup, which contains a mixture of 42% fructose, 52% dextrose and 6% higher saccharides, since increasing the milkfat content caused little change in the phase angle patterns as shown for sucrose (20 to 40%); moduli ratios were unchanged at temperatures above 50C. Similar patterns were seen with high fructose corn syrup and maltose composites (data not shown).

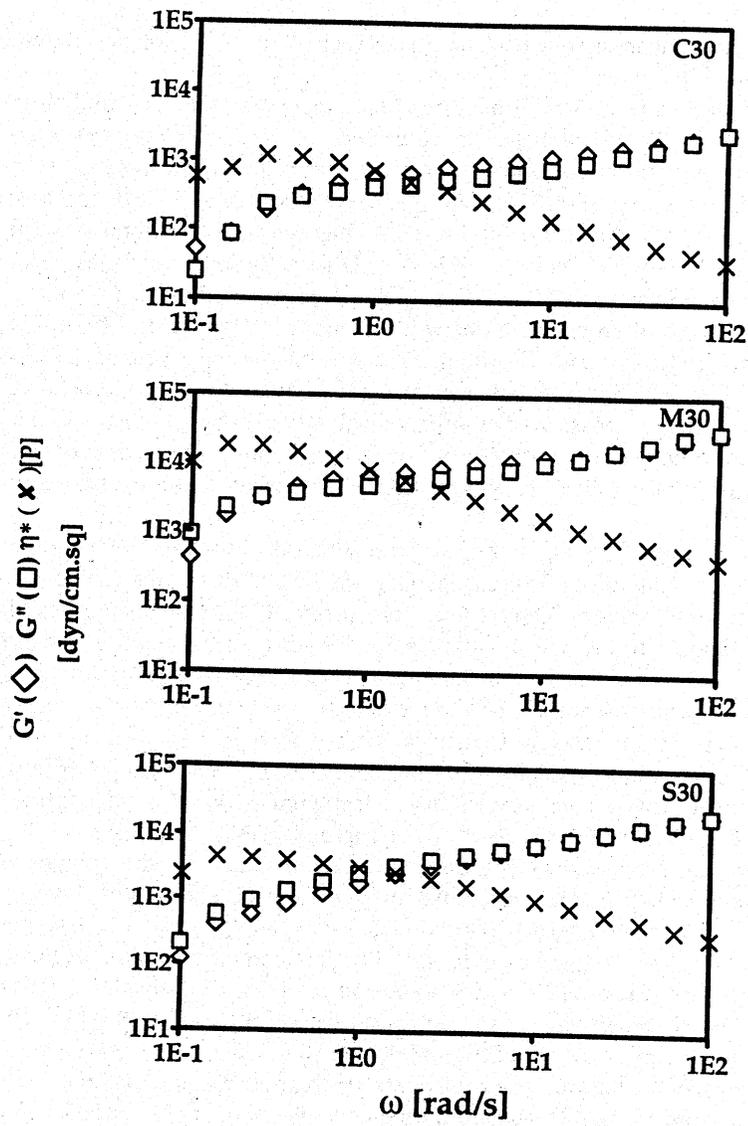


FIG. 3. FREQUENCY SWEEP OF THE DYNAMIC VISCOSITY OF SACCHARO-GLYCERIDE COMPOSITES AT AMBIENT TEMPERATURE
 C30: 30% butteroil encapsulated in high fructose corn syrup; M30; 30% butteroil encapsulated in maltose; and S30: 30% butteroil encapsulated in sucrose. Storage modulus (G'), loss modulus (G'') and complex viscosity (η^*). Frequency 0.1-100 rad/s at 25C.

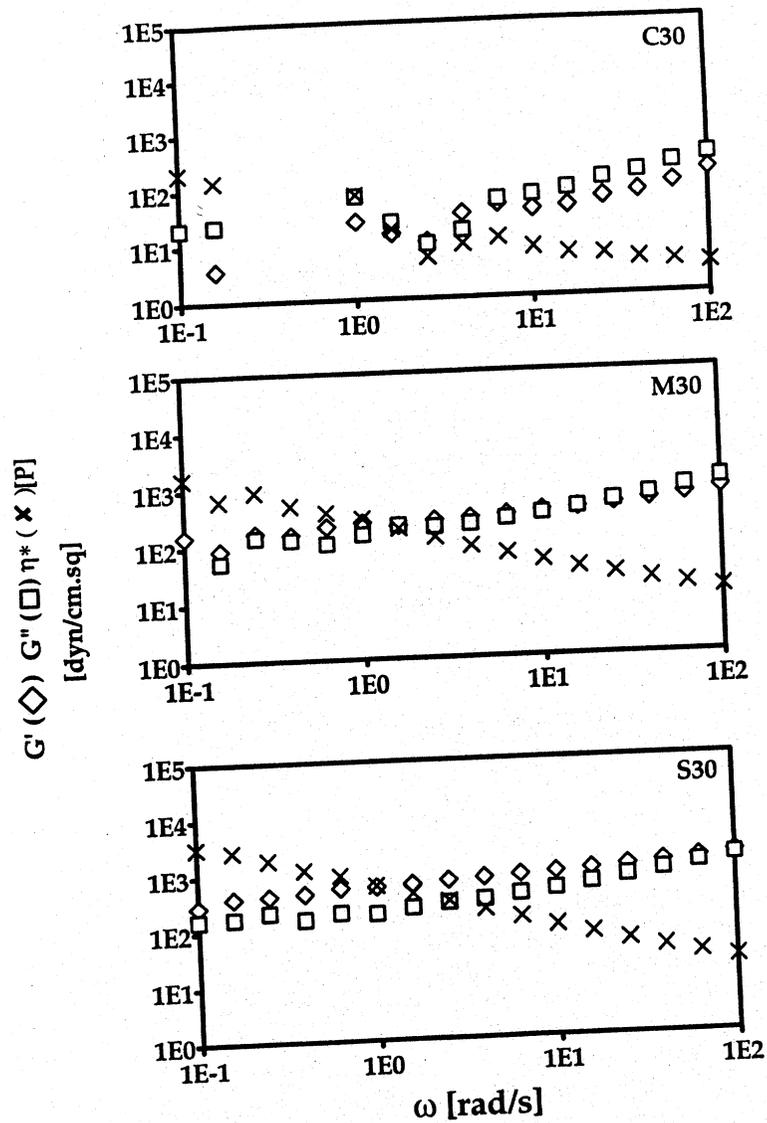


FIG. 4. FREQUENCY SWEEP OF THE DYNAMIC VISCOSITY OF SACCHARO-GLYCERIDE COMPOSITES (ADJUSTED TO 30% MOISTURE) AT AMBIENT TEMPERATURE
 C30: 30% butteroil encapsulated in high fructose corn syrup; M30: 30% butteroil encapsulated in maltose; and S30: 30% butteroil encapsulated in sucrose. Storage modulus (G'), loss modulus (G'') and complex viscosity (η^*). Frequency 0.1–100 rad/s at 25C.

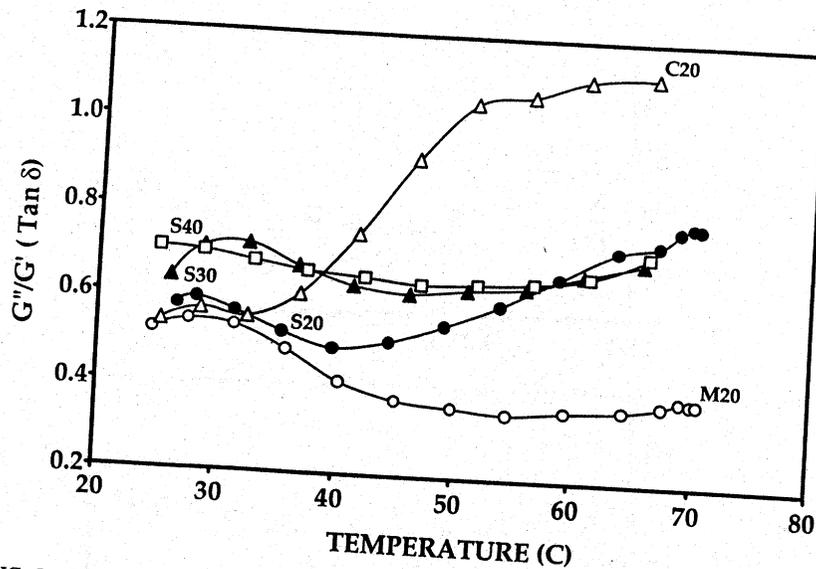


FIG. 5. THE EFFECT OF TEMPERATURE ON TAN δ OF SACCHARO-GLYCERIDE COMPOSITES AT 20% MILKFAT; C20: HIGH FRUCTOSE CORN SYRUP, M20: MALTOS, AND S20: SUCROSE
(Sucrose also shown at 30 and 40% milkfat).

Temperature and complex viscosity (η^*) data of the SG syrups with 20% milkfat were fitted to the following model:

$$\eta^* = A \exp (b/T) \quad (3)$$

where A and b are constants, T is the temperature in deg Kelvin, and η^* = apparent viscosity. b is the constant comprised of the activation energy, E_a (kJ/mol), and R, the gas constant (8.3144 kJ/mol K). The log complex viscosity of the composites plotted against the reciprocal temperature is presented in Fig. 6. The slope of the lines for all SG composites containing 20% butteroil varied among composites reflecting differences in activation energies. The slope of the high fructose corn syrup composite (C20) is clearly different than the slopes of the other two. The activation energies of the flow at different fat levels are in Table 1. Maltose and sucrose showed a nonlinear increase in flow activation energy as butterfat decreased from 40 to 20%, while high fructose corn syrup composites were relatively stable, showing less effect of temperature. Maltose and sucrose showed a minimum activation energy at 30% milkfat content. Generally, as the ratio of saccharide to fat increased, the activation energy of the syrups increased, agreeing with reported increases in activation energy at higher sugar concentrations for fruit juices (Saravacos 1970).

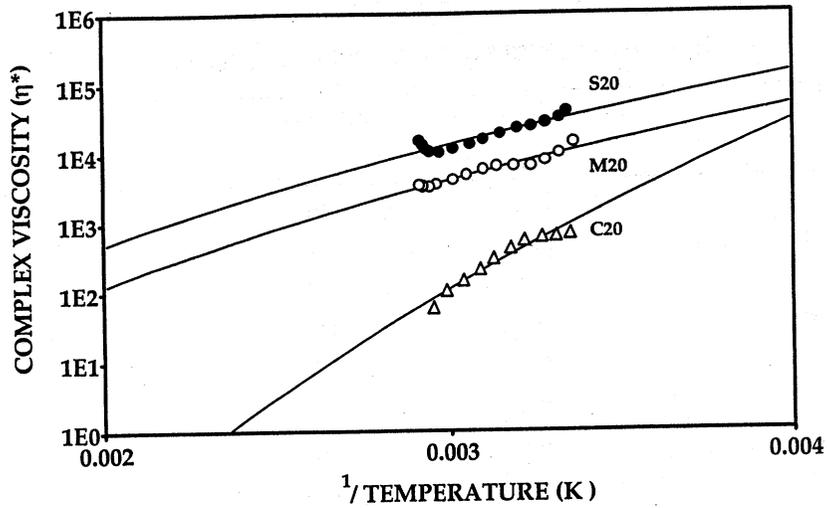


FIG. 6. EFFECT OF INCREASING TEMPERATURE ON SACCHARO-GLYCERIDE COMPOSITES CONTAINING 20% BUTTEROIL
 Arrhenius plot of complex viscosity versus inverse temperature (K). ∇ = high fructose corn syrup; \circ = maltose; and \bullet = sucrose.

TABLE 1.
 APPARENT ACTIVATION ENERGIES AND ARRHENIUS
 EQUATIONS OF COMPOSITES*

Sample	E_a (kJ/mol)	C-Factor	R^2
C40	58660	-17.1	0.99
C30	54390	-14.6	0.86
C20	51230	-13.6	0.94
M40	29123	-3.2	0.95
M30	21624	-0.5	0.92
M20	35294	-8.17	0.99
S40	28251	-3.7	0.99
S30	25198	-2.6	0.97
S20	48706	-11.2	0.99

Complex viscosity ($\log \eta^*$) = $E_a/T - (\text{C-Factor})$ T = temperature (K): *Undiluted composites. C20, C30, C40: 20, 30 or 40% butteroil-high fructose sugar syrup; M20, M30 or M40: 20, 30 or 40% butteroil and maltose; S20, S30, S40: 20, 30 or 40% butteroil and sucrose.

TABLE 2.

SOME PHYSICAL PROPERTIES OF SACCHARO-GLYCERIDE COMPOSITES*

Sample	Moisture	Melting Peak	Melting Range
	%	C	C
C40	33.1	121	114-134
C30	28.3	128	98-143
C20	34.2	131	126-143
M40	25.6	114	109-133
M30	23.5	111	104-138
M20	25.1	125	106-134
S40	22.9	130	96-165
S30	26.5	127	121-135
S20	19.5	130	107-136

*SEM for the composites was 3.28; CV = 0.11. C20, C30, C40: 20, 30 or 40% butteroil and high fructose corn syrup; M20, M30, M40: 20, 30 or 40% butteroil and maltose; S20, S30, S40: 20, 30 or 40% butteroil and sucrose.

Moisture content of the composites varied despite the same processing time and temperature (Table 2). Composite moisture content varied from 20 to 34%. High fructose corn syrup composites were higher in moisture content than maltose or sucrose composites, possibly reflecting the mixture of mono- and higher saccharides. This variance may explain the high fructose corn syrup composites being consistently lower in viscosity than the others.

The association of sugars, proteins and milkfat in forming composites also modified the melting patterns of the sugars. The main melting peaks of the individual components were as follows: butteroil (18, 32C); emulsifier (46C); high fructose corn syrup (120C); maltose (123C) and sucrose (198C). These were essential in identifying shifts and patterns on the thermograms of the composites. Typical differential scanning calorimetric thermograms for the three 30% milkfat-containing composites with the transition zones of the saccharide components are presented in Fig. 7. The thermal profiles show one broad melting zone for each composite. Melting peaks were integrated within a temperature range (111 to

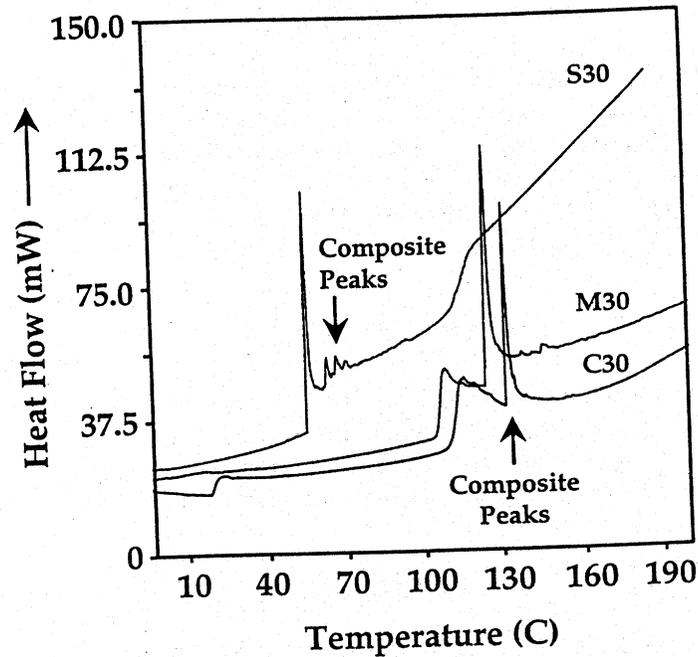


FIG. 7. DSC THERMOGRAMS OF SACCHARO-GLYCERIDE COMPOSITES WITH 30% BUTTEROIL; C30: HIGH FRUCTOSE CORN SYRUP, M30: MALTOSE AND S30: SUCROSE

149C), which does not correspond to any particular peak of the individual constituents. This is indicative of the formation of structured bodies. In our earlier study, we observed a downward shift of melting peaks for the sugars used for encapsulation of milkfat (Onwulata and Holsinger 1995). It is known that complexes formed in the vicinity of saccharides change the melting patterns of the components. Krog *et al.* (1989) reported a dissociation of the amylose-lipid complex at 100–120C, with the heat of dissociation increasing in the presence of monoglycerides; lipid-saccharose complexes also showed high melting peaks. Starch degradation studies have shown a rise in peak temperature in the presence of sugar and emulsifiers (Buck and Walker 1988; Donovan 1977). However, SG composites show lower melting points at higher milkfat content (Table 2).

SG syrup composites could also be made by reconstitution of our spray dried encapsulated powders (Onwulata *et al.* 1994), followed by vacuum evaporation as described above. Composites made in this manner had consistency and flow properties similar to those described above and were not investigated further.

CONCLUSIONS

SG composites showed well cross-linked and stable elastic and functional structures. These defined rheological and thermal properties make SG composites ideal adjuncts for a variety of processed foods. The melting behavior offers an advantage in products demanding sharper and specific melting zones. The elastic, pseudoplastic and Arrhenius-type response to heat makes the SG composites desirable confectionery products.

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