ABSTRACT

Ice cream mixes and frozen ice creams at milk fat levels of 12%, 8%, 6%, 6% plus a protein-based fat replacer, and 6% plus a carbohydrate-based fat replacer were evaluated for viscoelastic properties by dynamic testing with sinusoidal oscillatory tests at various frequencies. The storage modulus \(G'\), loss modulus \(G''\), and \(\tan \delta = (G''/G')\) were calculated for all the treatments to determine changes in the viscous and elastic properties of the mixes and frozen ice creams due to fat content. In ice cream mixes, \(G'\) and \(G''\) exhibited a strong frequency dependence. The \(G''\) was higher than \(G'\) throughout the frequency range (1 to 8 Hz) examined, without any crossover, except for the 12% mix. Elastic properties of the ice cream mixes decreased as fat content decreased. \(\tan \delta\) values indicated that fat replacers did not enhance the elastic properties of the ice cream mixes. In all frozen ice creams, \(G'\) and \(G''\) again showed a frequency dependence throughout the range tested (0.5 to 10 Hz). The amount of fat in ice creams and the degree of fat destabilization affected the elasticity in the frozen product. Even though the ice creams did not have significant elastic properties, when compared as a group the samples with higher fat content had higher elastic properties. The combination of milk proteins and partially coalesced fat provides strength and structure to the ice cream (Goff and Jordan, 1989; Hegenbart, 1996; Marshall and Arbuckle, 1996). Thus, creating and stabilizing the desired structure in low-fat frozen dessert products is difficult, because the coalesced fat fraction is lowered, whereas the protein fraction may be increased. These structural changes can be detected by evaluating physical and sensory properties of the frozen dairy desserts.

Stampanoni Koeferli et al. (1996) found that addition of fat increased the buttery and creamy notes as well as mouth coating in ice creams. Guinard et al. (1996) reported that ice creams with higher fat content had better flavor and texture ratings as determined by a sensory panel. Specter and Setser (1994) reported that replacing milk fat in ice cream with tapioca or potato dextrin increased coarseness and decreased creaminess relative to dairy fat. Schmidt et al. (1993) found that the use of carbohydrate-based fat replacers in reduced-fat ice creams resulted in mixes with higher viscosities. All of the above studies focused on the effects of fat content on the sensory and physical properties of the ice cream. No conclusive studies were found on the influence of fat reduction and use of fat replacers on the dynamic rheological properties of ice cream mixes and frozen ice creams.

INTRODUCTION

Structure development in ice cream often is attributed to the macromolecules present in the ice cream mix—milk fat, protein, and complex carbohydrates.
most foods having rheological properties somewhere in between; thus, they are termed “viscoelastic” (Stanley et al., 1996). Viscoelastic refers to the simultaneous existence of solid and fluid behaviors. Generally, to measure viscoelastic properties an input wave is administered to a sample in the form of sinusoidally varying stress or strain while the corresponding stress or strain is computed. If the stress and strain are in phase, i.e., \( \delta = 0^\circ \), the sample is considered an ideal solid and if the stress and strain are out of phase, i.e., \( \delta = 90^\circ \), the sample is considered an ideal liquid. The in-phase component of the response curve is called the storage modulus (\( G' \), elastic) and the out-of-phase component of the response curve is called the loss modulus (\( G'' \), viscous).

Traditionally, the firmness of frozen dairy products was determined by a penetrometer test, which is a one-dimensional approach. With the advent of rheometers, measuring the complex nature of viscoelasticity has become easier. Goff et al. (1995) measured the rheological properties of ice cream mixes made with and without stabilizers and found that stabilized mixes exhibited significantly greater storage and loss moduli at temperatures of less than \(-8^\circ C\). The unstabilized samples also had greater tan \( \delta \) values at all temperatures than stabilized samples. They reported that as overrun in the frozen product increased from 20 to 60%, tan \( \delta \) decreased significantly, confirming that air contributes to the elasticity of the final product. The way air bubbles interact with fat in the surrounding medium may be a way to manipulate the elasticity component in low-fat ice creams and produce a more desirable product.

The primary objective of this project was to determine the effect of milk fat and fat replacers on the viscoelastic properties of ice cream mixes and frozen ice creams. Fat replacers should have characteristics, like shear thinning, associated with creamy foods (Clark, 1994). Ingredients that develop extensively networked gel structures, so that their structural units cannot move independently, cannot promote creaminess even though they may bind water very effectively (Clark, 1994). Thus, measurement of the viscoelastic behavior of frozen products containing different fat replacers should elucidate structural changes that occur as a result of differences among ingredients present during freezing and frozen storage and lead to a better understanding of frozen dessert systems.

### MATERIALS AND METHODS

#### Ice Cream Mix Preparation

Ice cream mixes were formulated to contain 12%, 8%, 6% milk fat; 6% milk fat plus a protein-based fat replacer (PFR), containing whey proteins, and 6% milk fat plus a carbohydrate-based fat replacer (CFR), containing microcrystalline cellulose and guar gum. Formulas for the mixes are shown in Table 1. During mix preparation, stabilizers (Danisco Ingredients USA, Inc., New Century, KS) and fat replacers (PFR, Dairy-Lo, Cultor, Terre Haute, IN; CFR, Novagel, FMC Corporation, Newark, DE) were blended with cane sugar (Imperial Sugar Company, Sugar Land, TX), 36 DE corn syrup solids (Roquette America, Keokuk, IA), and added to milk ingredients (milk and cream, KSU Dairy Plant, Manhattan, KS; nonfat dry milk, Farmers Cooperative Creamery, McMinnville, OR) at 35°C. Ice cream mixes (35 L) were batch pasteurized at 74°C for 30 min (Groen Manufacturing Company, Chicago, IL) and then homogenized in a single-stage homogenizer (Multi-Flo homogenizer, model 3DD3, Creamery Packaging Company, Chicago, IL) at 10.4 MPa. Homogenized mixes were cooled to 4°C and stored at that temperature for 1 d before tests were performed. The remaining product was frozen.

#### Whipping Ability

Whipping ability was estimated by the overrun method (Phillips et al., 1987) at \(-28^\circ C\) (air temperature) in a walk-in freezer. A 280-ml sample (4°C) was whipped in a mixer (Classic Kitchen Aid, model K45SS, Kitchen Aid, St. Joseph, MI) at speed 10. Overrun values were calculated at 4-min intervals. After weighing, the foam was returned to the mixer for further whipping within 60 s. The test was stopped after overrun values peaked. Whipping ability was expressed as the percentage of overrun and calculated as follows:

\[
\text{% overrun} = \frac{(\text{weight of unit volume of mix} - \text{weight of unit volume foam})}{\text{weight of unit volume of foam}} \times 100.
\]

#### Ice Cream Mix Freezing

After overnight refrigeration at 4°C, the mixes were stirred thoroughly and frozen in a Cherry-Burrell Vogt VS-85 continuous ice cream freezer (Cherry-Burrell Corporation, Cedar Rapids, IA) to \(-5^\circ C\) temperature and 80 ± 2% overrun. The overrun values were calculated as follows:

\[
\text{% overrun} = \frac{(\text{weight of unit volume of mix} - \text{weight of unit volume of ice cream})}{\text{weight of unit volume of ice cream}} \times 100.
\]

When appropriate freezing conditions were reached, ice cream was filled immediately into sample containers—plastic petri dishes of 100 mm diameter (Fisher Scien-
Table 1. Composition (percent weight) of five ice cream mixes.

<table>
<thead>
<tr>
<th>Mix</th>
<th>Fat</th>
<th>Nonfat milk solids</th>
<th>Sugar</th>
<th>Corn syrup solids</th>
<th>Stabilizer</th>
<th>Fat replacer</th>
<th>Total solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>12%</td>
<td>12</td>
<td>11.0</td>
<td>12</td>
<td>4</td>
<td>0.35</td>
<td>***</td>
<td>39.35</td>
</tr>
<tr>
<td>8%</td>
<td>8</td>
<td>15.0</td>
<td>12</td>
<td>4</td>
<td>0.35</td>
<td>***</td>
<td>39.35</td>
</tr>
<tr>
<td>6%</td>
<td>6</td>
<td>15.5</td>
<td>12</td>
<td>4</td>
<td>0.35</td>
<td>***</td>
<td>37.85</td>
</tr>
<tr>
<td>6% + PFR(^1)</td>
<td>6</td>
<td>11.5</td>
<td>12</td>
<td>4</td>
<td>0.35</td>
<td>3.5</td>
<td>37.85</td>
</tr>
<tr>
<td>6% + CFR(^2)</td>
<td>6</td>
<td>11.5</td>
<td>12</td>
<td>4</td>
<td>0.35</td>
<td>2.0</td>
<td>35.85</td>
</tr>
</tbody>
</table>

\(^1\) Protein-based fat replacer, Cultor, Terre Haute, IN.
\(^2\) Carbohydrate-based fat replacer, FMC Corporation, Newark, DE.

Sample Preparation

All ice cream samples for rheological testing were prepared inside the hardening room to avoid any melting. Ice cream was taken out of the petri dishes and smaller samples were prepared. A precooled (−30°C) metallic, hollow cylinder (30 mm i.d.) was used to cut smaller cylindrical samples. These samples were loaded onto the rheometer stage for rheological measurements.

Viscoelastic Measurements

**Ice cream mixes.** Measurements were made using a Rheometer (Bohlin Rheometer, VOR, Bohlin Rheology, Lund, Sweden). For ice cream mixes, a double gap cylindrical measuring system was used with a torque element of 1.43 g-cm. The measurement interval was set at 10 s with a strain of 0.02%. A preliminary strain sweep was performed to determine the linear viscoelastic region of the ice cream mixes. This region is the zone where the stress-to-strain modulus is constant, and generally this is the region where viscoelastic properties are measured. The frequency range used for the mixes was 1 to 8 Hz.

**Frozen ice creams.** For frozen ice creams, parallel plate geometry was used (top plate 30 mm diameter), and plates were allowed to equilibrate (−8°C) with samples before testing to avoid problems associated with melting at the surface of the geometry. The measurements were made at −8°C. A gap of 11.5 mm and a torque bar of 92 g-cm were used in this study. Before making the measurements, we conducted a strain sweep to determine the linear viscoelastic region (0.25% strain). Storage modulus (G\(^\prime\)) and loss modulus (G\(^\prime\)\(^\prime\)) were measured by using sinusoidal oscillatory tests at frequencies of 0.5 to 10 Hz.

Fat Destabilization Measurements

Fat destabilization measurements were done as described by Keeney and Josephson (1958). Turbidity measurements were made with a Klett-Summerson colorimeter (model # 800-3, Klett Manufacturing Co., Inc., New York, NY) set at 540 nm. Fat destabilization was calculated as the percentage of turbidity in frozen ice cream samples compared with turbidity in ice cream mixes. The formula used to determine the percent fat destabilization was:

\[
\text{Turbidity of the ice cream/turbidity of the mix} \times 100.
\]

The smaller the percent fat destabilization values, the greater is the fat destabilization in the ice cream.

Statistical Analysis

The experiment was replicated with three mixes made on different days, and the data were analyzed with SAS software program (SAS Institute, 1996). One-way analyses of variance (ANOVA) were conducted; ice cream mixes or frozen ice creams were the treatments, and means were compared using least significant differences generated by the general linear procedure (PROC GLM). Results were considered significant for \( P < 0.05. \)

RESULTS AND DISCUSSION

**Ice Cream Mixes**

Overrun values compared at 4 min showed that ice cream mix with 6% fat produced significantly higher air incorporation than any other mix, followed by the 12% mix. But at 8 min, there was no statistical difference between the 6 and 12% mixes. The mix with 6% milk fat had a mean overrun of 156% at 8 min (Figure 1). At 12 min, the overrun decreased to 118%. Mixes containing 8 and 12% milk fat followed the same pattern. Overrun for the mix containing PFR peaked in 4 min, decreased at 8 min, and then leveled off. The CFR mix incorporated the least amount of air (82% at 4 min),

but overrun remained unchanged during the next 8 min of whipping.

The CFR mix could have such low overruns at 4 min because of the higher viscous component, which could have prevented air incorporation. High viscous systems do not favor foaming capacity but do favor foam stability (Stanley et al., 1996). Despite the similar fat content, the PFR mix had lower foam overrun values at 4, 8, and 12 min than the 6% milk fat mix (P \leq 0.05). Therefore the protein-based fat replacer did not enhance the whipping ability. Ice cream mixes containing carbohydrate-based fat replacers exhibit a viscous behavior because of the capability for imbibing water, which would increase the viscosity of the system (Cottrell et al., 1979; Schmidt et al., 1993). This increased viscosity could have been the primary reason for decreased whipping abilities. Previous research in whipped creams (Stanley et al., 1996) has shown that use of stabilizers in whipped creams led to increased viscosities which in turn resulted in lower overruns but a more stable foam than foam from creams that were unstabilized.

The G’ and G” of all mixes increased as the frequency increased (Tables 2 and 3). This strong frequency dependence of G’ and G” is an indication of the typical viscoelastic behavior. In the mix containing 12% milk fat, G’ was greater than G” at frequencies 1, 1.5, and 2 Hz but, from 3 Hz onwards, the G” was greater than G’, indicating that a crossover point occurred at a frequency of 2 Hz. This crossover point represents the transition of the mix from a more elastic behavior (G’ < G”) at lower frequencies to that of a more viscous behavior (G” > G’) at higher frequencies and also shows that G” increased at a faster rate than G’. The other four mixes had greater G” than G’ throughout the frequency range tested (1 to 8 Hz) without any crossover points. This frequency dependence of the G’ and G” allows these samples to be categorized as “physical gels” (Telis and Kieckbusch, 1997). The network formed in physical gels is of linkages that are more susceptible to disruption when force is applied. The G’ values of the 12% mix were significantly higher (P \leq 0.05) than G” values of all other mixes at 1 Hz. But, at 4 Hz frequency (Table 4), the G’ values of 12% mix were only different from the 6% + PFR mix. The CFR mix had a higher G” than the PFR mix because of the increase in viscosity brought about by the carbohydrates. This was expected, because carbohydrates are known to be good water binding agents, sometimes better than proteins (Clark, 1994; Akoh, 1998).

Tan δ values (Table 4) increased as milk fat content decreased, verifying that the mixes with higher milk fat content resulted in a system that responded more elastically than the mixes having lower levels of fat content. As can be seen in Table 4, the 8 and 6% mixes were not significantly different from each other but differed from the 12% mix, which indicates that a variation in fat content or fat: protein ratio could alter the elastic properties significantly. The two mixes containing the fat replacers had similar elasticities, but the mix containing CFR had lower elastic properties compared with 6, 8, and 12% mixes. This indicates that as fat is replaced by a complex carbohydrate or additional protein, the viscous component becomes the predominant characteristic.

**Frozen Ice Creams**

The average G’ values of 12% milk fat ice cream were significantly higher than the 6% milk fat ice cream not

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**Table 2.** Mean loss modulus (G”) of ice cream mixes measured at a frequency of 1, 4, and 8 Hz.

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>12% Mix</th>
<th>8% Mix</th>
<th>6% Mix</th>
<th>6% + PFR Mix</th>
<th>6% + CFR Mix</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.11 ± 0.38&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.04 ± 0.08&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.12 ± 0.57&lt;sup&gt;b&lt;/sup&gt;</td>
<td>1.54 ± 0.05&lt;sup&gt;b&lt;/sup&gt;</td>
<td>2.05 ± 0.52&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>4</td>
<td>4.37 ± 0.59&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>4.48 ± 0.11&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>4.65 ± 0.99&lt;sup&gt;b&lt;/sup&gt;</td>
<td>3.65 ± 0.09&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.77 ± 1.13&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>8</td>
<td>5.79 ± 0.75&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>6.02 ± 0.15&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>6.11 ± 1.10&lt;sup&gt;a&lt;/sup&gt;</td>
<td>4.96 ± 0.10&lt;sup&gt;b&lt;/sup&gt;</td>
<td>6.40 ± 1.41&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a,b</sup>Means of three replicates in the same row with same superscripts do not differ significantly (P \leq 0.05).

<sup>1</sup>6% milk fat mix containing the protein-based fat replacer.

<sup>2</sup>6% milk fat mix containing the carbohydrate-based fat replacer.
Table 3. Mean storage modulus (G') of ice cream mixes measured at a frequency of 1, 4, and 8 Hz.

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>12% Mix</th>
<th>8% Mix</th>
<th>6% Mix</th>
<th>6% + PFR Mix</th>
<th>6% + CFR Mix</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.23 ± 0.61(^a)</td>
<td>1.66 ± 0.12(^b)</td>
<td>1.61 ± 0.66(^b)</td>
<td>1.08 ± 0.06(^b)</td>
<td>1.32 ± 0.30(^b)</td>
</tr>
<tr>
<td>4</td>
<td>4.13 ± 0.96(^a)</td>
<td>3.58 ± 0.20(^a)</td>
<td>3.68 ± 1.30(^a)</td>
<td>2.56 ± 0.11(^b)</td>
<td>3.28 ± 0.80(^b)</td>
</tr>
<tr>
<td>8</td>
<td>5.31 ± 1.07(^a)</td>
<td>4.91 ± 0.19(^ab)</td>
<td>5.12 ± 1.58(^a)</td>
<td>3.75 ± 0.13(^b)</td>
<td>4.76 ± 1.10(^ab)</td>
</tr>
</tbody>
</table>

\(^a,b,c\)Means of three replicates in the same row with same superscripts do not differ significantly (P ≤ 0.05).

Table 4. Mean storage modulus (G'), loss modulus (G''), and tan δ values of five ice cream mixes measured at 4 Hz and 4°C.

<table>
<thead>
<tr>
<th>Mix</th>
<th>G' (Pa)</th>
<th>G'' (Pa)</th>
<th>Tan δ</th>
</tr>
</thead>
<tbody>
<tr>
<td>12%</td>
<td>4.13(^a)</td>
<td>4.37(^ab)</td>
<td>1.07(^b)</td>
</tr>
<tr>
<td>8%</td>
<td>3.58(^a)</td>
<td>4.48(^ab)</td>
<td>1.25(^c)</td>
</tr>
<tr>
<td>6%</td>
<td>3.68(^a)</td>
<td>4.65(^a)</td>
<td>1.31(^b)</td>
</tr>
<tr>
<td>6% + PFR(^1)</td>
<td>2.56(^b)</td>
<td>3.65(^b)</td>
<td>1.42(^ab)</td>
</tr>
<tr>
<td>6% + CFR(^2)</td>
<td>3.28(^b)</td>
<td>4.77(^a)</td>
<td>1.45(^a)</td>
</tr>
</tbody>
</table>

\(^a,b,c,d\)Means of three replicates in the same column with different superscripts differ (P ≤ 0.05).

\(^1\)6% milk fat mix containing the protein-based fat replacer.

\(^2\)6% milk fat mix containing the carbohydrate-based fat replacer.

CONCLUSIONS

Milk fat content influenced the elastic component in ice cream and mix samples. Besides the amount of milk fat, factors that influence fat destabilization and whip-
RHEOLOGICAL PROPERTIES OF ICE CREAM

Table 5. Mean storage modulus ($G'$), loss modulus ($G''$), and tan $\delta$ values of five ice creams measured at 1 Hz.

<table>
<thead>
<tr>
<th>Ice Cream</th>
<th>$G'$ (kPa)</th>
<th>$G''$ (kPa)</th>
<th>Tan $\delta$</th>
</tr>
</thead>
<tbody>
<tr>
<td>12%</td>
<td>39.42$^a$</td>
<td>30.70$^{ab}$</td>
<td>1.02$^b$</td>
</tr>
<tr>
<td>8%</td>
<td>22.12$^{ab}$</td>
<td>25.55$^{bc}$</td>
<td>1.44$^{ab}$</td>
</tr>
<tr>
<td>6%</td>
<td>6.05$^b$</td>
<td>11.85$^c$</td>
<td>2.01$^{ab}$</td>
</tr>
<tr>
<td>6% + PFR$^1$</td>
<td>22.25$^{ab}$</td>
<td>47.60$^a$</td>
<td>2.44$^a$</td>
</tr>
<tr>
<td>6% + CFR$^2$</td>
<td>18.65$^{ab}$</td>
<td>46.15$^a$</td>
<td>2.43$^a$</td>
</tr>
</tbody>
</table>

$^a,b,c$Means of three replicates in the same column with different superscripts differ ($P \leq 0.05$).

$^1$6% milk fat mix containing the protein-based fat replacer.

$^2$6% milk fat mix containing the carbohydrate-based fat replacer.

ping abilities, may play minor roles in the elastic behavior of a frozen dairy product. Both types of fat replacers appeared to increase the viscous component while decreasing or not affecting the elastic component. Protein- and carbohydrate-based fat replacers may be more helpful in increasing the viscous properties than the elastic properties in a dairy-based system. Thus, in lower fat ice creams, a good balance of milk fat, protein, and carbohydrate may be important in producing a desirable structure, rather than replacing milk fat by one product alone.

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