

## Ice Cream Structural Elements that Affect Melting Rate and Hardness

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### ABSTRACT

Statistical models were developed to reveal which structural elements of ice cream affect melting rate and hardness. Ice creams were frozen in a batch freezer with three types of sweetener, three levels of the emulsifier polysorbate 80, and two different draw temperatures to produce ice creams with a range of microstructures. Ice cream mixes were analyzed for viscosity, and finished ice creams were analyzed for air cell and ice crystal size, overrun, and fat destabilization. The ice phase volume of each ice cream were calculated based on the freezing point of the mix. Melting rate and hardness of each hardened ice cream was measured and correlated with the structural attributes by using analysis of variance and multiple linear regression. Fat destabilization, ice crystal size, and the consistency coefficient of the mix were found to affect the melting rate of ice cream, whereas hardness was influenced by ice phase volume, ice crystal size, overrun, fat destabilization, and the rheological properties of the mix.

**(Key words:** melting rate, hardness, ice cream, ice crystals)

**Abbreviation key:** CS = corn syrup, DE = dextrose equivalent, DP = depth of penetration, HFCS = high fructose corn syrup, PS80 = polysorbate 80, SUC = sucrose, T<sub>f</sub> = freezing point temperature.

### INTRODUCTION

Ice cream is a frozen foam that consists of air cells dispersed in an aqueous matrix (Marshall et al., 2003). The three main structural components of ice cream are air cells, ice crystals, and fat globules, which are distributed throughout a continuous phase of unfrozen solution (serum). Destabilized fat, in the form of partially coalesced fat globules, coats and stabilizes air cells. The physical structure of ice cream affects its

melting rate and hardness, although the specific relationships have not all been worked out.

The melt-down rate of ice cream can be determined by placing a sample of ice cream on a wire-mesh screen at warm temperatures and measuring the rate of fluid accumulation beneath the screen (Hartel et al., 2003). As the ice cream melts, heat transfers from the warm air surrounding the product into the ice cream to melt the ice crystals. Initially the ice melts at the exterior of the ice cream and there is a local cooling effect (in the vicinity of the melting ice). The water from the melting ice must diffuse into the viscous unfrozen serum phase, and this diluted solution then flows downwards (due to gravity) through the structural elements (destabilized fat globules, air cells, and remaining ice crystals) to drip through the screen on which the ice cream rests. During melting, the flow of this diluted solution is initially over the exterior of the ice cream. When sufficient heat penetration causes melting of the ice crystals in the interior of the ice cream, the diluted solution begins to flow from the interior as well.

The melt-down rate of ice cream is affected by many factors, including the amount of air incorporated, the nature of the ice crystals, and the network of fat globules formed during freezing. Sakurai et al. (1996) found that ice creams with low overruns melted quickly, whereas ice creams with high overruns began to melt slowly and had a good melting resistance. This slower melting rate in the ice creams with high overruns was attributed to a reduced rate of heat transfer due to a larger volume of air but may also be due to the more tortuous path through which the melting fluid must flow (Hartel et al., 2003).

The fat network plays a significant role in determining the melting rate of ice cream. Polysorbate 80 (PS80) promotes destabilization and partial coalescence of fat globules. Increasing levels of destabilized fat increase the fat network. The addition of PS80 to ice cream (at levels of 0.02% and above) has been shown to greatly decrease the melting rate of ice cream and to promote shape retention (Campbell et al., 1998; Tharp et al., 1998; Bolliger et al., 2000) through development of a more extensive fat network (Hartel et al., 2003).

Hardness of ice cream is measured as the resistance of the ice cream to deformation when an external force

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is applied. The hardness of ice cream is affected by such factors as the overrun, ice crystal size, ice phase volume, and extent of fat destabilization. An inverse relationship between hardness and overrun has been noted by many researchers (Tanaka et al., 1972; Goff et al., 1995; Wilbey et al., 1998). Both the ice crystal size and ice phase volume contribute to the hardness of ice cream. Sakurai et al. (1996) found that ice creams with more large ice crystals were harder than ice creams that had fewer large ice crystals. Wilbey et al. (1998) found that the hardness of ice cream was exponentially related to the ice phase volume. The fat network also affects hardness. Tharp et al. (1998) found that the hardness of ice cream increased as the level of PS80 or level of destabilized fat increased.

Although many factors have been shown to influence melting rate and hardness of ice cream, our understanding of these factors is not complete. For example, more information is needed on how air cell and ice crystal size, as well as ice phase volume, affect the melting rate of ice cream. The impact of air cell size on the hardness of ice cream is also unknown. The problem in generating this information, however, is that it is difficult to change just a single property of the ice cream. Changing one aspect of formulation or process conditions often causes changes in several structural aspects of ice cream. In this study, the effects of structural attributes on the melting rate and hardness of ice cream were examined by producing ice creams with as wide a range of structural attributes as possible and then using statistical models to separate the effects of each parameter on melting rate and hardness.

## MATERIALS AND METHODS

### Composition

Ice cream formulations were designed according to a 3 × 3 factorial with 3 levels of the emulsifier PS80 and 3 types of sweeteners. A constant level of mono- and diglycerides was added to the mixes and only the level of PS80 (0, 0.05, and 0.1%) was varied. The sweeteners used were [sucrose (**SUC**), 42 dextrose equivalent (**DE**) high fructose corn syrup (**HFCS**), and 20 DE corn syrup (**CS**)]. The sweeteners were selected based on their affect on the freezing point and not on their sweetness relative to sucrose. The mixes were made in triplicate to test each level of PS80 with each sweetener.

The ice cream formulations contained 12% milk fat, 17% sweetener, 0.15% stabilizer, 0.15% mono- and diglycerides, 11.49 to 11.58% milk solids not fat (**MSNF**), and 40.89% total solids. To accommodate the changing

level of PS80 and maintain a constant total solids level in the mix, the level of MSNF was varied at 11.59, 11.54, or 11.49%, depending on the level of PS80. The emulsifiers and stabilizers were obtained from Germantown International Limited (West Chester, PA). The stabilizer was a blend that consisted of 12% carageenan, 33% guar gum, and 55% carboxymethylcellulose.

### Processing

The ice cream mixes were made in 22.7-kg (50 lb) batches and pasteurized for 30 min at 71°C. The mixes were homogenized in a double-stage homogenizer at 71°C (APV Fluid Handling, type 125"E, Lake Mills, WI) with homogenization pressure of  $1.4 \times 10^4$  kPa on the first stage and  $3.4 \times 10^3$  kPa on the second stage. The mixes were then cooled and aged at 4 to 5°C for 48 h.

The ice cream was frozen in a batch freezer (Carpigiani, Coldelite model LB-502, Winston-Salem, NC) and drawn at 2 target draw temperatures, dependent on the type of sweetener in the mix. The draw temperatures were selected to provide a wide range of ice phase volumes at draw, which led to different ice crystal sizes. The mixes made with sucrose were first drawn at -5°C after 16 min of freezing, after which the settings were switched, and the second set of samples drawn when the temperature had reached -6°C. Because of the lower freezing point, the mixes made with 42 DE HFCS were first drawn at -6°C after 8 min of freezing, and the second set of samples were drawn when the temperature had reached -7°C. The mixes made with 20 DE CS, with higher freezing point, were frozen at draw temperatures of -2.5 and -3.5°C. It was not necessary to specify exact draw temperatures because the target variable was the percentage of ice frozen, which would result in a wide range of the important variables (ice crystal size, extent of fat destabilization, overrun, and air cell size).

For each mix at each draw temperature, samples were collected in two rectangular half-gallon containers and one pint container. The pints were immediately analyzed for ice crystal and air cell size (draw conditions). For the first two replicates, the half-gallon containers were hardened in a still-air freezer at -26°C for 24 h. For the third sample, the half-gallon containers were hardened in a forced-air freezer at -29°C for 24 h. In this case, the different hardening conditions led to wider differences in ice crystal size in the hardened ice creams. A third sample was included in this study to provide a wider range of ice cream structures for the melting rate and hardness models. Because the data were not averaged over the 3 samples, the

difference in hardening conditions provided a wider range of ice crystal sizes for the statistical model. Prior to analysis, the samples were tempered in a freezer at  $-15^{\circ}$  for approximately 18 h.

## Analyses

**Freezing point depression.** The freezing point of each mix was calculated based on a method by Leighton (1927) as modified by Bradley and Smith (1983). The freezing point of each mix was also measured by supercooling the mix (800 ml) to  $-6^{\circ}\text{C}$  under agitation with a mixer set at 500 rpm. The mix was seeded with ice, and due to the release of latent heat upon freezing, warmed to an equilibrium temperature. The plateau temperature was taken as the freezing point. This measurement was done in triplicate. The ice content was calculated according to the method of Bradley (1984) in which freezing points of the mix are calculated at various percentages of frozen water. The calculated freezing points can be plotted against the percentages of frozen water in order to generate freezing curves.

**Mix viscosity.** A concentric cylinder viscometer (model LVT, Brookfield, Stoughton, MA) was used to measure the viscosity of 600 ml of ice cream mix at 4 to  $5^{\circ}\text{C}$  after 48 h of aging. Spindle #2H was used to take torque measurements at 100, 50, 25, 10, 5, 2.5, 1.0, and 0.5 rpm. One measurement was taken per mix. The shear stress and the shear rate of the mixes were calculated, and the power law model was used to determine the flow behavior index ( $n$ ) and consistency coefficient ( $K$ ). The flow behavior index signifies how close the mix is to Newtonian. The consistency coefficient gives an indication of the flow properties of the mix. The apparent viscosity was calculated according to the manual for the Brookfield Viscometer.

**Fat Destabilization.** Fat destabilization during freezing was measured with a method similar to that used by Keeney and Josephson (1958). The absorbance of diluted mix was compared to that of diluted, melted ice cream. The difference in absorbance is related to the formation of fat globule agglomerates.

**Draw temperature.** A thermocouple, accurate to  $\pm 0.1^{\circ}\text{C}$ , was carefully placed in the flow of ice cream upon exiting the draw of the freezer to measure draw temperature.

**Overrun.** One overrun measurement was taken per sample by comparing the weight of ice cream mix and ice cream in a fixed volume container. Overrun (in %) was calculated as follows.

$$\text{Overrun} = \frac{\text{weight of mix} - \text{weight of ice cream}(100)}{\text{weight of ice cream}} \quad (1)$$

**Air cell and ice crystal size.** Analysis of air cell and ice crystal size was done in a temperature-controlled glove box according to methods developed by Chang and Hartel (2002) and Donohue et al. (1991), respectively. The glove box temperature was set at  $-6^{\circ}\text{C}$  for analysis of air cells and ice crystals in fresh drawn ice creams and for air cells in hardened ice creams. (It was set at  $-4.5^{\circ}\text{C}$  for hardened ice creams with 20 DE corn syrup). The glove box temperature was set at  $-15^{\circ}\text{C}$  for ice crystal analysis in hardened ice creams. For ice crystal size analysis, a small sample of ice cream was placed on a prechilled microscope slide (equilibrated in the glove box), dispersed with a few drops of kerosene, and smeared into a thin layer with a second slide placed on top of the sample. Images were taken with a photo-lens and video camera that were attached to the microscope to accumulate at least 300 ice crystals for image analysis. For air cell size analysis, a small sample of ice cream was placed on a prechilled microscope slide and smeared into a thin layer with a second slide. At  $-6^{\circ}\text{C}$ , the air cells were sufficiently buoyant to rise to the bottom of the top slide. Images were taken to accumulate at least 300 air cells for image analysis. Images of both ice crystals and air cells were manually digitized to generate a size distribution based on equivalent circular diameter. The size of each air cell or ice crystal was defined as the diameter of a circle with an area equivalent to the projected area of the air cell or ice crystal. Distribution statistics, including population-based mean size, were calculated with a custom-written program.

**Melting rate.** A sample of ice cream was cut from the end of a rectangular half-gallon container to make a rectangular block of ice cream that weighed  $100 \pm 1$  g. The dimensions of the sample varied slightly from one ice cream to another (6 to 7 cm  $\times$  6 to 7 cm  $\times$  3 cm) due to the differences in physical characteristics (e.g., overrun). The sample of ice cream (initially at  $-15^{\circ}\text{C}$ ) was placed on a wire screen (6 holes/cm) on top of a funnel that was attached to a graduated cylinder. The ice cream was placed in a controlled temperature chamber at  $25^{\circ}\text{C}$  (RH  $\approx$  50%). Every 5 min, for up to 3 h, the dripped volume was recorded. Pictures of the ice cream were taken with a digital camera every 20 min. The time (min) was plotted against the dripped volume (mL) and the slope of the main melting event was taken as the melting rate.

**Hardness.** A penetrometer (Precision Penetrometer, Precision Scientific Company, Chicago, IL) was

**Table 1.** Ice crystal content (calculated) at different temperatures and freezing temperatures (measured) of ice creams with either sucrose (SUC), 42 dextrose equivalent (DE) high fructose corn syrup (HFCS), or 20 DE corn syrup (CS) at various temperatures.

Mix	Freezing point (°C)	Temp. (°C)	Ice (%)
SUC	-2.9	-5	38
		-6	47
		-7	53
		-15	77
HFCS	-4.6	-6	22
		-7	32
		-8	38
		-15	65
		-2.5	28
20 DE CS	-1.8	-3	42
		-3.5	48
		-15	85

used to measure the hardness of the ice cream at  $-15^{\circ}\text{C}$ . Samples were quickly transported to the penetrometer, held at room temperature, and analyses completed within 30 s to minimize variability due to sample warming. A 42-g stainless steel probe ( $60^{\circ}$  cone with a maximum diameter of 6.5 cm) was aligned so that it touched the surface of the ice cream and then allowed to penetrate the samples under the force of gravity for five seconds. The depth of penetration (DP) was inversely proportional to the hardness. Three measurements were taken per sample.

**Statistical analysis.** Analysis of variance (ANOVA) and a multiple comparison test (SAS version 8.2, SAS Institute, Inc.) were used to find relationships between melting rate and hardness and the physical properties of the ice creams. Separate multiple linear regressions for melting rate and hardness were also performed to show how the observed physical properties related to the melting rate and hardness. The regressions initially included all of the physical properties (apparent viscosity, consistency coefficient, flow behavior index, air cell size, ice crystal size, overrun, fat destabilization, and ice phase volume). After the first regression, all parameters with  $P$ -values above 0.1 (not significant at 10% confidence interval) were removed and the regression performed again on a smaller set of variables. This procedure was repeated until only the physical properties with  $P < 0.1$  remained.

## RESULTS AND DISCUSSION

### Ice Phase Volume

Table 1 shows the measured freezing point temperatures ( $T_f$ ) as well as the calculated ice crystal contents

for each mix at different temperatures. As expected, the mix made with 20 DE CS had the highest  $T_f$  and the mix made with HFCS had the lowest  $T_f$  based on the greater level of monosaccharides in HFCS. Thus, at any storage temperature, the ice creams made with 20 DE CS had the highest ice content. Note that the values of ice crystal content are calculated based on the assumptions of thermal equilibration and maximal crystallization of ice at each temperature.

### Mix Viscosity

Table 2 shows the consistency coefficient, flow behavior index, and apparent viscosity (averaged over triplicates) for ice cream mixes with different sweeteners. As expected, the ice cream mixes with 20 DE CS were the most viscous and those made with HFCS were the least viscous. This can be attributed to the differences in molecular weight and polymer chain length among the sweeteners. The addition of PS80 had no effect on the rheological properties of the mix.

### Draw Time and Temperature

The draw times and temperatures are shown in Table 3. The actual draw temperatures were close enough to the target draw temperatures to provide ice creams with different ice crystal contents at draw.

### Fat Destabilization

The ice creams made with 20 DE CS generally had the highest levels of destabilized fat, whereas those made with HFCS had the lowest levels of destabilized fat (Table 4). The higher level of destabilized fat in the ice creams made with 20 DE CS can be attributed to the high viscosity and percentage of ice during freezing, which led to high shear forces and enhanced fat destabilization. The amount of destabilized fat also significantly increased ( $P < 0.05$ ) from the first draw temperature to the second for ice creams of all sweetener types due to both higher shear forces (more ice) and longer shearing time. Furthermore, the level of destabilized fat generally increased with the level of PS80, in agreement with the results of Tharp et al. (1998). However, ice creams made with 20 DE CS had lower levels of fat destabilization at the highest level of PS80, suggesting that fat creaming, butter granules formed by extensive destabilization, had occurred under these conditions.

**Table 2.** Consistency coefficient, flow behavior index (power law model), and apparent viscosity for ice cream mixes made with sucrose (SUC), 42 dextrose equivalent (DE) high fructose corn syrup (HFCS) or 20 DE corn syrup (CS) and different levels of polysorbate 80 (PS80).

Sweetener in ice cream	PS80 (%)	Apparent viscosity <sup>1</sup> at 20 rpm (cP)	Consistency coefficient <sup>1</sup> (K) (dynes/cm <sup>2</sup> )	Flow behavior index <sup>1</sup> (n)
SUC <sup>g</sup>	0	712 ± 115 <sup>a</sup>	163 ± 23 <sup>b</sup>	0.50 ± 0.02 <sup>a</sup>
	0.05	621 ± 62 <sup>b</sup>	145 ± 13 <sup>a</sup>	0.49 ± 0.02 <sup>a</sup>
	0.1	716 ± 58 <sup>a</sup>	166 ± 18 <sup>b</sup>	0.50 ± 0.03 <sup>a</sup>
HFCS <sup>g</sup>	0	671 ± 111 <sup>cd</sup>	158 ± 26 <sup>c</sup>	0.49 <sup>c</sup>
	0.05	719 ± 27 <sup>c</sup>	170 ± 11 <sup>c</sup>	0.48 ± 0.01 <sup>c</sup>
	0.1	584 ± 74 <sup>d</sup>	165 ± 23 <sup>c</sup>	0.48 ± 0.03 <sup>c</sup>
20 DE CS <sup>h</sup>	0	935 ± 134 <sup>e</sup>	209 ± 36 <sup>e</sup>	0.53 ± 0.04 <sup>e</sup>
	0.05	925 ± 196 <sup>e</sup>	211 ± 51 <sup>e</sup>	0.53 ± 0.05 <sup>e</sup>
	0.1	752 ± 95 <sup>f</sup>	169 ± 19 <sup>f</sup>	0.53 ± 0.02 <sup>e</sup>

<sup>1</sup>Mean values (± standard deviation) for all ice creams of a given sweetener type and emulsifier level.

<sup>a,b</sup>Denote significant differences for ice creams with sucrose.

<sup>c,d</sup>Denote significant differences for ice creams with 42 DE HFCS.

<sup>e,f</sup>Denote significant differences for ice creams with 20 DE CS.

<sup>g,h</sup>Denote significant differences for all rheological properties individually averaged over sweetener type.

## Overrun

As seen in Table 4, the overrun generally increased with the higher level of PS80 and lower (second) draw temperature. Higher levels of PS80 and longer whipping times (lower freezing temperature) promoted increased levels of destabilized fat, which provides air cell stability. However, the range of overruns produced in this study was relatively narrow, varying only from about 40 to 70%.

## Air Cell and Ice Crystal Size

At draw, all ice creams had air cells (mean sizes varied from 14 to 20 μm) and ice crystals (27 to 33 μm) of similar size. Differences in air cell size among the mixes were only observed after hardening, as seen in Table 5. The air cells in the ice creams made with 20 DE CS were the smallest, which can be attributed to

the fact that these ice creams had the most ice, highest apparent viscosities, and thus, the highest shear stresses breaking down the air cells. The air cells were also generally smaller at the second draw temperatures for all mixes due to the increased residence times during shearing.

After hardening, the ice creams made with sucrose had the smallest ice crystals, with mixes made with 20 DE CS having the largest ice crystals. The larger ice crystals in the ice creams made with 20 DE CS can be attributed to the greater ice crystal content. The ice crystals were also generally smaller at the lower (second) draw temperatures, particularly in the ice creams with HFCS and CS.

## Melting Rate

The melting rates of the ice creams are shown in Table 6. The ice creams made with 20 DE CS had the

**Table 3.** Draw times and temperatures for hardened ice creams made with sucrose (SUC), 42 dextrose equivalent (DE) high fructose corn syrup (HFCS) or 20 DE corn syrup (CS) frozen to different draw temperatures.

Sweetener in the ice cream	Draw temperature number <sup>1</sup>	Draw time <sup>2</sup> (sec)	Target draw temperature (°C)	Actual draw temperature <sup>2</sup>
SUC	1	401 ± 53	-5.0	-5.1 ± 0.2
	2	1112 ± 36	-6.0	-6.8 ± 0.4
HFCS	1	348 ± 49	-6.0	-6.1 ± 0.2
	2	643 ± 35	-7.0	-7.8 ± 0.4
20 DE CS	1	327 ± 36	-2.5	-2.9 ± 0.2
	2	476 ± 47	-3.5	-3.4 ± 0.2

<sup>1</sup>Either first or second target draw temperature.

<sup>2</sup>Mean values (± standard deviation) for all ice creams (with all levels of polysorbate 80) of a given sweetener type and draw temperature.

**Table 4.** Extent of fat destabilization and overrun for ice creams made with either sucrose (SUC), 42 dextrose equivalent (DE) high fructose corn syrup (HFCS) or 20 DE corn syrup (CS) at different levels of polysorbate 80 (PS80) and draw temperatures.

Sweetener in ice cream	Draw temp. number <sup>1</sup>	PS80 (%)	Fat destabilization <sup>2</sup> (%)	Overrun <sup>2</sup> (%)
SUC	1	0	7.0 ± 4.3 <sup>a</sup>	58.7 ± 6.0 <sup>a</sup>
		0.05	9.7 ± 3.2 <sup>a</sup>	69.3 ± 9.3 <sup>b</sup>
		0.1	29.0 ± 16.2 <sup>b</sup>	70.7 ± 8.0 <sup>bc</sup>
	2	0	25.6 ± 12.9 <sup>b</sup>	49.8 ± 10.9 <sup>a</sup>
		0.05	55.5 ± 6.3 <sup>c</sup>	60.2 ± 10.6 <sup>ac</sup>
		0.1	68.4 ± 12.1 <sup>c</sup>	55.0 ± 18.8 <sup>a</sup>
HFCS	1	0	6.7 ± 4.6 <sup>d</sup>	37.7 ± 8.3 <sup>d</sup>
		0.05	6.0 ± 8.0 <sup>d</sup>	39.2 ± 11.9 <sup>df</sup>
		0.1	18.6 ± 8.1 <sup>de</sup>	66.0 ± 13.0 <sup>eg</sup>
	2	0	10.4 ± 2.3 <sup>d</sup>	58.2 ± 4.2 <sup>ef</sup>
		0.05	28.7 ± 6.3 <sup>e</sup>	66.7 ± 5.6 <sup>eg</sup>
		0.1	45.8 ± 9.2 <sup>f</sup>	71.3 ± 14.5 <sup>g</sup>
20 DE CS	1	0	24.2 ± 10.7 <sup>h</sup>	43.7 ± 3.8 <sup>j</sup>
		0.05	48.4 ± 5.8 <sup>i</sup>	51.7 ± 4.0 <sup>hij</sup>
		0.1	37.5 ± 5.8 <sup>i</sup>	56.5 ± 3.4 <sup>hi</sup>
	2	0	44.8 ± 7.8 <sup>i</sup>	48.8 ± 8.6 <sup>ij</sup>
		0.05	68.9 ± 9.7 <sup>j</sup>	54.7 ± 6.0 <sup>hij</sup>
		0.1	33.0 ± 11.3 <sup>hi</sup>	61.0 ± 4.7 <sup>h</sup>

<sup>a,b,c</sup>Denote significant differences among ice creams with sucrose.

<sup>d,e,f,g</sup>Denote significant differences among ice creams with 42 DE HFCS.

<sup>h,i,j</sup>Denote significant differences among ice creams with 20 DE CS.

<sup>1</sup>Either first or second target draw temperature (see Table 3).

<sup>2</sup>Mean values (± standard deviation) for all ice creams of a given sweetener type, emulsifier level, and draw temperature.

**Table 5.** Air cell and ice crystal sizes in hardened ice creams made with either sucrose (SUC), 42 dextrose equivalent (DE) high fructose corn syrup (HFCS) or 20 DE corn syrup (CS) at different levels of polysorbate 80 (PS80) and draw temperatures.

Sweetener in ice cream	Draw temp. number <sup>1</sup>	PS80 (%)	Air cell size <sup>2</sup> (μm)	Ice crystal size <sup>2</sup> (μm)
SUC	1	0	21.9 ± 2.7 <sup>a</sup>	51.1 ± 4.0 <sup>a</sup>
		0.05	21.5 ± 2.9 <sup>a</sup>	52.6 ± 3.0 <sup>a</sup>
		0.1	19.9 ± 2.6 <sup>a</sup>	48.8 ± 2.0 <sup>a</sup>
	2	0	20.4 ± 1.1 <sup>a</sup>	48.9 ± 7.6 <sup>a</sup>
		0.05	19.6 ± 2.4 <sup>a</sup>	48.5 ± 5.5 <sup>a</sup>
		0.1	19.3 ± 2.1 <sup>a</sup>	41.4 ± 3.1 <sup>b</sup>
HFCS	1	0	24.7 ± 2.2 <sup>d</sup>	58.3 ± 3.7 <sup>c</sup>
		0.05	22.0 ± 3.3 <sup>cd</sup>	59.6 ± 5.0 <sup>c</sup>
		0.1	20.8 ± 2.7 <sup>c</sup>	59.4 ± 3.6 <sup>c</sup>
	2	0	22.6 ± 1.7 <sup>cd</sup>	49.1 ± 2.9 <sup>d</sup>
		0.05	21.6 ± 2.2 <sup>cd</sup>	46.1 ± 6.0 <sup>d</sup>
		0.1	19.8 ± 2.0 <sup>c</sup>	46.2 ± 3.4 <sup>d</sup>
20 DE CS	1	0	17.5 ± 3.0 <sup>e</sup>	64.0 ± 7.1 <sup>f</sup>
		0.05	17.3 ± 1.3 <sup>e</sup>	63.9 ± 5.4 <sup>f</sup>
		0.1	18.1 ± 1.2 <sup>e</sup>	62.9 ± 6.9 <sup>fg</sup>
	2	0	17.0 ± 2.1 <sup>e</sup>	55.3 ± 1.3 <sup>e</sup>
		0.05	15.9 ± 2.1 <sup>e</sup>	52.5 ± 3.1 <sup>e</sup>
		0.1	16.4 ± 1.3 <sup>e</sup>	56.3 ± 2.0 <sup>eg</sup>

<sup>a,b</sup>Denote significant differences among ice creams with sucrose.

<sup>c,d</sup>Denote significant differences among ice creams with 42 DE HFCS.

<sup>e,f,g</sup>Denote significant differences among ice creams with 20 DE CS.

<sup>1</sup>Either first or second draw temperature (see Table 3).

<sup>2</sup>Mean values (± standard deviation) of all ice creams of a given sweetener type, emulsifier level, and draw temperature.

**Table 6.** Mean melting rate and depth of penetration of ice creams made with either sucrose (SUC), 42 dextrose equivalent (DE) high fructose corn syrup (HFCS) or 20 DE corn syrup (CS) at different levels of polysorbate 80 (PS80) and draw temperatures.

Sweetener in ice cream	Draw temp. number <sup>1</sup>	PS80 (%)	Melting rate <sup>2</sup> (ml/min.)	Depth of penetration <sup>2</sup> (mm)
SUC	1	0	3.2 ± 0.3 <sup>a</sup>	17.5 ± 0.6 <sup>ad</sup>
		0.05	2.8 ± 0.3 <sup>a</sup>	18.0 ± 1.3 <sup>a</sup>
		0.1	1.7 ± 1.1 <sup>b</sup>	17.4 ± 0.8 <sup>ac</sup>
	2	0	1.2 ± 0.5 <sup>b</sup>	14.2 ± 2.3 <sup>b</sup>
		0.05	0.3 <sup>c</sup>	15.7 ± 1.0 <sup>bcd</sup>
		0.1	0.2 <sup>c</sup>	14.2 ± 1.4 <sup>b</sup>
HFCS	1	0	3.3 ± 0.3 <sup>e</sup>	19.2 ± 1.2 <sup>e</sup>
		0.05	3.5 ± 0.4 <sup>e</sup>	21.4 ± 1.1 <sup>e</sup>
		0.1	3.4 ± 0.3 <sup>e</sup>	20.4 ± 1.4 <sup>e</sup>
	2	0	3.6 ± 0.4 <sup>e</sup>	19.8 ± 1.7 <sup>e</sup>
		0.05	1.3 ± 0.6 <sup>f</sup>	20.6 ± 1.5 <sup>e</sup>
		0.1	0.2 <sup>g</sup>	19.8 ± 1.1 <sup>e</sup>
20 DE CS	1	0	2.5 ± 0.2 <sup>h</sup>	7.4 ± 1.1 <sup>i</sup>
		0.05	1.0 ± 0.9 <sup>i</sup>	9.0 ± 1.0 <sup>hi</sup>
		0.1	2.4 ± 0.2 <sup>h</sup>	9.1 ± 0.5 <sup>hi</sup>
	2	0	0.6 ± 0.6 <sup>j</sup>	8.4 ± 0.3 <sup>hi</sup>
		0.05	0.2 <sup>k</sup>	8.6 ± 0.5 <sup>hi</sup>
		0.1	2.4 ± 0.3 <sup>h</sup>	9.6 ± 0.9 <sup>h</sup>

<sup>a,b,c,d</sup>Denote significant differences for ice creams with sucrose.

<sup>e,f,g</sup>Denote significant differences for ice creams with 42 DE HFCS.

<sup>h,i,j,k</sup>Denote significant differences for ice creams with 20 DE CS.

<sup>1</sup>Either first or second draw temperature (see Table 3).

<sup>2</sup>Mean values (± standard deviation) for ice creams based on sweetener type, draw temperature, and level of emulsifier.

slowest melting rate, whereas those made with HFCS had the fastest melting rates. The melting rates slowed with increasing levels of PS80 and at the lower (second) draw temperatures. Statistical analysis was used to distinguish the important structural elements that influenced melting rate. At the 90% confidence level, the extent of fat destabilization ( $P < 0.0001$ ), ice crystal size ( $P < 0.0001$ ), and the consistency coefficient of the mix ( $P = 0.0508$ ) were found to influence melting. The linear regression equation that describes the collective effect of these physical properties on the melting rate is given as:

$$\text{Mean melting rate} = 2.006 - 0.046F - 0.004K + 0.039I \quad (2)$$

Here, F is extent of fat destabilization (%), K is consistency coefficient (Pa-s), and I is the ice crystal size ( $\mu\text{m}$ ). According to Equation (2), the mean melting rate (mL/min) increased as the level of fat destabilization decreased, the consistency coefficient decreased, and the ice crystal size increased. Fat destabilization and ice crystal size were the most important factors (largest coefficients) with the consistency coefficient having only a small effect.

To evaluate the randomness of the statistical model, predicted melting rates were compared to the residual

(difference between observed and the predicted melting rates). Most of the predicted melting rates were within the residual standard error ( $\pm 0.5$ ) given by the model, and there were no set patterns to explain the differences between the observed and predicted melting rate. The  $R^2$  value was 0.73, so the model was able to explain a good portion of the variation in the data. Perhaps other physical properties not included in this model may affect the melting rate and strengthen this model. For example, overrun has been found to affect melting rate in previous studies, but it did not appear as a factor in this study because of the narrow range of overruns obtainable in this batch freezer.

Fat destabilization has the largest effect on the melting rate of ice cream. Destabilized fat in ice cream takes the form of clumps of fat globules that coat and support the air cells and also chains of fat globules that build a fat network in the ice cream (Marshall et al., 2003). In this study, ice creams made with high levels of PS80 and frozen to lower temperatures promoted increased levels of destabilized fat, which contributed to slower melting rates. Bolliger et al. (2000) also found that ice creams with lower levels of destabilized fat had faster melting rates. A greater extent of destabilized fat increased the resistance to flow of the serum phase as ice melted, which led to slower melt down (Hartel et al., 2003).

The ice creams with high levels of destabilized fat also retained their shape well during melting. The fat network in these ice creams helped maintain the ice cream on the screen (Tharp et al., 1998; Bolliger et al., 2000). The shape retention in the ice creams during melting depended on the level of destabilized fat. Ice creams with low levels of destabilized fat (up to 30%) melted quickly and did not retain their shape. Ice creams with high levels of destabilized fat (above 50%) retained their shape well.

Visually, the fluid that dripped during melting also differed depending on the level of destabilized fat. Upon comparing the observations recorded during the melt-down test with the fat destabilization data, it became apparent that in ice creams with less than approximately 50% destabilized fat, the dripped fluid was white and contained air cells. That is, the dripped fluid was a white emulsion with dispersed air cells. In ice creams with greater than about 50% destabilized fat, the dripped fluid was clear and did not contain any visible air cells. In these ice creams, the air cells were most likely trapped by the fat network and remained on top of the screen.

As seen in Equation (2), ice crystal size also was shown to affect the melting rate of ice cream. Over the range of mean ice crystal sizes in this study (41 to 64  $\mu\text{m}$ ), melting rate increased as ice crystal size increased. This can be related to the flow path of melted ice cream (Hartel et al., 2003). With many small crystals, the flow path of the serum phase as the ice melts is more tortuous, meaning that the fluid must travel around more obstacles (the numerous ice crystals). Thus, melting rate, measured as the rate of drip loss, is reduced when there are many smaller ice crystals. Interestingly, melt-down rate was not found to be influenced by ice crystal content, suggesting that it was indeed flow of melted fluid rather than differences in heat transfer that caused the observed effect from ice crystal size.

The flow behavior of the mix was also found to have a slight effect on the melting rate of ice cream in this study. To a first approximation, the viscosity of the mix may be taken as a measure of the viscosity of the unfrozen phase (serum) in the ice cream. As the ice crystals melt, the water must diffuse into this serum phase. Ice creams with high consistency coefficients had a greater resistance to flow. Since the melting rate was measured in terms of the amount of fluid dripping through a screen, ice creams that did not flow as well as others would not drip through the screen as fast and thus, melted slower. However, the consistency coefficients were not that different among mixes and the variability in their measurement was quite high, so despite the statistical significance put on consistency

coefficient by the statistical model, further work is needed to verify these results.

Overrun did not appear to have a significant effect on the melting rate of ice cream in this study. This contradicts research by Sakurai et al. (1996) and Sofjan (2002), who showed that ice creams with lower overruns had faster melting rates. The most likely reason why overrun did not appear to be significant in this study was the low range of overruns obtained during batch freezing. Most ice creams had overruns around 50 to 60% and only a few ice creams had higher or lower values. The use of a batch freezer in this study did not allow for sufficient differences in overruns. A wider range in overrun is needed to see an effect on melting rate.

### Hardness

The DP of the ice creams is shown in Table 6. In general, the ice creams made with 20 DE CS were the hardest, whereas those made with HFCS were the softest. Ice creams made with sucrose had intermediate hardness.

Statistical analysis was used to distinguish the structural attributes that affected hardness of ice cream. According to the statistical analysis ice crystal content ( $P < 0.0001$ ), flow behavior index ( $P < 0.0001$ ), consistency coefficient ( $P < 0.0001$ ) of the mix, fat destabilization ( $P = 0.0005$ ), overrun ( $P = 0.0077$ ), and ice crystal size ( $P = 0.073$ ) all influenced hardness at the 90% confidence level. The linear regression equation that describes the collective effect of those physical properties on the DP is given as:

$$\text{DP} = 66.016 - 0.420V - 27.073N - 0.0237K - 0.045I + 0.035R - 0.033F \quad (3)$$

Here, V is ice crystal content (%), N is flow behavior index (dimensionless), K is consistency coefficient (Pa-s), I is ice crystal size ( $\mu\text{m}$ ), R is overrun (%), and F is extent of fat destabilization (%). The DP is inversely related to the hardness, so that the greater the depth of penetration, the softer the sample. The DP increased (hardness decreased) as the ice phase volume, the consistency coefficient, flow behavior index, ice crystal size, and level of destabilized fat decreased and the overrun increased.

A plot (not shown) of the predicted DP versus the residual (difference between observed and predicted DP) showed that most of the predicted DP values were within the residual standard error ( $\pm 2.6$ ). Only a few points were outside this standard error. The randomness of the residuals suggests that this model describes hardness in ice cream well. However, the R



squared value was 0.46, so the model explains about half of the variation in the data. This may be due to inherent variability in the hardness measurement, due to the lack of sensitivity of the penetrometer, although the standard errors of the measurements (Table 6) were not that large.

The ice phase volume was found to significantly impact the hardness of the ice creams. In this study, the ice phase volume was varied by changing the sweetener used in the ice cream. Ice creams made with 20 DE CS had the greatest ice phase volumes and were the hardest. The more ice in the ice cream, the lower the depth of penetration by the penetrometer, and the harder the sample. In the sense of a composite material, a higher volume of a solid dispersed phase leads to greater resistance to an applied force (Hartel et al., 2003). A positive correlation between hardness of ice cream and the amount of ice was also found by Wilbey et al. (1998).

Ice crystals comprise a large portion of the bulk of ice cream, and their size also was found to affect the hardness of ice cream. In this study, as the ice crystal size increased in the ice creams, mostly through control of freezing temperature, the hardness increased. Sakurai et al. (1996) also found that ice creams with large ice crystals were harder than those with smaller ice crystals.

The rheological properties of the mix were shown to have the largest effect on the hardness of the ice cream. The flow behavior index gives an indication of how close the ice cream mix is to a Newtonian fluid. Surprisingly, the flow behavior index had the largest coefficient in the hardness equation. The flow behavior index is related to the apparent viscosity. As the flow behavior index increased, the apparent viscosity increased and the DP decreased (hardness increased). The ice creams were harder when the apparent viscosity was greater. The consistency coefficient of the mix is a measure of the flow of the unfrozen phase of the ice cream and was also shown to contribute to the hardness of ice cream. Ice cream mixes with high consistency coefficients were very viscous, and this viscosity increased the resistance to penetration by the probe.

The overrun in ice cream also affects hardness. As seen by Wilbey et al. (1998), ice creams with high overruns are softer. The hardness model in this study indicated that as overrun increased in ice cream, the hardness decreased, in agreement with the results of Wilbey et al. (1998). In this case, the larger volume of a compressible dispersed phase led to less resistance to an applied force (Hartel et al., 2003).

The level of fat destabilization also had an effect on the hardness of ice cream in this study. Destabilized

fat provides a network between the air cells in the ice cream and can thereby increase the hardness of ice cream. According to the model, as fat destabilization increased, the hardness increased. Tharp et al. (1998) also found this trend in ice creams with increasing levels of PS80. The ice creams with the highest levels of PS80 had the highest levels of destabilized fat and were the hardest.

## CONCLUSIONS

In this study, a statistical approach was used to distinguish the effects of various structural elements on melting rate and hardness. Extent of fat destabilization and ice crystal size had the largest effect on melting rate, with the consistency coefficient of the ice cream mix having a lesser effect. Due to the narrow range of overruns in the ice cream produced in this study, overrun was not found to be a determinant factor in the melting rate even though previous studies have documented this effect. Numerous factors (ice crystal content, ice crystal size, extent of fat destabilization, overrun, and the rheological properties of the mix) were found to influence hardness of ice cream, as measured by a penetrometer.

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