FDC FROZEN DESSERT CENTER

**Babcock Hall / Department of Food Science**
**University of Wisconsin-Madison**
**1605 Linden Drive, Madison, WI, 53706**

**Analysis of Frozen Desserts**

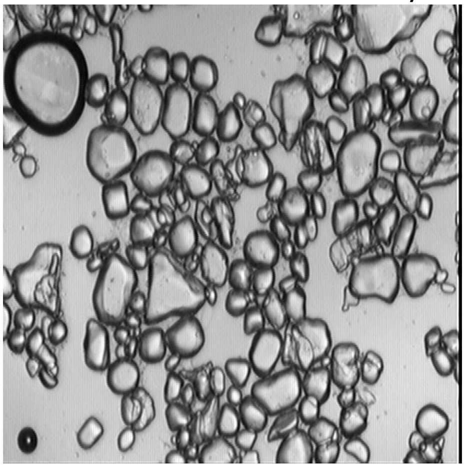
The Frozen Dessert Center offers a wide range of analytical services. We provide the following comprehensive tests:

**Ice crystal size distribution**

Ice crystal size and changes in ice crystal size are one important component of ice cream and frozen dessert quality. In general, as ice crystals grow, the icier the finished product will be perceived. Measuring ice crystal size is a way to gain insight into how formulation, processing parameters, and freezing systems are impacting ice crystal size and growth over time.

Ice crystals are measured by using a light microscope housed in an insulated glove box system (Donhowe and others, 1991) at a temperature of −15 °C. For analysis, product samples are taken from the middle of the container in the glove box, disposing the top layer of the product, and are transferred to a chilled microscope slide. A drop of chilled 50% pentanol and 50% kerosene dispersing solution is added to aid in dispersing ice crystals and to assist in image quality. A chilled cover slip is placed on top of the sample and by applying a twisting motion and minimal pressure with chilled tweezers, the sample is gently spread thin for additional image clarity. Ten-20 images at 40x magnification are taken to insure that at least 300 crystals can be counted amd sized for each replicate. Ice crystal images are edited and then analyzed using Image Pro Plus software.

Donhowe, D. P., Hartel, R. W., & Bradley, R. L. (1991). Determination of ice crystal size distributions in frozen desserts. Journal of Dairy Science, 74, 3334-3344.



**Air cell size distribution**

Air is an important ingredient in ice cream and some frozen desserts. If a product is high in overrun (amount of air whipped in) it will be fluffy and light, if a product is low in overrun, it will be denser. The same formula can have very different sensory properties at different levels of overrun. Mix formulation, freezer type, as well as processing conditions can have an impact on the size of the air cells in the final product. Air cell size, like ice crystal size, can change during storage and temperature abuse.

Similar to ice crystal size analysis, air cells are analyzed using the same refrigerated glove box (Chang and Hartel 2002). To properly image air cells, special slides are made by gluing 2 cover slips, approximately 1 to 1.5 cm apart, on a microscope slide, this creates a well, approximately 100 to 200 µm deep. In the glove box (−15 °C), a thin slice of hardened ice cream is taken from the middle of each container, discarding of the top layer of the product, and transferred to the well of the prepared microscope slide. A drop of chilled

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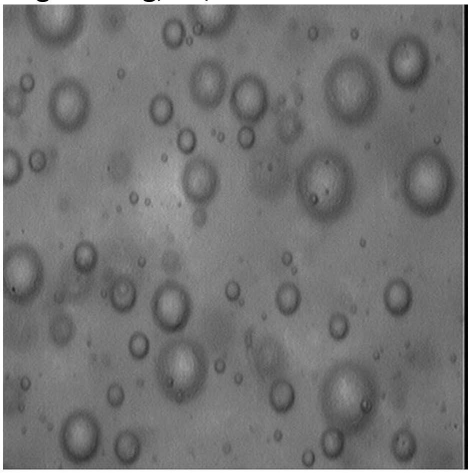
50% pentanol and 50% kerosene dispersing solution is added to assist in image quality, where needed, and a cover slip is placed over the well. The glove box temperature is then raised to −6 °C allowing for air cells to float to the surface of the cover slip. Using a light microscope and camera, images of the air cells are taken at 40× magnification. Air cells are traced and analyzed for their size using Image Pro Plus software. For each replicate, at least 300 air cells are traced.

Chang, Y.-H., & Hartel, R. W. (2002). Development of air cells in a batch ice cream freezer. Journal of Food Engineering, 55, 71-78.



**Fat Globule Size Distribution**

The size distribution of fat globules in a sample can give us insight into an important structural element found in some frozen desserts. Partial coalescence, or fat destabilization, is a phenomenon that occurs in frozen desserts containing butterfat and possibly some types of vegetable fats. Because of the complex array of fatty acids and triglycerides, butterfat has a very wide melting range. Because of this property, even at low temperatures, within each fat droplet, there is a portion of solid and liquid fat. During the dynamic freezing process, when fat droplets collide, they stick together, but because of the solid fat portion, the majority of the original structure of each droplet is retained. This allows for clusters of fat droplets to form. These partially coalesced fat globules will help stabilize air cells and create a sort of internal scaffolding in the finished product. The degree of partial coalescence can impact melt rate as well as the perceived creaminess of the final frozen dessert. However, this phenomenon can be overexpressed, and lead to the formation of butter granules, which is considered a defect.

Fat globule size is measured using a Mastersizer 2000 (Malvern Instruments LTD, Malvern, Worcestershire, UK). Deionized water (refractive index (RI) 1.33) is used to disperse the melted frozen dessert. The refractive index of the dispersed phase is adjusted based on the fat source. A refractive index of 1.46 is used when the fat source of the dispersed phase is butterfat. Drops of diluted ice cream are added until obscuration values of 13-15% were obtained (Goff & Hartel, 2013).

This generates a particle size distribution. In general, the particle size distributions of ice cream mix and melted ice cream have characteristic peaks, see figure below (Bolliger and others, 2000). Ice cream mixes exhibit two peaks; the peak appearing at about 0.3-0.4µm represents the casein micelles in the sample, the second peak, usually centered around 1µm, is the emulsion peak, representing the individual fat globules. Melted ice cream generally has a third peak beginning somewhere between 3 and 10 µm; this peak represents the partially coalesced fat clusters that have formed during the freezing process (Goff & Hartel, 2013). The extent of partial coalescence can be determined by comparing the particle size distribution of aged ice cream mix with that of the melted ice cream.

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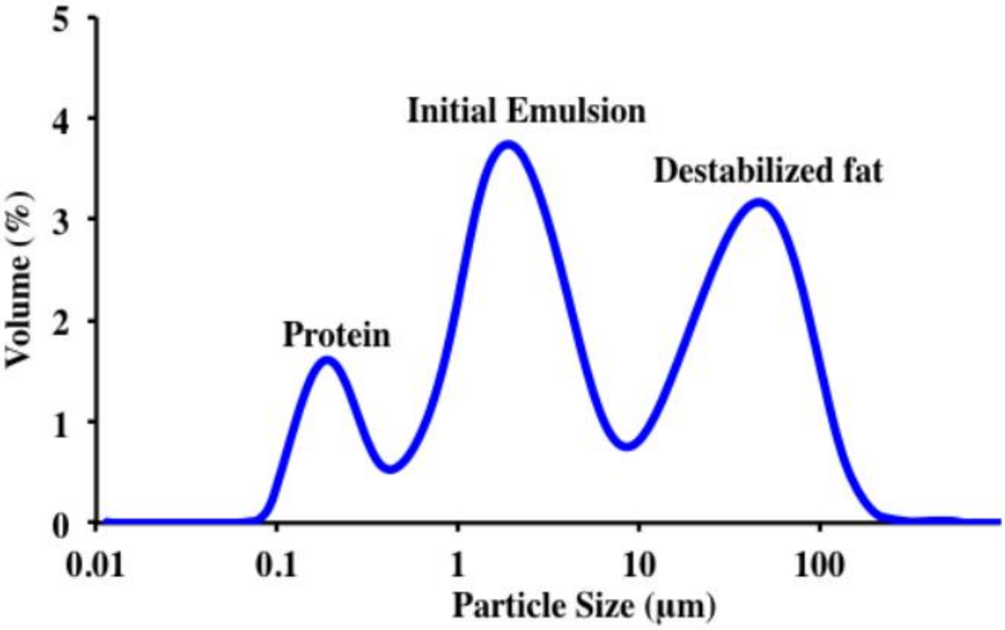
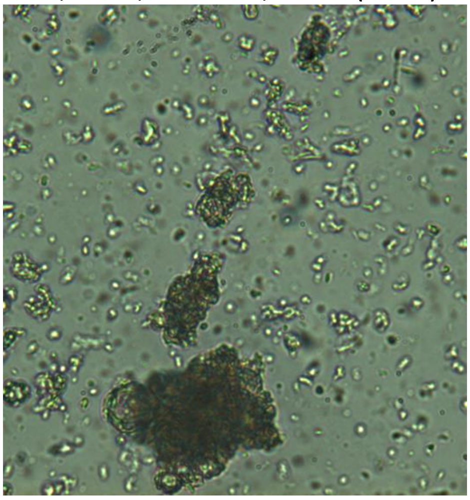



To qualitatively verify the Mastersizer data, samples are diluted by mixing two drops of the melted sample with 2 mL of water. Drops of the dilute sample are placed on glass slides and viewed under the microscope at 400x magnification using a light microscope. Images are taken of unique features found in the sample.

Bolliger S, Goff HD, Tharp BW. 2000. Correlation between colloidal properties of ice cream mix and ice cream. Int Dairy J 10:303–9.
Goff, H. D., & Hartel, R. W. (2013). Ice cream (7th ed.). New York, NY, USA: Springer US.



**Melt-down rate**

Melt-down behavior of a frozen dessert is influenced by a complex relationship between formulation, size and number of ice crystals and air cells, overrun, fat destabilization, mix viscosity, freezing point depression, and other properties yet to be discovered. Frozen desserts can melt quickly and completely into a homogenous puddle or can even appear to not melt and hardly lose their shape over time. This property is something that is very noticeable to the consumer, as seen in a viral video about ice cream sandwiches (https://youtu.be/MoqDoet_RtA). Depending on how a product is consumed, in a dish, on a cone, or between two chocolate wafers, melt rate is an important attribute to consider.

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The melt rate of a frozen dessert is determined by the screen drip-through test (Goff & Hartel, 2013). The sample is allowed to equilibrate to 15°C for 60 min. An 80 g sample of ice cream is then cut from the pint and placed on a metal screen (three holes/cm). The screen is placed on a ring stand suspended over a beaker on a scale. As the ice cream melts, the weight of the ice cream that drips through is measured every 5 min. The test is allowed to continue until all the ice cream has dripped completely through the screen or for a total of 120 min. The rate of melting is determined by plotting the dripped through weight (g) against time (minutes) and finding the slope of the linear portion of the curve (Muse & Hartel, 2004). The test is conducted in a room with an ambient temperature of 21.5 ± 0.5C.

Goff, H. D., & Hartel, R. W. (2013). Ice cream (7th ed.). New York, NY, USA: Springer US.
Muse, M. R., & Hartel, R.W. (2004). Ice cream structure elements that affect melting rate and hardness. Journal of Dairy Science, 87, 1-10.

**Heat Shock and Temperature Abuse Studies**
Frozen desserts have numerous delicate structures, especially ice crystals and air cells, that can change dramatically during storage and especially when exposed to temperature fluctuations (Goff & Hartel, 2013). Putting a frozen dessert through cycles of temperature abuse, can give insights into how well product quality will hold up or deteriorate as it goes through the distribution chain, to store shelves, to a consumer's home freezer. Testing a product's resilience can also guide formulation changes or an appropriate best by date.

An accelerated shelf-life is simulated using a freezer cabinet that under goes regular temperature cycling. Product is put into a cabinet set to -13°C (8.6°F) that cycles between -10°C to -16°C (14°F to 3.2°F) every 30 minutes. A study typically lasts 4 weeks, but can extend as long as desired. Samples can be taken at various points during the study to measure changes in microstructural elements, most commonly to track changes in mean ice crystal size.

Goff, H. D., & Hartel, R. W. (2013). Ice cream (7th ed.). New York, NY, USA: Springer US.

**Rheology**
Rheology is the measure of how a substance deforms or reacts to to a force. Given the complex structure of frozen desserts, there are many ways to measure these phenomena. We can help select or design a test that is right for your application.

Common tests include: mix viscosity, temperature ramps (structure deformation with temperature change), frequency or strain sweeps (structure deformation with strain/stress).

We have a DHR-2 rheometer (TA instruments) complete with multiple geometries for a variety of applications. We have cooling and heating capabilities that allow for testing from -15°C to ~40°C (5°F to 104°F) for frozen desserts.

**Sensory Evaluation**
Consumer evaluation and testing can be a great way to understand how your product will be received and perform in the market. We have capabilities to run a number of panel types and will design a test that is right for your application.

**Tests available**:
Hedonic consumer panel
Triangle test

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Duo trio test
Paired comparison
In-home use tests
Focus groups
Trained descriptive panel analysis

**Basic product specs**
If you need to know some general composition and quality attributes of internal or competitor samples, we can give you a basic profile using these methods:

Total milk fat determination- Babcock method
Moisture/Total Solids- CEM Smart System 5
pH- pH Meter
Microbial analysis- inquire for specific capabilities
Freezing point depression- differential scanning calorimetry

Schedule a meeting to discuss your unique opportunity. Quotes are available on request.

Contact: (Dieyckson Freire, FDC Director) dfreire@wisc.edu