APPLICATION NOTE

Thermal Analysis

Characterization of Fats in Cookies Using Power Compensation DSC

Introduction

Differential scanning calorimetry (DSC) is a useful technique for the characterization of food products, including:

- The gelatinization and staling (retrogradation) behavior of starches
- Polymorphism of fats such as cocoa butters and chocolate
- Effects of moisture content or absorbed moisture
- Aging effects
- Protein denaturation
- Determination of fat content or solid fat index (SFI)

The processing and handling behavior of food fats has been found to depend upon the solid-to-liquid fat ratio in the food sample. Many rheological or flow properties, and their resultant effect on the texture of the final product, stem from this fat ratio index.

The study of the fat content and the nature of the fats of foods is becoming increasingly more important due to health considerations, especially with regards to the level of solid fats, saturated fats and trans fats in food products. There is a variety of fats with different levels of solid fats available in food products. An example of this is the Oreo® Cookie where there is the regular Oreo® and the reduced fat version. There are also Oreo®-like cookies with no solid, hydrogenated fats present.

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For the Better

With the current customer concerns with low fat and no-trans fat foods, an opportunity for fraud has been created by mislabeling foods that contain cheaper and less healthy fats. Fats are very complex materials and the analysis can be difficult for many reasons. Part of this complexity is fats can exist in both crystalline and amorphous forms. This can be further complicated by the presence of polymorphic melting forms of the given fat. A polymorph is an unstable melting form and this can be controlled by processing. Different polymorphic forms are sometimes desired in order to obtain a certain desired texture for the fat. For example, the fat used in chocolates, cocoa butter, has six polymorphic forms and only one of them gives the "melting in your mouth" feel to foods.

The successful analysis of fats in foods requires a DSC with high sensitivity and high resolution. The resolution performance aspect is important to be able to separate out the glass transition (Tg) and the different melting events associated with fats and possible polymorphic forms. The DSC with the best resolution and sensitivity performance available is the Power Compensation DSC from PerkinElmer.

Power Compensation DSC

The ideal DSC for the characterization of foods and fats is the Power Compensation or double furnace DSC. The very low mass furnaces provide low thermal inertia and the fastest response time of any DSC instrument on the market. This allows for the best peak definition and separation of overlapping peaks of any commercially available DSC. The Power Compensation DSC uses two independently controlled, ultra low mass furnaces (mass of 1 g) in the design of the DSC cell. The very low mass provides low thermal inertia and a very fast DSC response time, which is critical for high resolution.

In contrast, heat flux DSCs or Boersma DTAs, with their more massive furnace or those using a large silver block, have a more sluggish responsiveness. This translates to a higher inherent thermal inertia and a much slower DSC response time. The resolution from DSC instruments with a large mass furnace is much poorer than with the Power Compensation DSC. Some instrument companies attempt to correct for the problems caused by using a large silver block with algorithms. These algorithms try to adjust the data to account for the slower response time of that kind of DSC cell. However, there are concerns with such treatments since this alters the actual heat flow results. The Double Furnace or Power Compensation DSC provides the true sample response based on actual DSC hardware rather than mathematical manipulation of results.

In this study, the fats associated with three different fillings of cookies were assessed:

- Regular Oreo®
- Reduced fat Oreo®
- Oreo®-like cookie with no hydrogenated (solid) fats

Experimental

The following experimental conditions were used to analyze the fillings of the three different cookies.

Experimental Conditions	
Instrument	Pyris™ Power Compensation DSC
Cooling	Intracooler II
Sample Pan	Open aluminum pan
Sample Mass	Approximately 11 mg
Temperature Range	-60 °C to 100 °C
Heating Rate	20 °C/min
Purge Gas	Nitrogen

The DSC was calibrated for temperature and enthalpic responses using high purity indium metal.

Results

Displayed in Figure 1 are the DSC results generated for the filling of the regular Oreo® cookie. The plot shows the DSC heat flow as a function of sample temperature. The sample yields a complex DSC thermograph due to the nature of the fats and polymorphic forms associated with the as-received Oreo® cookie filling. The fat undergoes melting beginning at –18.8 °C. A series of melting peaks are observed at –2.6 °C, 16.1, 28.0, 35.1 and 44.5 °C for the fats in the filling. The complex melting spectrum reflects the occurrence of polymorphic forms due to the particular processing conditions used to produce the Oreo® filling. The total heat of melting of the cookie filling is found to be 28.2 J/g. The DSC results show that a significant amount of the fat in the Oreo® cookie filling melts above room temperature and this behavior is due to the hydrogenated fats in the filling.

The high resolution response of the Pyris™ Power

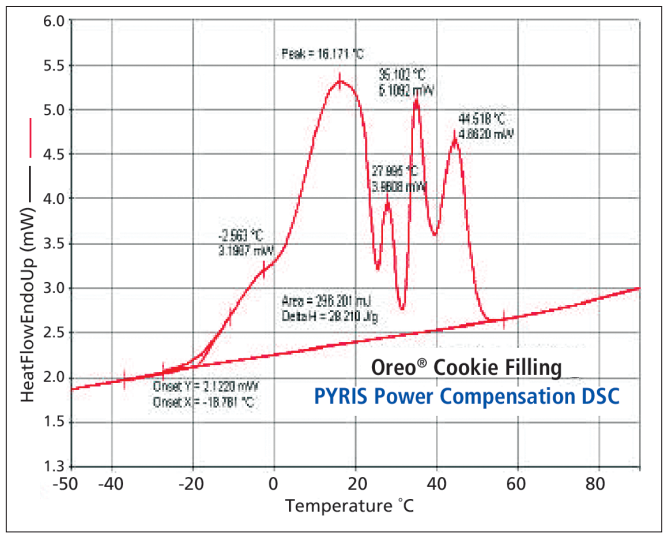

Figure 1. DSC results for regular Oreo® cookie filling.

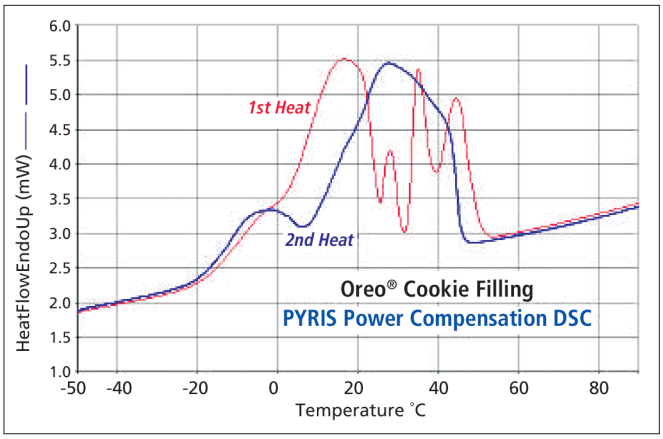

Figure 2. Comparison of first and second DSC heats for filling.

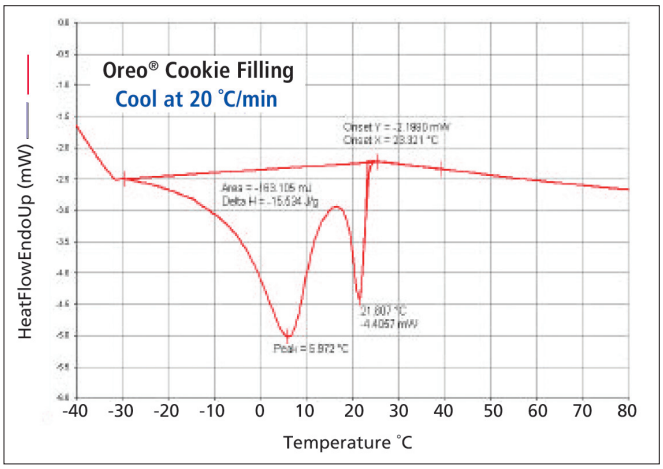

Figure 3. DSC cooling results for Oreo® cookie filling.

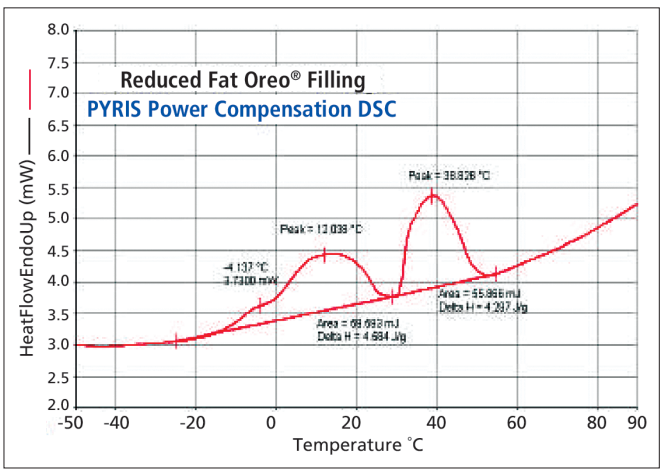

Figure 4. DSC results for reduced fat Oreo® cookie filling.

Compensation DSC is necessary to be able to detect the various peaks associated with the polymorphic forms of the cookie filling, even at the fast heating rate of 20 °C/min.

Heat flux DSC instruments, especially those using a massive silver block, would tend to smear out the various transitions associated with the fats and polymorphic forms in the filling making the characterization less definitive and incomplete. With the Pyris™ Power Compensation DSC, all of the important transitions, both large and small, are observed.

The regular Oreo® cookie filling was cooled back to –60 °C and then reheated at 20 °C/min and the results of the reheat experiment are displayed in Figure 2. The filling now exhibits a very different thermal response and this reflects the differences due to thermal history. Melting, cooling and reheating produces a new morphology or structure in the fat. DSC is a valuable technique for studying the effects of thermal history on fats and their polymorphic forms.

The Power Compensation DSC also provides excellent results during cooling experiments. Fats yield well-defined crystallization events during cooling and this information is valuable for characterization and process control purposes. Displayed in Figure 3 are the DSC results generated for the Oreo® cookie filling by cooling from 100 to –60 °C at a rate of 20 °C/min. The crystallization of a fat component occurs sharply at 23 °C. The cooling data reflects two different crystallizable fats in the cookie filling.

The DSC results obtained for the reduced fat Oreo® cookie filling are displayed in Figure 4. This filling undergoes multiple melting transitions at –4.1 °C, 12.0 and 38.8 °C. Although the reduced fat filling contains solid or hydrogenated fats, the amount of fat is reduced as is demonstrated by the lower values of the heats of melting. The first and second melting transition yield heats of melting of 4.6 and 4.3 J/g, respectively, for a total heat of 8.9 J/g. This is much lower than the value obtained for the regular Oreo® cookie filling (28.2 J/g).

The Oreo®-like cookie contains a filling with no hydrogenated or solid fats and the DSC results for this sample are shown in Figure 5. The melting of the fat in this filling completely takes place below 0 °C with melting peaks occurring at –26.0 and –17.3 °C. The total heat of melting for the non-hydrogenated fat filling is 16.1 J/g.

Displayed in Figure 6 is a direct overlay of the DSC results obtained for the three different cookie fillings. The differences in the melting responses of the fats comprising the fillings are very evident from these results.

Summary

The Power Compensation or double furnace DSC yields excellent results for foods including the fat nature and content. The fast responsiveness of the Power Compensation DSC provides the highest possible resolution and this is critical for characterizing the various polymorphic melting forms associated with fats in foods. Even at the fast heating rate of 20 ˚C/min, the Power Compensation DSC is able to provide high resolution to be able to detect the multiple melting peaks of the polymorphic forms for the as-received Oreo® cookie filling. This data is important for the full characterization of the food fats, quality assurance, product uniformity and process control purposes.

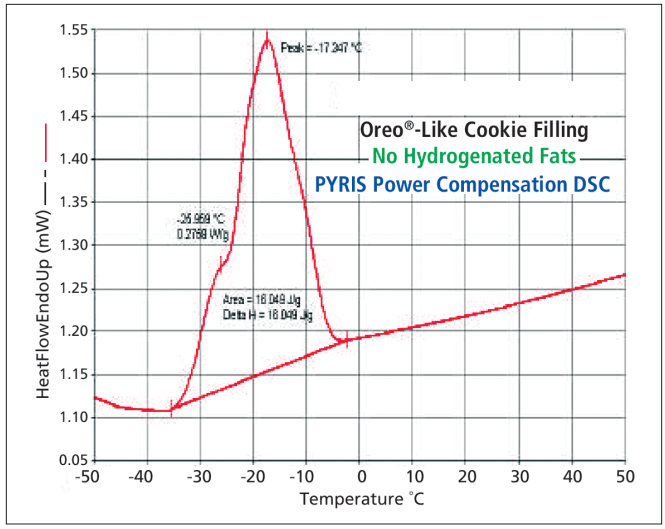

Figure 5. DSC results for Oreo®-like cookie filling (no hydrogenated fats).

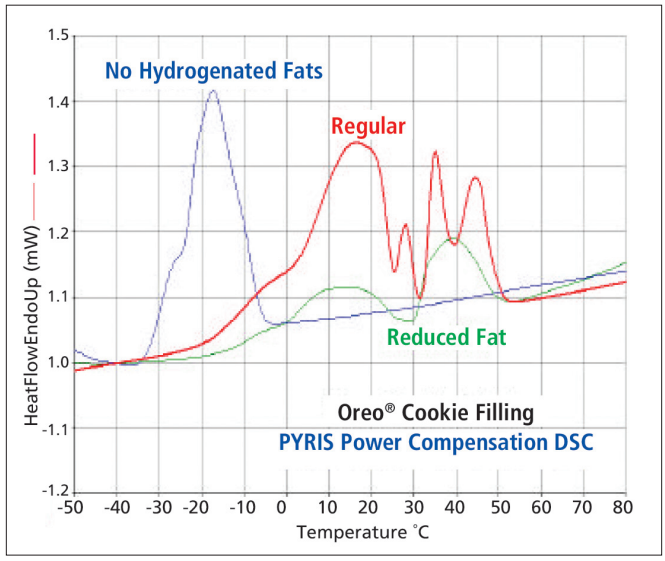

Figure 6. Overlay of DSC results for regular Oreo®, reduced fat and non-hydrogenated fillings.

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