

## APPLICATION BRIEF

## TA no.85

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## Estimating the Solid Fat Content of Chocolate and Butter Products

Using DSC to investigate of the taste of edible fats

## 1. Introduction

The safety and quality of food products are analyzed from various perspectives. Fat, one of the main constituents of food products, has a low melting temperature and many crystal forms. Factors such as changes in crystal formation when fat is melted and the extent of solid fat at room temperature determine characteristics such as hardness or fluidity. Manufacturing and storage environment are important considerations in creating fat-based foods with the desired crystal form or solid fat content. To evaluate these characteristics, Differential Scanning Calorimeters (DSC) is used to measure the melting temperature of fats.

In this brief, chocolate, butter and margarine are measured as examples of food products whose main constituent is fat.

## 2. Measurements and Results

### 2.1 DSC measurements of chocolate

Chocolate is a fat-based food product with crystallized cocoa butter from cacao beans as its main constituent. There are 6 known types of crystal polymorphs (Type I to VI) of cocoa butter. The higher the number, the more thermally stable the structure is. By analyzing the crystal polymorphs of cocoa butter, product characteristics such as heat resistance, storage stability and texture can be determined, which makes it possible to manufacture high-quality products.

#### 2.1.2 Measurement conditions

Samples were commercially-available milk choco-

late, high-cacao chocolate (with a cocoa content of 86%) and creamy chocolate.

Measurements were performed using a DSC6220 differential scanning calorimeter. The heating rate was 10°C/min and the temperature range was -50 to 60°C. The sample weighed 5mg and was measured in an open aluminum pan.

#### 2.1.2 Measurement results

Figure 1 shows the DSC curves for all samples. The creamy chocolate showed endothermic peaks at around 10°C and 28°C. Melting started at a lower temperature than the other types of chocolate. This means that the majority of the fat is melted at room temperature (around 25°C) and explains the distinctive soft texture of creamy chocolate.

On the other hand, the milk chocolate and high-cocoa chocolate had melting peak temperatures of around 33°C and 34°C, respectively. This means that the majority of the fat melts above room temperature. These types of chocolate are hard when placed in the mouth and then gradually melt.

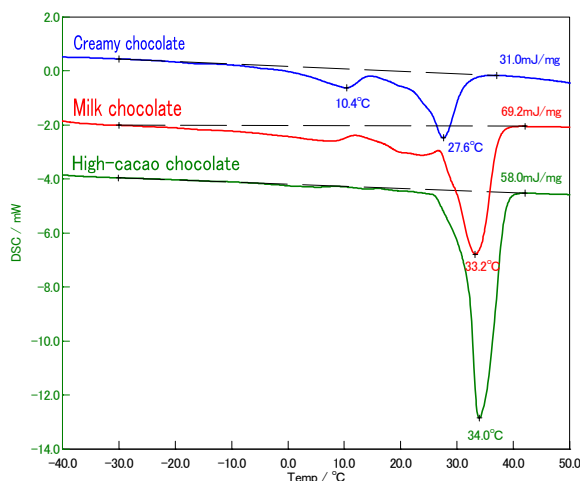


Figure 1 DSC curves for Chocolate (1st Run)

It is known that cooling conditions change the crystal structure of melted cocoa butter. The 3 types of chocolate were cooled to  $-50^{\circ}\text{C}$  at  $10^{\circ}\text{C}/\text{min}$  to crystallize them. Figure 2 shows the DSC curves of 2nd heating for the 3 types of chocolate.

By comparing the 1st and 2nd heating curves, it can be seen that the melting temperature of all samples lowered and that a portion of the chocolate was still melted at room temperature. This indicates that melting chocolate once greatly changes the melting temperature.

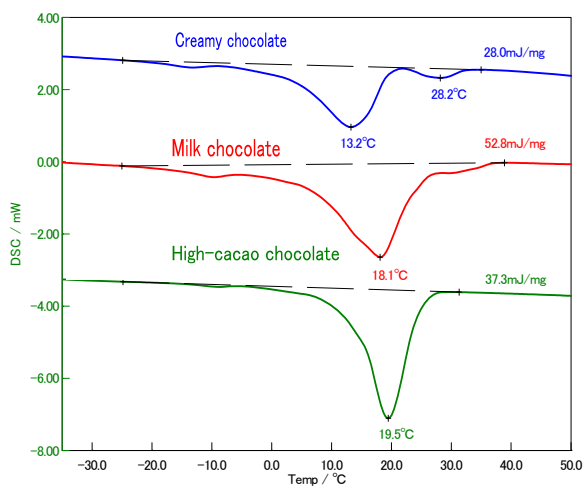


Figure 2 DSC curves for Chocolate (2nd heating)

Solid Fat Content (SFC) is another method to evaluate fat and is generally performed using pulse nuclear magnetic resonance (pulse NMR). This method is important for evaluating the softness and snappiness of fat.

Using DSC to investigate the integral curve for melting is an easy way to perform an evaluation comparable to SFC curve created by pulse NMR.

Figure 3 shows the DSC curve and the integral curve for creamy chocolate and the integral curve for melting. A 2-stage integral curve corresponding to the melting peaks was obtained. The melting integral is considered equivalent to SFC so the proportion of melted fat at any temperature can be obtained from the melting integral curve.

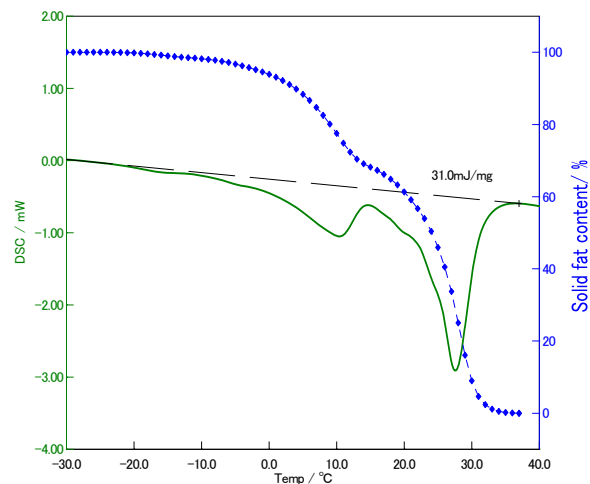


Figure 3 The DSC curve and the Melting integral curve for Creamy Chocolate (1st heating)

Figure 4 shows the melting integral curves for the 3 types of chocolate. It can be seen that the proportion of solid fat in the creamy and milk chocolate gradually decreased from the minus temperature range. On the other hand, the proportion of solid fat in the high-cocoa chocolate did not change very much up to room temperature but started to decrease rapidly from around  $30^{\circ}\text{C}$ .

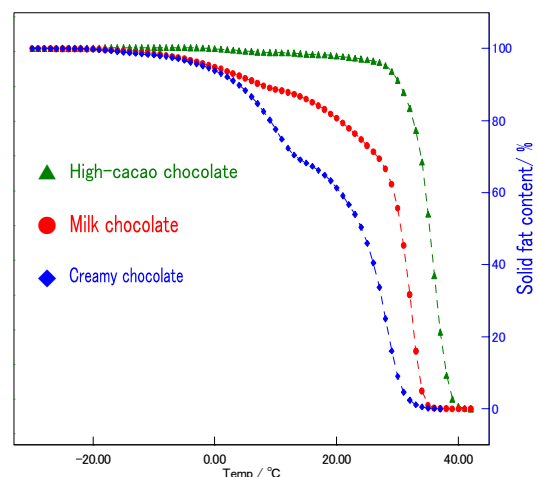


Figure 4 Melting integral curves for Chocolate (1st heating)

## 2.2 DSC Measurement of Butter and Margarine

Butter is made by separating fat content in raw milk and then churning it to harden it and its principal constituent is saturated fatty acid.

On the other hand, margarine is made by emulsifying plant or animal fat and its principal constituent is unsaturated fatty acid. While butter and margarine have different hardness and cooking purposes, their fat and water contents are roughly equal. Recently, fat-based food products that combine the good points of both products have been released.

### 2.2.1 Measurement conditions

Three samples were measured: commercially-available butter, margarine and 1/3 fat butter. Measurements were performed using a DSC6220 differential scanning calorimeter. The heating rate was 10°C/min and the temperature range was -50 to 50°C. The samples weighed 5mg and were measured in an allogene-treated aluminum sealed pan.

### 2.1.2 Measurement results

Figure 5 shows the DSC curves for butter, margarine and 1/3 fat butter. Between 0 and -5°C, an endothermic peak appeared due to the melting of water. Fat melting peaks for butter were detected as endothermic peaks at -25°C, 15°C and 30°C. For margarine, peaks appeared at -25°C and 30°C but, unlike butter, a peak was not found at 15°C. This data indicates that the peak at 15°C is the saturated fatty acid peak because butter is the only sample that contains it.

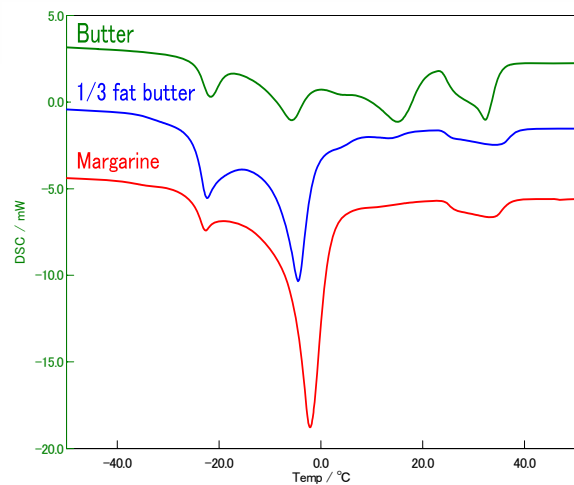


Figure 5 DSC curves for Butter, Margarine and 1/3 fat butter

Overall, the 1/3 fat butter DSC curve was the same as the curve for margarine. However, the peak at 15°C that appeared in the butter results was also detected here. (See Figure 6)

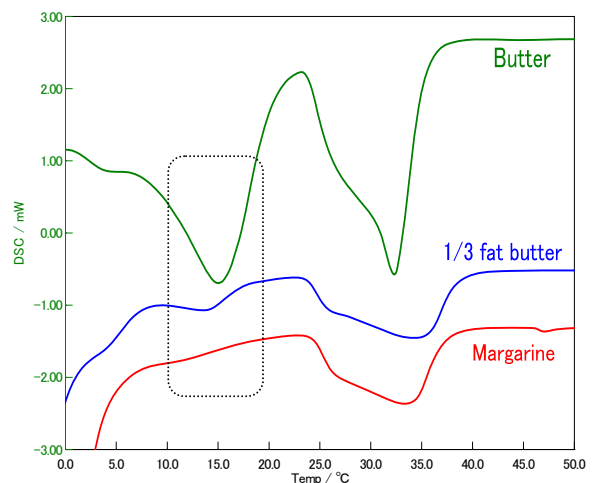


Figure 6 Enlarged View of DSC curves for Butter, Margarine and 1/3 fat butter

The solid fat content of butter and margarine was also investigated. Figure 7 shows the results of the melting integral curve from the DSC curves.

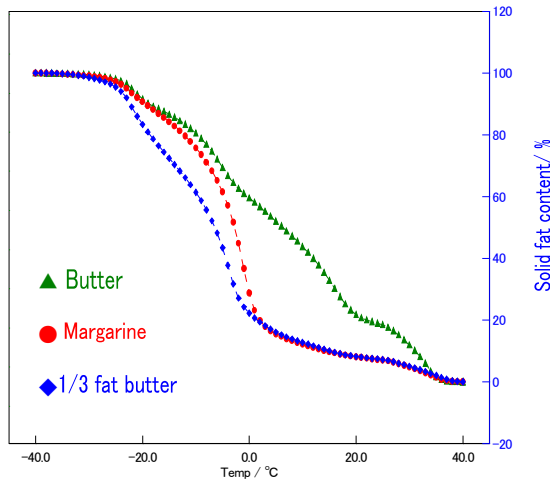


Figure 7 Melting integral curves for Butter, Margarine and 1/3 fat butter

For all 3 measurement samples, melting caused solid fat content to decrease starting from around  $-25^{\circ}\text{C}$ . By around  $0^{\circ}\text{C}$ , solid fat content was only 20% for margarine and 1/3 fat butter.

Furthermore, the data shows that the solid fat content of the 1/3 fat butter decreased more rapidly than the margarine in the minus temperature range, despite the fact that it contains butter, which resists decreases in solid fat content. This data shows that its solid fat content is already low when refrigerated so it is soft and ready to use when it is removed from the refrigerator.

By using DSC to understand the relationship of fat melting and solid fat content, it is possible to investigate characteristics such as creaminess and spread ability.

### 3. Conclusion

The solid fat content of fat-based food products was investigated by using DSC to measure the fat melting peaks and then obtaining integral curves. Pulse NMR is usually used to investigate solid fat content but DSC can easily measure it from the minus temperature range.

Furthermore, this method is useful for product development and quality evaluations because data can also be obtained about the crystal structure of measurement samples with different melting behaviors or thermal histories.