

Measurement of Fats with DSC

Oil and fat products are generally mixtures of multiple components, and the melting process is complex. This investigation, starting from minus temperatures, is effectively conducted using DSC analysis. In that case, aside from the requirement to determine such characteristics as melting temperature and heat of fusion, it is also possible to perform heat treatment

due to precise temperature control, and to investigate the heat history and apparent changes in crystalline structure. Here, lipstick and chocolate were analyzed using an electric cooling DSC. In particular, measurement of chocolate was performed using repetitive heating and cooling cycles.

The electric cooling DSC is a DSC system that allows continuous heating and cooling at user-defined speeds over the temperature range of -50°C to 500°C , without requiring the replenishment of liquid nitrogen or other refrigerant during the analysis. This instrument is used for performing transition and melting, crystallization and other measurements on various types of samples, including fats, drugs, cosmetics and plastics, etc.

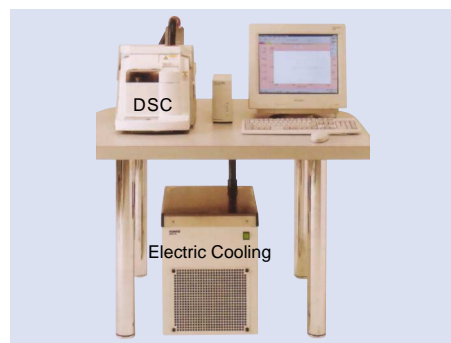


Fig. 1 Electric Cooling DSC

■ Measurement of Lipsticks

Lipstick consists of a mixture of wax, fats and oils in which dyes are dissolved or distributed, and in particular, the types and amounts of wax and fats influence the heat resistance, strength (cracks) and feel, etc. In addition, since the temperature environment to which lipstick is subjected is complex, changes in the crystalline form of the wax could alter the initial characteristics. Here, 3 types of commercial lipstick were analyzed, and the results were

compared. The melting peak of sample A was observed over the temperature range of 10°C - 80°C , and relatively high heat resistance is expected. Since sample B shows a large melting peak in the vicinity of -40°C to 0°C , it is expected to maintain a soft, smooth consistency even at room temperature. Sample C shows melting between the melting temperatures of A and B, so its heat resistance and softness will display intermediate characteristics.

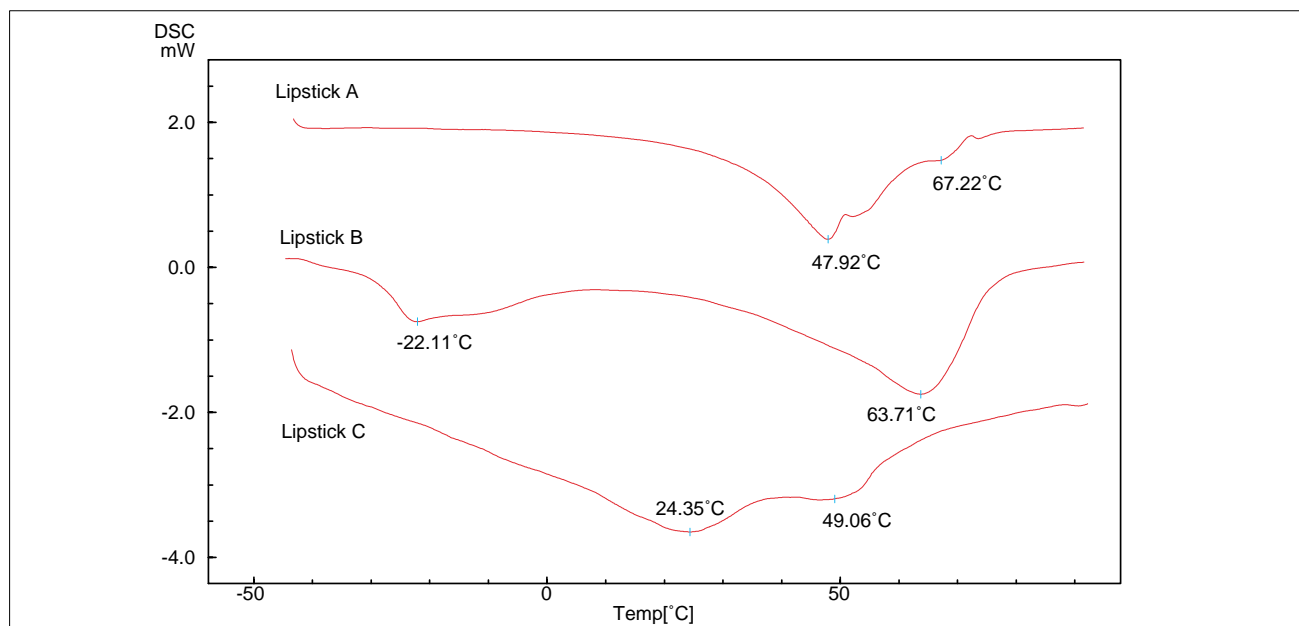


Fig. 2 Comparison of Three Lipsticks

Measurement of Chocolate

The melt characteristic of chocolate as an index of its quality is thought to vary depending on the heat it was subjected to during its manufacture and storage. To obtain chocolate of constant quality, it is necessary to examine the heat history and changes in crystalline form. With the DSC, it is possible to apply various types of heat-treatment to the sample placed within the instrument, and the melting patterns can be compared. Fig. 3 shows a comparison of data in which commercial chocolate was heated at 3°C/min and after being cooled within the instrument at 3°C/min, it was once again heated at 3°C/min. The two heating periods are compared. It is clear that the crystals changed, and differences in the melting peak temperature and peak shape appeared.

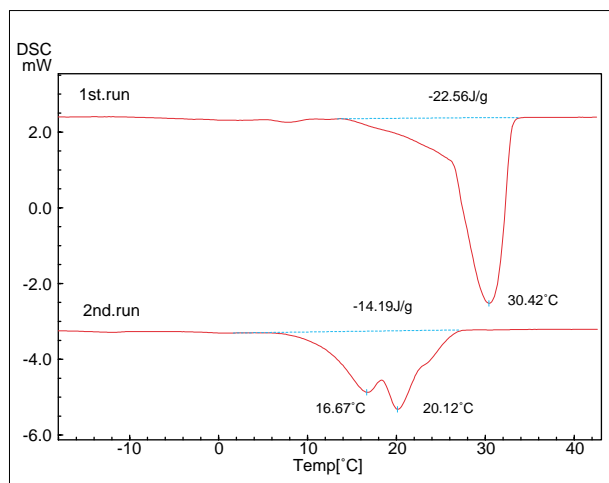


Fig. 3 Melting of Chocolate

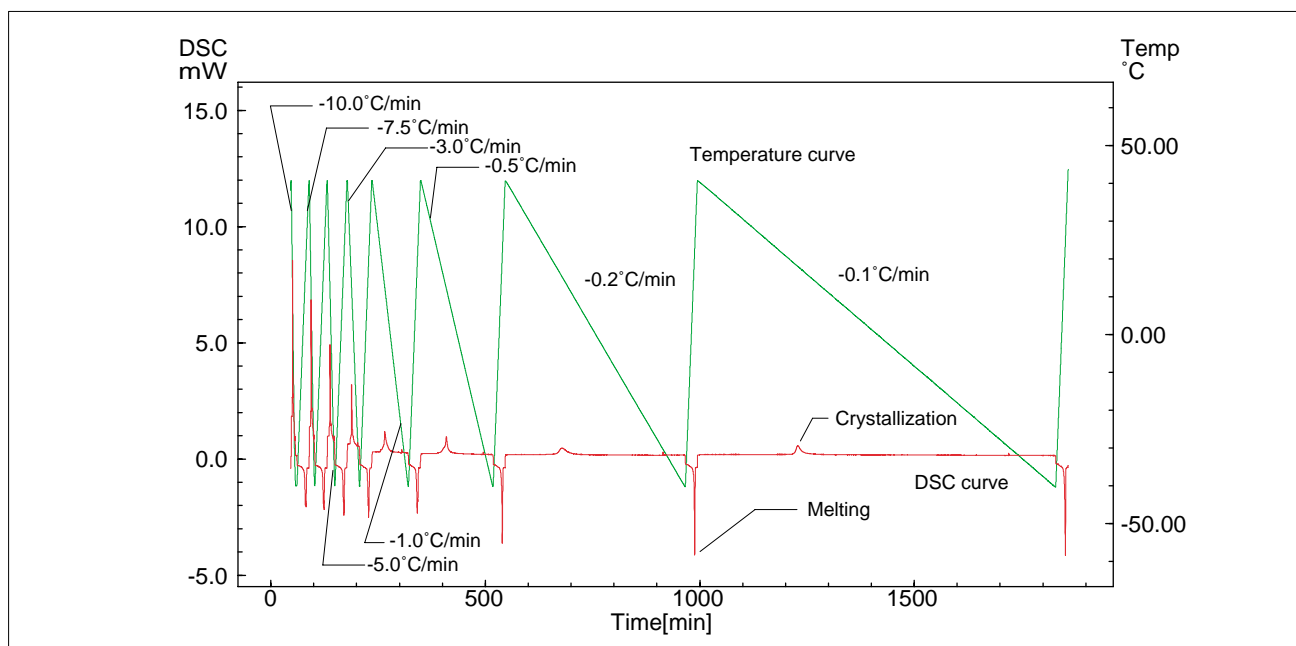


Fig. 4 Continuous Heating-Cooling Process

Fig. 4 records the continuous cooling-heating-cooling cycle used to investigate the cooling speed and changes of crystalline form in chocolate. Heating was conducted at 3°C/min, and the cooling rate was changed within the range of 10°C/min – 0.1°C/min. With the electric cooler DSC, it is possible to perform cooling measurement automatically over long periods without the need for replenishing refrigerant.

Fig. 5 shows the DSC curve profile data of the heating intervals only from Fig. 4, rearranged for comparison. It is shown that the crystalline structure was altered due to the cooling rate, and melting peak temperature and shape were changed.

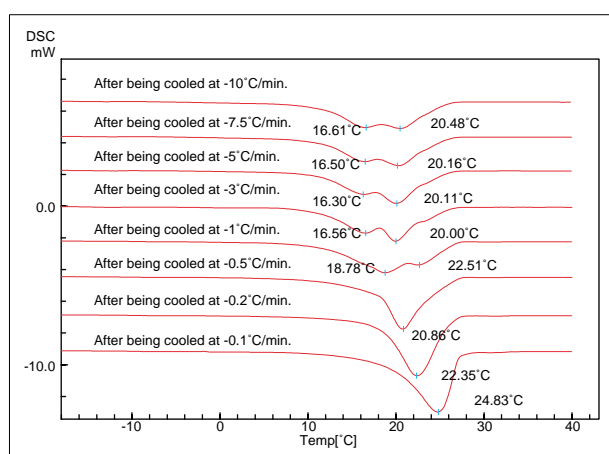


Fig. 5 Dependency on Cooling Rate