APPLICATION NOTE

Thermal Analysis

Tg of Semisolid Material by Isothermal Frequency Sweeps in Compression

Summary

This application note demonstrates the ability of DMA to investigate glass transitions in semisolid viscous materials using a PerkinElmer® DMA 8000. The sample chosen was honey, but the technique is suitable for all materials of this type. Instead of scanning temperature as in a conventional DMA experiment, multiple frequencies were used at three discrete temperatures. A clear relaxation is observed in both the modulus and tan δ against frequency plots. This note also demonstrates the use of a little used geometry, compression, to obtain results from the sample.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. The glass transition (Tg) is a key process in any material, and can be observed with ease by DMA. In a DMA experiment, the temperature is not the only variable that can be changed by the user. The frequency of oscillation of the sample can also be changed, allowing the user to both investigate frequency dependencies in the material and mimic the conditions of actual use. If various frequencies are run at several temperatures, the instrument results (modulus and tan δ) can be plotted against frequency instead of temperature.

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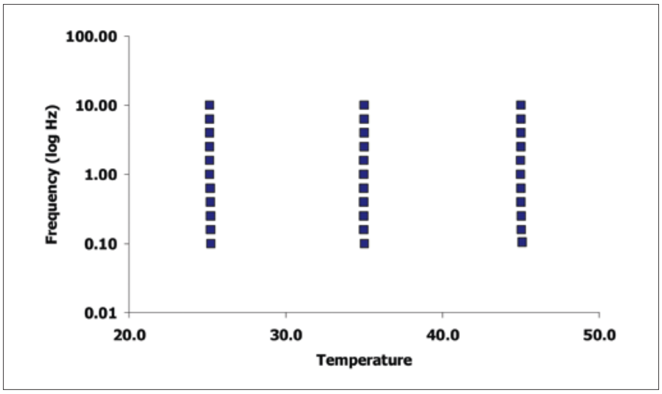

Figure 1. Temperature vs. frequency.

DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and the modulus and tan δ can be calculated. Tan δ is the ratio of the loss modulus to the storage modulus. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition.

Honey is a partially or fully amorphous solid that is composed of predominantly sugar with some pollen and water. Dextrose and levulose are the main sugar components but smaller quantities of sucrose, maltose and higher sugars can be found. This makes it a complex formulation, but as the sugar components are predominantly amorphous, strong relaxation events are expected.

Experimental

Isothermal frequency scan of honey.

A 1 mm thick sample of honey was applied to the compression clamps of the DMA. Modulus and tan δ were collected at 25, 35 and 45 °C at a variety of frequencies.

Equipment	Experimental Conditions	
DMA 8000	Sample:	Supermarket brand set honey
	Geometry:	Compression (10 mm x 1 mm)
	Temperature:	25, 35 and 45 °C
	Frequency:	0.1 to 10 Hz (11 discrete frequencies)

Results and conclusion

Figure 1 shows the temperature against log frequency plot where data was collected. Note, the frequencies chosen are approximately of equal spacing when plotted on a log plot. Figure 2 shows storage modulus, loss modulus and tan δ against frequency.

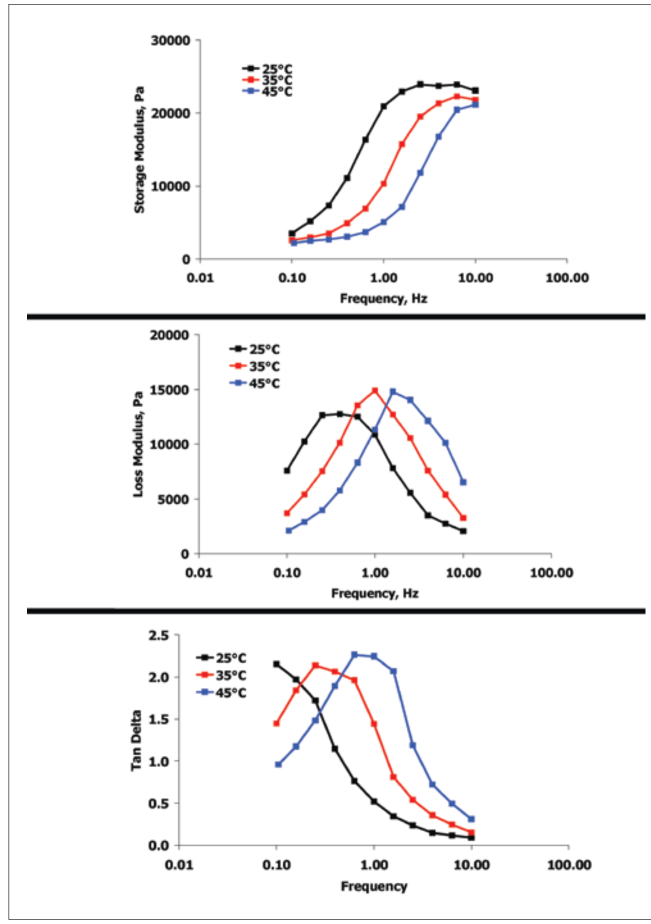

Figure 2. Storage modulus, loss modulus and tan δ vs. frequency.

The frequency dependant response of honey is clear from all the data obtained.

The storage modulus plot shows that as the frequency is increased, the modulus also increases which is a typical property of viscous materials. With increasing temperature, a higher frequency is required to achieve an equivalent modulus observed at a lower temperature.

The loss modulus plot shows the glass transition of the material clearly as a peak. The peak shows the point at which the material is most viscous and loses most energy as a result of passing through the glass transition.

The tan δ plot shows a similar result to the loss modulus. The peak value corresponds to the glass transition. The trend is the same in that the frequency of the glass transition increased with the temperature of the sample.

These data show the utility of the DMA technique to be able to investigate glass transitions, not only as a function of temperature, but also as a function of frequency. The sample chosen for this demonstration, honey, displayed typical frequency dependence behavior when investigated in compression geometry.

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