Investigation of Amorphous Sucrose Using Material Pockets and Humidity Generator

Summary

Sucrose is a well known material used for a variety of applications. In its simplest form it is used as sugar in cooking or for coffee. It is also used as an excipient in some pharmaceutical preparations. This study shows how the amount of water present in a sugar sample will greatly affect its mechanical properties. Using the Triton Technology Humidity Controller linked to the PerkinElmer® DMA 8000, it is shown how the Tg of amorphous sucrose changes when exposed to relative humidity. In addition, a comparison of a very dry sample of sucrose with one exposed to lab atmospheric moisture is shown.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. This fact, until now, has not been exploited for powdered materials due to the difficulty in handling powders. Some work has been done with dilatometry, but with the development of the Material Pocket, it has become easier for powdered materials to be investigated in a DMA 8000.

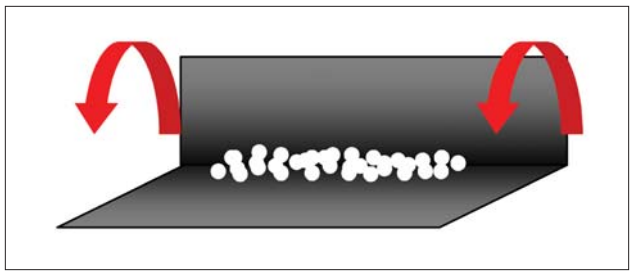
DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and the modulus and tan δ can be calculated. Tan δ is the ratio of the loss modulus to the storage modulus. By measuring the phase lag in the 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.

The following example shows sucrose to be very hygroscopic, at least in the initial adsorption of water. It also shows the effect of moisture on the glass transition temperature.

Results and conclusion

Figure 1 shows a simple thermal scan of the slightly damp sucrose sample. The experiment was performed at two frequencies. The initial peak at about 75 °C is frequency dependant indicating a relaxation, in this case the Tg. The second broader peak is not frequency dependant and can be attributed to the recrystallization of the amorphous material.

The response when the relatively dry sample of sucrose was exposed to 50% relative humidity is shown in Figure 2. The tan δ curve starts to increase as the sample is plasticized and the material starts going through its Tg. The temperature was chosen to be just below the Tg of the unhumidified sample (shown in Figure 1).



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precisely.

Experimental

Thermal scan of sucrose.
Approximately 50mg of sucrose was loaded into a Material Pocket and the pocket was mounted in the DMA 8000. Three samples were investigated. (a) A very dry sample. (b) A dry sample exposed to laboratory atmosphere for 24 hours (c) Same as sample "b" but run at 50% RH.

Equipment	Experimental Conditions	
DMA 8000	Sample:	Spray dried sucrose
Fluid Bath	Geometry:	Single Cantilever Bending
Humidity Generator	Support:	Material Pocket
Circulator	Temperature:	20 °C to 200 °C at 5 °C min^{-1}
	Frequency:	1.0 Hz
	Humidity:	0 and 50%

Figure 3 shows the comparison between the three samples of sucrose. Sample A was very dry and shows the Tg at the highest temperature. Sample B was exposed to a modest amount of water and shows a small reduction in the Tg and Sample C was run while exposed to 50% relative humidity. A clear trend in the plasticizing of the material is observed as a decrease in Tg as a function of water exposure. The recrystallization peak is also shown to decrease as a function of water proving that this process is water mediated.

These data demonstrate that using the DMA 8000 in conjunction with the Material Pocket and the Humidity Controller can give information on the relaxation processes and the influence of water for amorphous powdered materials.

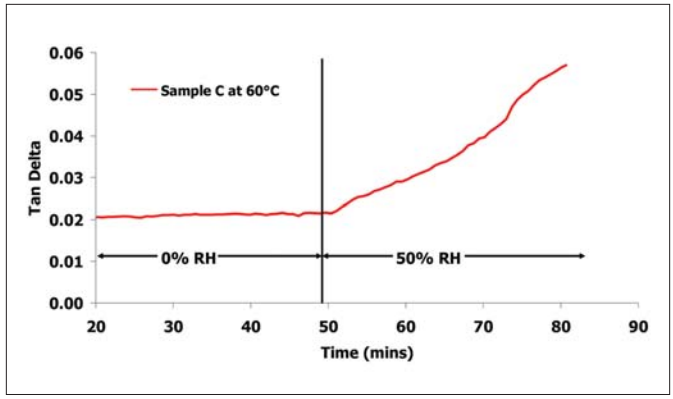

Figure 2. Dry sucrose sample exposed to 50% relative humidity.

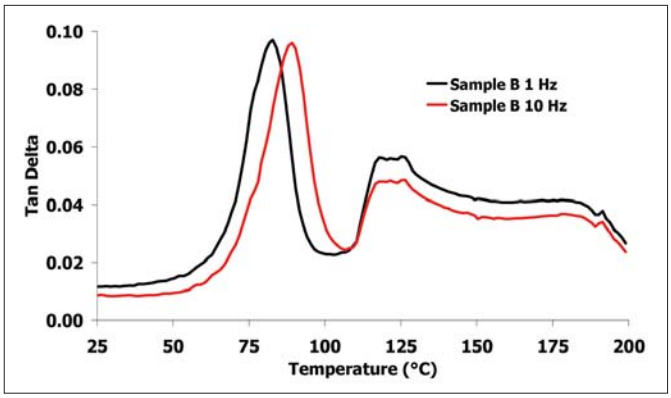

Figure 1. Thermal scan of damp sucrose sample.

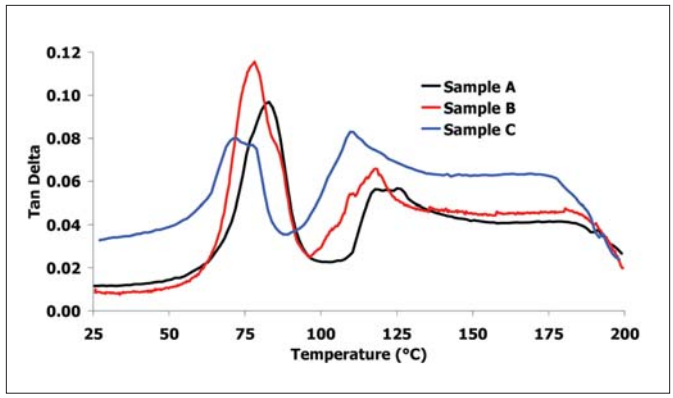

Figure 3. Comparison of all three sucrose samples.

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