DETA Dielectric Thermal Analyser

Application Note
Dielectric Thermal Analysis of Sucrose Powder

This is another example of the ability of the DS6000 DETA to examine powdered materials such as sugars. The Glass Transition (Tg) is a very important characteristic of foodstuffs affecting the texture of many foods in their final state. Sugars such as lactose are also very important in the pharmaceutical industry. It is therefore useful to be able to examine such materials with different approaches in order to fully understand the Tg process. With techniques such as differential scanning calorimetry (DSC), the Tg process is energetically weak and hard to fully characterise. This is not the case with relaxation spectrographic techniques such as DETA.

Introduction

Dielectric **Thermal** Analysis (DETA) is a powerful analytical tool for studying relaxation processes in materials or behaviour of polar species within a material. The glass transition (Tg) is a key process in any material, and can be observed with ease by DETA for many materials. This technique provides very revealing information about these relaxations through the tan δ vs temperature data. The form of the material can be anything from a thin film, sheet material, powder or a liquid.

Dielectric measurements are the electrical analogue of dynamic mechanical measurements. The mechanical stress is replaced by an alternating voltage across the sample (a.c.field) and the alternating strain becomes the stored charge (Q) in the sample. The sample in effect behaves as a simple capacitor. Q is always measured as its derivative dQ/dt = a.c.current.

The dielectric data is obtained from phase and amplitude measurements of current and voltage to resolve the components e* = Capacitance with sample/Capacitance with an identical air gap.

As in DMA, tan δ is the ratio of the loss factor(e″) to the storage component (e′ , dielectric constant or permitivity) . 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 size of this peak quantifies the amount of amorphous material present in the sample.

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Equipment

DS6000 DETA
1L ‘Mini’ LN_2 Cryo

Experimental Conditions

Sample: Freshly ground sucrose powder

Geometry: Cup and Plate 33mm diameter, 1mm thickness

Frequency 1 kHz

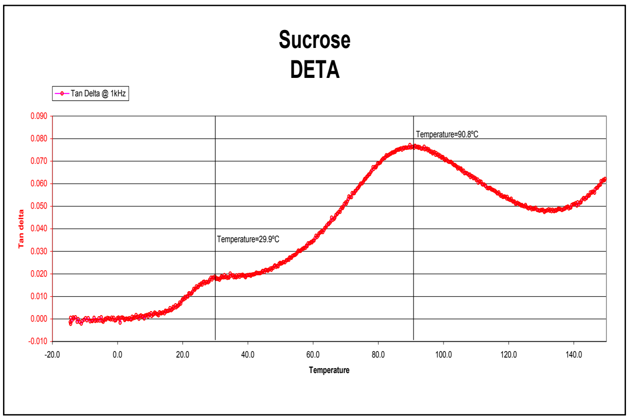
Ramp Rate: 2ºC/minute

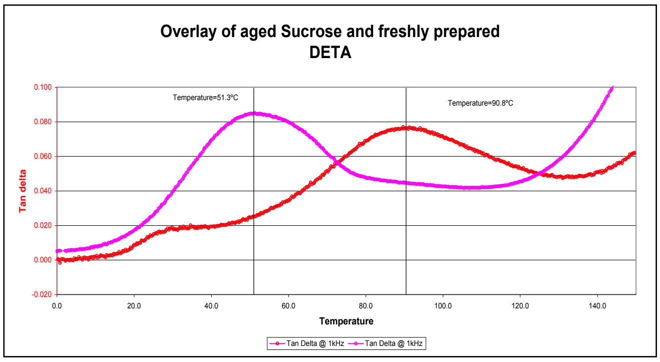
Experimental

Approximately 100mg of freshly milled sucrose was placed in the cup electrodes of the DS6000 DETA. The sample was run from around -20ºC. A second sample prepared in the same manner was exposed to ambient humidity for a day. This was then run in the same manner and compared with the first set of data.

Result and Conclusion

The data to the left was from the freshly prepared sample run immediately. It illustrates very clearly the relaxation processes in this particular material. Two processes are observed on this sample. One at 30ºC and another at around 90ºC maximum. The process at the lower temperature may be a plasticized form of the higher Tg observed. This could be due to humidity affecting the surface of the powder particles. The effect would be to lower the Tg both in temperature and amplitude as the material changed from the amorphous form to a crystalline form. On the other hand, it could be an entirely different material.





A way to check this would be to ‘age’ the powder and re-run. If the process was as previously described, the higher peak at 90ºC would gradually diminish towards the first peak observed. The data shown in pink in the overlay data adjacent, was produced on the aged sample as described above. This confirms the plasticization effect was occurring with this particular material.