The Glass Transition of Casein by Rapid Heat-Cool (RHC) DSC

INTRODUCTION

As a technique, interest has been growing in performing differential scanning calorimetry (DSC) at higher than typical (10 °C/min) temperature-scanning rates. This is because a variety of material characterization challenges exist that can benefit dramatically from rapid heating or cooling rate experiments. For example, the investigation of metastable states and time-dependent transitions would profit greatly from fast scanning rates. In general, higher scanning rates will also increase the heat flow sensitivity for subtle transitions although this benefit is usually tempered by the small mass requirement of the rapid scanning rates.

A DSC has been designed specifically for operation at high scanning rates – up to 2000 °C/min in heating with similarly high cooling rates.[1] Key technologies introduced by TA Instruments are essential to, and have been incorporated into the instrument known as Project RHC. For example, Tzero® technology improves the resolution and the sensitivity of the measured sample heat flow rates, especially for very weak effects, and improves the instrument baseline. Also, infrared heating, introduced in the Q5000IR TGA, provides a “massless” infrared heat source. Readers interested in further details on the instrument design should refer to reference 1.

This applications note reports on the detection of the glass transition in the casein. Casein is a phosphoprotein that accounts for nearly 80% of all protein in milk. It is also used as a binding agent in foods.

RESULTS and DISCUSSION

Figure 1 contains the TGA result of casein (Sigma-Aldrich) at 10 °C/min. up to 200 °C. The 5.5% weight loss detected between room temperature and 150 °C is due primarily to water evolution. The glass transition temperature of casein is approximately 100 °C, however, it is difficult to detect by classical DSC techniques because of the concurrent release of this water and the associated broad endotherm. Figure 2 contains an overlay plot of DSC scans of casein collected in the RHC instrument. For scans of 100 °C/min. or slower, the evolution of water from the sample overlaps the Tg and prevents its detection. As desorption and evaporation are kinetic events, scanning at faster rates will shift these processes to higher temperature. At elevated scanning rates it is possible to shift the water evaporation to a temperature well beyond the glass transition, thus separating the events in temperature and enabling the detection of the Tg. As seen in Figure 2, for scans at 500 °C/min. and above, the water evolution is elevated to higher temperatures and the casein glass transition is clearly resolved. Perhaps even more intriguing is that by using these high heating rates the Tg is measured with the water still present in the sample, in effect measuring the plasticized Tg. This data demonstrates the utility of the RHC in analysis of complex transitions.

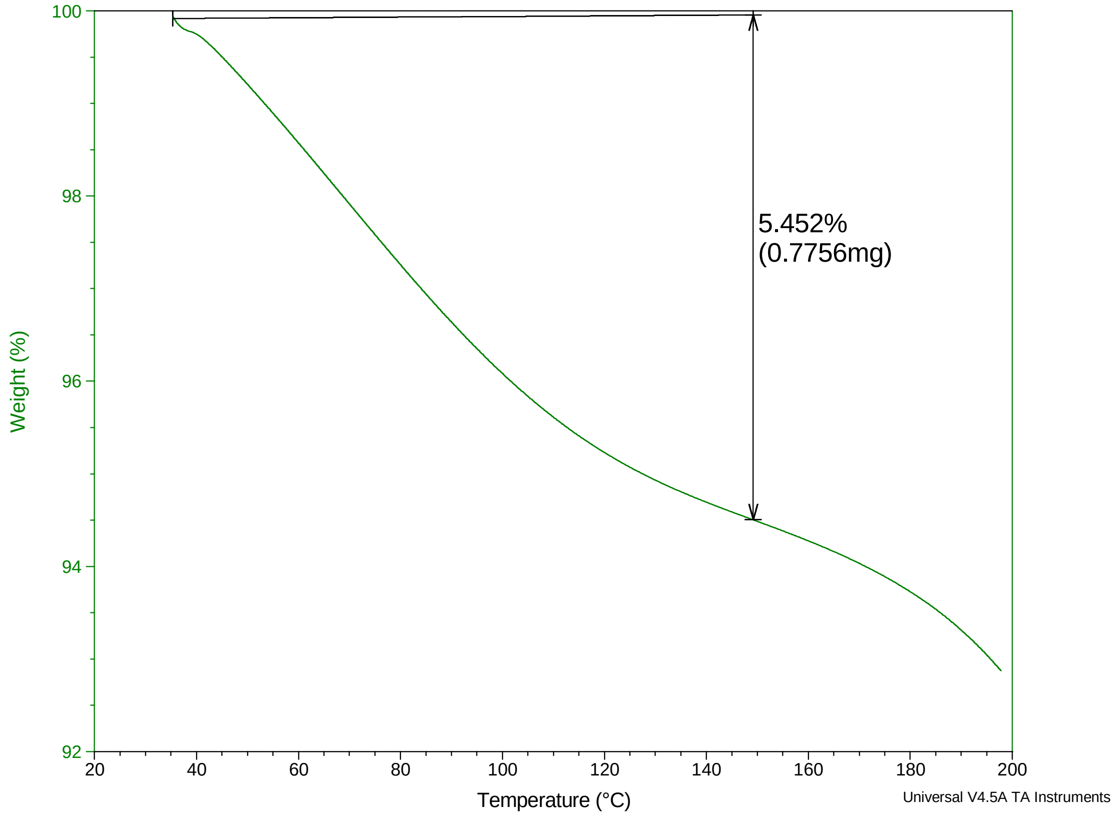

Figure 1 – TGA data of casein showing 5% loss due to water evolution.

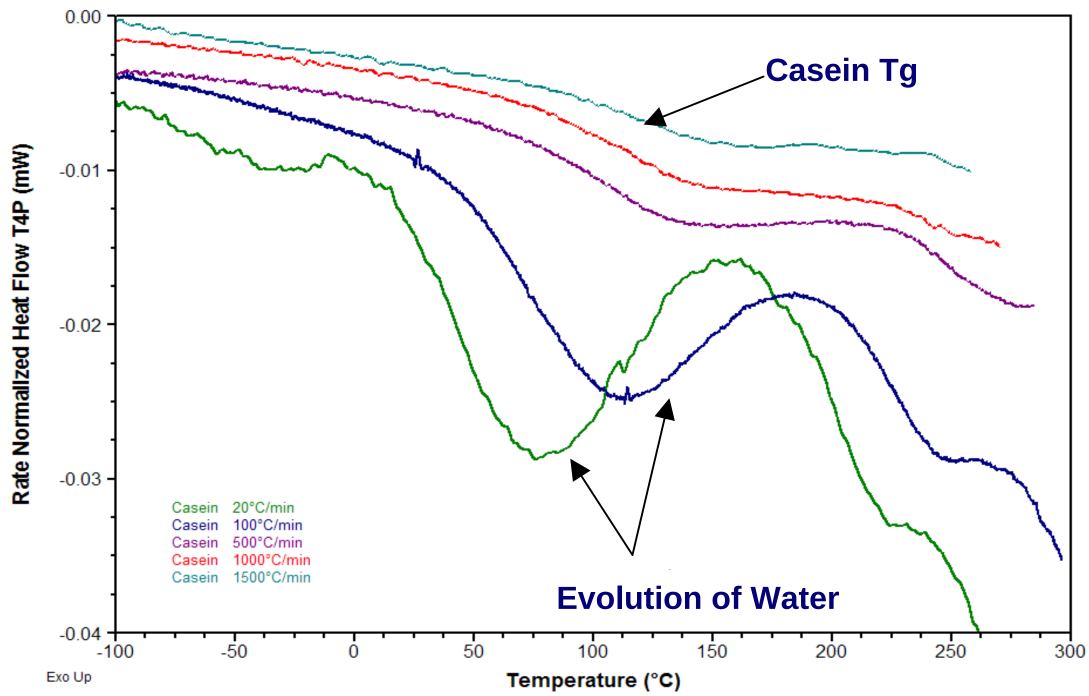

Figure 2 – DSC data of casein at various scan rates.

REFERENCES

1. Robert L. Danley, Peter A. Caulfield and Steven R. Aubuchon, *American Laboratory*, January 2008, pp. 9-11.

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