The Use of Tzero™ and Modulated DSC® for the Characterization of Milk Powders

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ABSTRACT

Milk powders are examples of biological samples that are often complex mixtures of sugars, fats and proteins. Thermal properties aid in product process control, the prediction of storage characteristics and in alimentation. Traditional limitations of DSC are overcome by the new Tzero™ technology along with its improvements in sensitivity, quality of baseline, resolution, and wider modulation conditions in MDSC experiments. Two examples from milk powders are studied here.

INTRODUCTION

Differential Scanning Calorimetry (DSC) is used widely to characterize biological materials. Typical observed transitions include the glass transition of the amorphous phase, melting and crystallization processes, denaturation, free and bound water, onset of oxidation, and heat capacity. Tzero™ DSC technology, with its attendant sensitivity and resolution, is ideal for examining these low energy processes. Modulated DSC® is another useful tool that aids in the determination and interpretation of the complex DSC profiles typical of biological samples.

EXPERIMENTAL

Two dry milk powders were characterized using Tzero™ DSC and Modulated DSC, to identify and quantify the components, and the type (amorphous or crystalline) of structure present. The first sample, a whey (lactoserum) powder, is a biological liquid derived from milk by ultra-filtration and extraction of lipids. It is then concentrated by vacuum evaporation, and crystallization of lactose (alpha monohydrate form, MW = 360 g/mol). The sample is then dried by atomization. The result is a lactose crystallized dry powder containing a minor amorphous phase (*2*). Fats from original milk are no longer present, and the resultant powder is a non-fat milk powder. The glass transition temperature of the amorphous phase depends on the drying conditions. To examine the material for the glass transition, heat-only modulated conditions (10 °C/min, amplitude ± 0.8 °C, period 30 s) are chosen, keeping the heating rate equivalent to standard DSC. Aluminum hermetic pans are used.

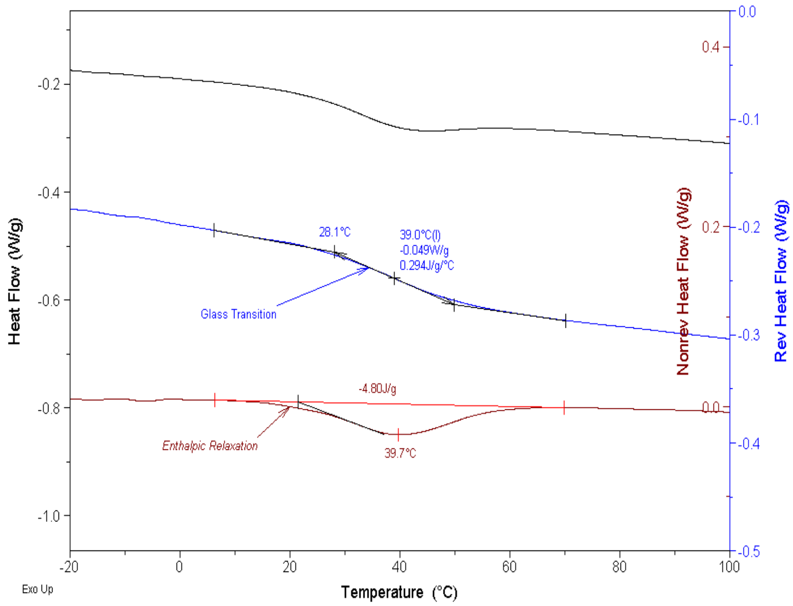

Figure 1 – Non-Fat Milk Powder

The second sample is a fat containing milk powder. The fabrication process is the same as that for the non-fat milk powder except that before drying, a determined quantity of fat (here coconut oil) is added. The major components of this powder are alpha lactose and lipids. Thermal characterization includes the determination of the glass transition of the amorphous phase of lactose, and the quantitation of the melting endotherm of the lipids. The sample is examined at 10 °C/min. The second sample is also run in Advanced Modulated DSC, at 10 °C/min, in heat-only conditions (amplitude ± 0.8 °C, period 30 s).

RESULTS AND DISCUSSION

Figure 1 shows the MDSC thermal curve for the non-fat milk powder. The upper curve represents the total heat flow, the middle curve the reversing heat flow and the lower curve, the nonreversing heat flow. The figure shows that the total heat flow curve is difficult to interpret because of the overlapping glass transition and enthalpic relaxation.

The measurement by Modulated DSC of the non-fat milk powder (first sample) separates the glass transition (resolved in the reversing heat flow) from the enthalpic relaxation (in the nonreversing heat flow). The use of high heating rate and short period produces several cycles in the glass transition, a necessary condition for deconvolution of MDSC signals. This set of conditions is only available with Advanced Tzero™ technology. The change in specific heat capacity (0.29 J g^{-1} $°C^{-1}$) at the glass transition is directly measured upon first heating by Modulated DSC. The amount of amorphous phase may then be quantified, becoming a quick control quality check of the milk powder.

The measurement by DSC of the fat containing milk powder (second sample) is shown in Figure 2. The figure shows only a two-step endotherm, which is the melt of the fats in the same temperature region. But the glass transition of the amorphous phase of lactose is hidden (figure 2). The fat level is quantified by integration of the melt endotherm from the DSC baseline (about 12.4 J g^{-1}) .

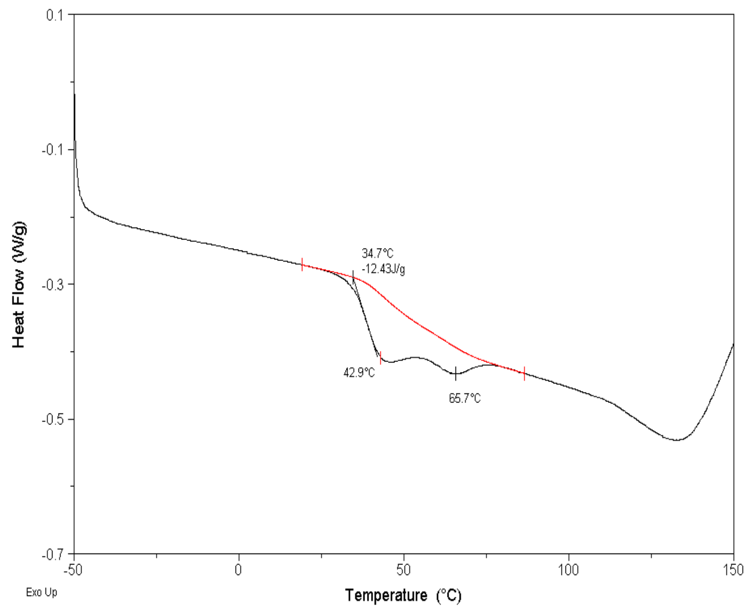

Figure 2 – Fat Containing Milk Powder

The measurement of the fat containing milk powder by modulated DSC gives a more complete understanding of the second sample, with the interpretation of reversing and nonreversing heat flows as shown in Figure 3. There is a clear separation of the glass transition of the amorphous phase of lactose (resolved in the reversing signal) from the melting of the fats (completely resolved in the nonreversing signal). The heat capacity change (0.17 J g^{-1} °C^{-1}) is then directly determined at first run, and also the correct quantification of the enthalpy of melting of fats in the nonreversing heat flow (10 J g^{-1}).

CONCLUSION

The glass transition of milk powders is used to quantify the lactose content even in the presence of overlapping melting of fat. These biological examples confirm the accuracy and usefulness of the Tzero™ DSC and Modulated Tzero™ DSC. Sensitivity, baseline quality, wide range of modulation conditions are determinant for accurate characterization of samples of biological materials.

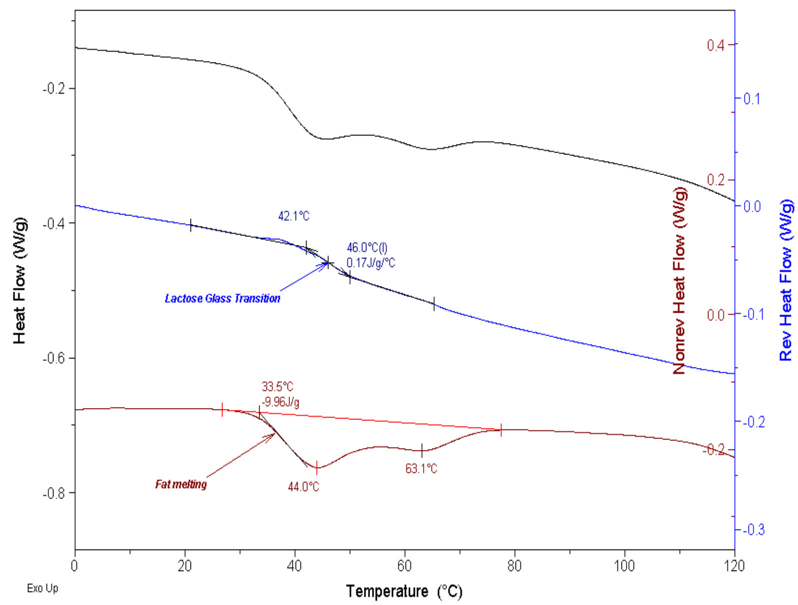

Figure 3 – MDSC of Fat Containing Milk Powder

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KEYWORDS

differential scanning calorimetry, biologicals, foods, glass transition, modulated differential scanning calorimetry

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