Using StepScan DSC to Measure the Amorphicity of Spray-dried Lactose Samples

Kalle Vähä-Heikkilä, Mikko Koivisto and Ensio Laine

Department of Physics, University of Turku, FIN-20014 TURKU

The effect of alternating the parameters of the heating program on the signal of StepScan DSC was studied. In this study samples were amorphous spray-dried lactose. The heating program includes two different states. In the first state, the heating state, sample is heated small temperature increments by some known heating rate. The second state, the isothermal state, includes a short isothermal time interval in which the sample reaches a thermal equilibrium. The equilibrium is determined with equilibrium criteria (mW). From the raw StepScan DSC data **Thermodynamic C_p** and **IsoK Baseline** curve can be calculated [Fig. 1]. Thermodynamic C_p curve shows the reversible or fast phenomena, such as glass transition. IsoK Baseline curve includes irreversible or slow phenomena. Glass transition and recrystallization of several spray-dried lactose samples having different amorphicity was studied after the determination the ideal measuring parameters. In this study the amorphicity of the samples was measured with isothermal microcalorimetry (IMC).

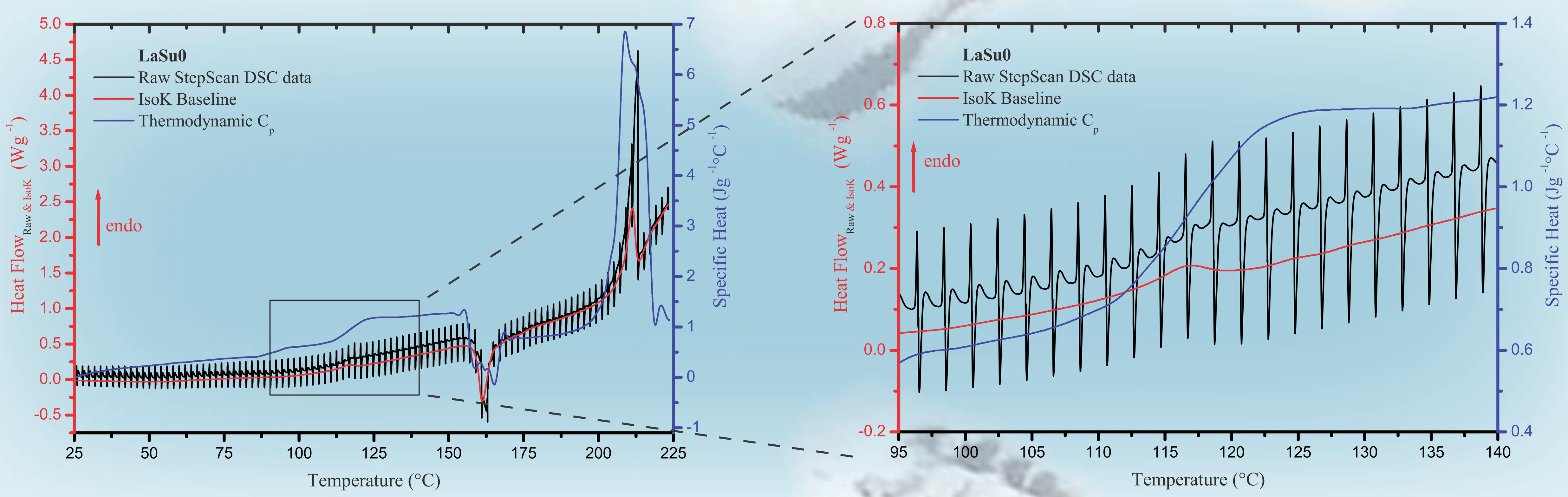

Fig. 1. From the raw StepScan DSC data can be calculated Thermodynamic C_p and IsoK Baseline curve. In this measurement the parameters of the heating program are: Step size (2 °C), heating rate (5 °C/min), isothermal time (1 min) and equilibrium criteria (±0.01 mW).

RESULTS

The different parameters of the heating program induce noise to the Thermodynamic C_p and IsoK Baseline curve in different ways. The effect of the alternation of parameters to the noise is presented in table 1. In addition, changing the parameters affects the sensitivity and the resolution of StepScan DSC [Table 2].

The change in specific heat C_p at glass transition was observed with lactose samples having different amorphicity. The values of ΔC_p at the glass transition with the different samples are represented in Fig. 2. The results show that the change of the specific heat is proportional to the amorphicity measured with IMC.

Table 1. The alternation of the parameters of heating program influences on the noise of StepScan DSC

Parameter	*Thermodynamic C_p curve* increasing *noise*	*IsoK Baseline curve* increasing *noise*
Step size	small	small
Heating rate	small	small
Equilibrium criteria	small	high
Size of the sample holder	increasing mass	increasing volume

Table 2. The alternation of the parameters of heating program affects the sensitivity and resolution of StepScan DSC

Parameter	Max. sensitivity		Max. resolution
Step size	1–4 °C		small
Heating rate	5 - 10 °C/min		small
Equilibrium criteria	*Thermodynamic C_p*	*IsoK Baseline*	high
	small	high	

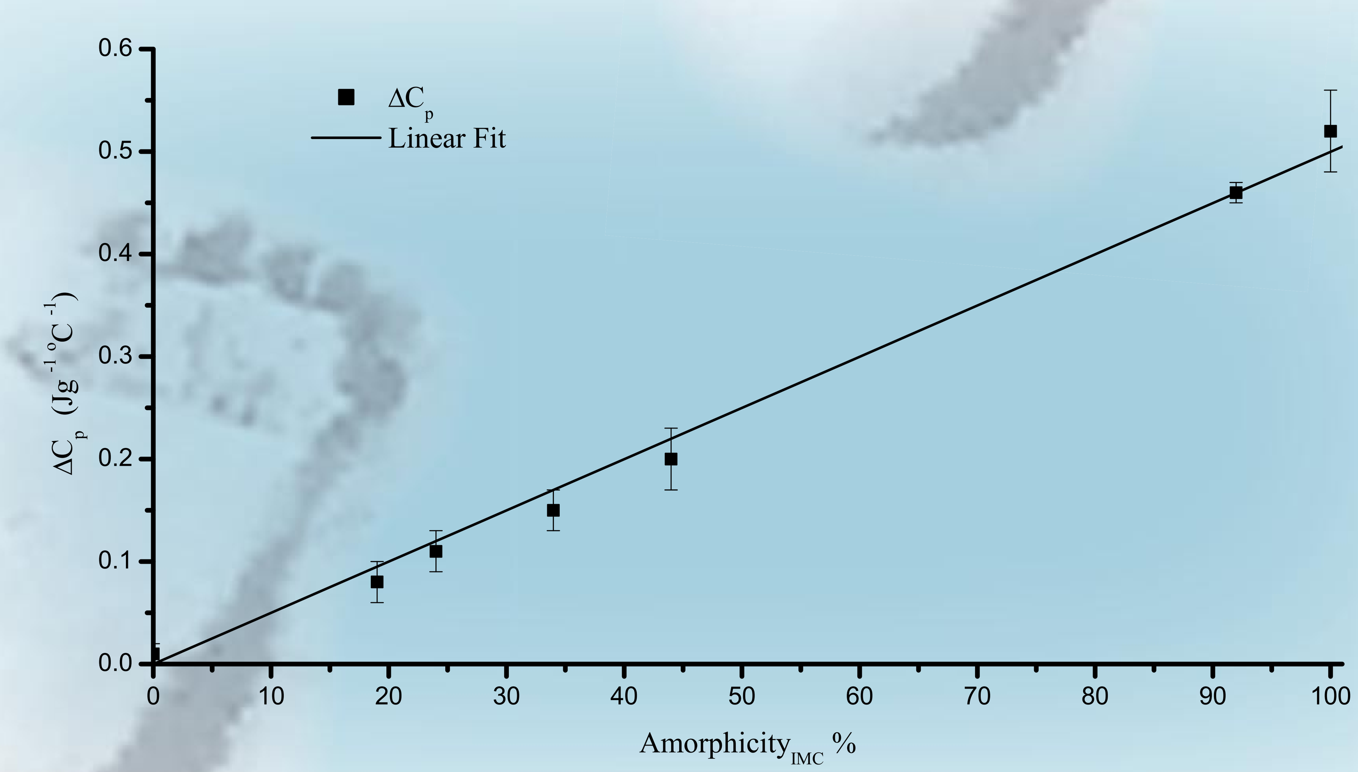

Fig. 2. StepScan DSC results for ΔC_p at the glass transition are proportional to the amorphicity of lactose samples measured with IMC. The parameters of heating program are: Step size (2 °C), heating rate (5 °C/min), isothermal time (1 min) and equilibrium criteria (±0.01 mW).

CONCLUSIONS

- One measurement separates irreversible and reversible phenomena in StepScan DSC
- By choosing the parameters of the heating program the sensitivity, the resolution and also the noise of StepScan DSC can be affected
- StepScan DSC is a potential method for determining the amorphicity