

Introduction

When measuring melting points of fats or oils, some anomalies can be detected due to supercooling, but mainly to the fact that solid triglycerides exist in different crystalline forms (polymorphism), each of them with a different melting point. The crystalline form depends on the rate of cooling or heating, on the storage conditions (time, temperature...) These crystalline forms have an influence on the functional properties of the fat : e.g. butter spreading, milky aspects of oils.

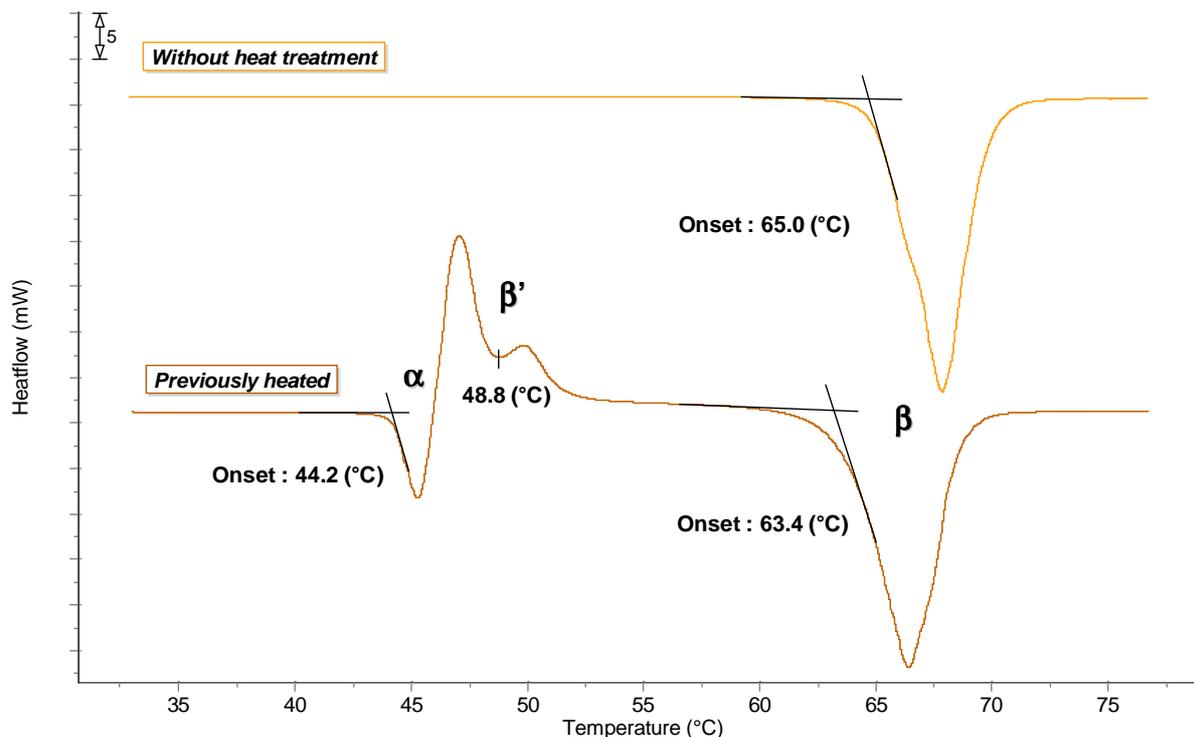


Figure 1 – Superimposed heating thermograms with and without thermal treatment

Experimental

Sample: Tripalmitin

DSC 131 Evo experimental conditions: Sample mass of about 10 mg in a 100 μ l sealed aluminum crucible

Profile: The temperature is programmed from RT up to 80°C at 5°C.min⁻¹

- Without heat treatment
- Previously heated at 80°C then cooled quickly to RT

Results

When the sample of tripalmitin is heated from 20°C up to 80°C, without any previous heat treatment, only one endothermic effect corresponding to the melting of the β -form is recorded at 65.0°C.

If the sample is previously heated at 80°C, then quickly cooled down to 20°C, the DSC curve shows three distinct melting peaks, corresponding to the α -form (44.2°C), the β' -form (48.8°C) and the β -form (63.4°C). The exothermic peaks indicate the transformation $\alpha \rightarrow \beta'$ and $\beta' \rightarrow \beta$.

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Tripalmitin melting by DSC : Influence of heat treatment

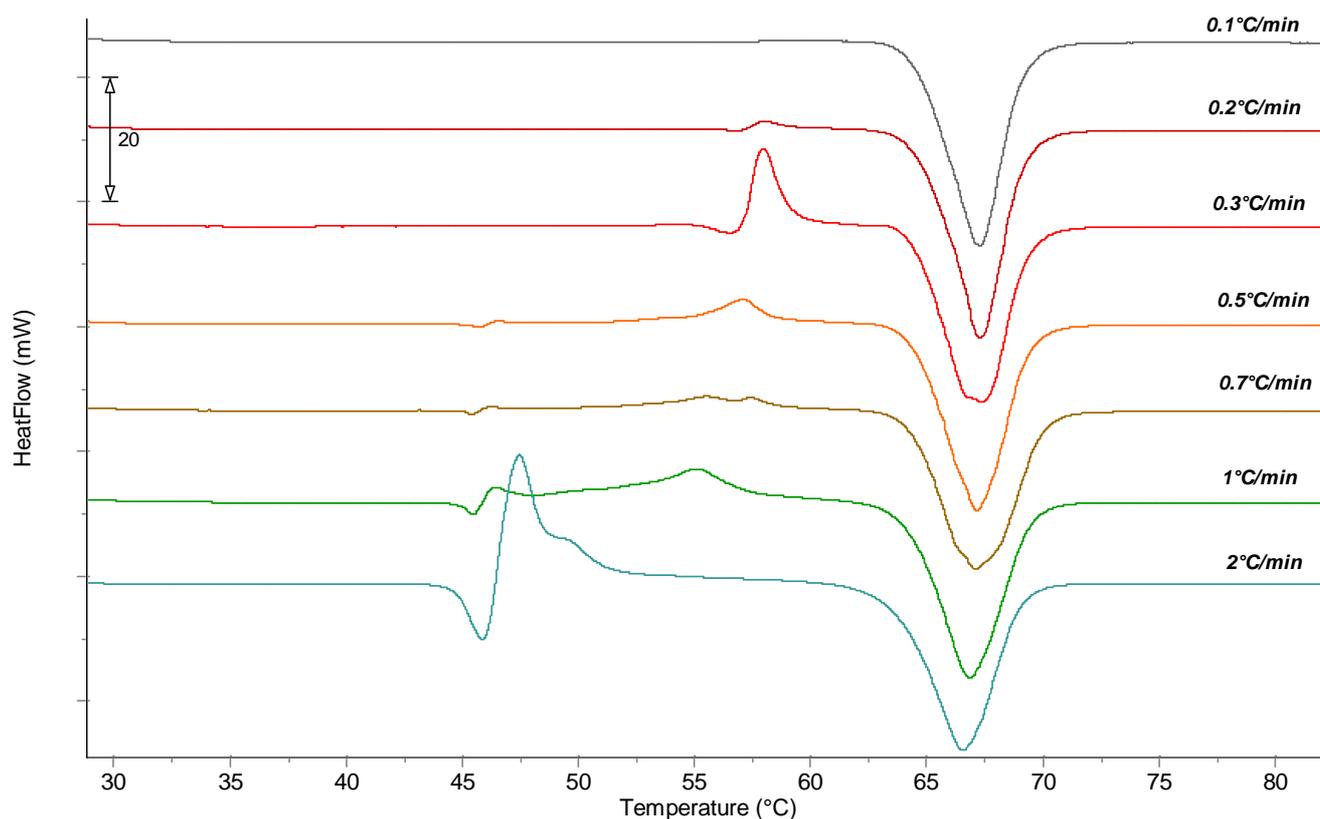


Figure 2 – Superimposed heating thermograms of the tripalmitin sample after different cooling rates (indicated on the right)

In this case, the sample was heated at 5°C/min then cooled at different temperature scanning rates. The here above figure presents the heating thermograms following those cooling phases.

A cooling rate of 0.1°C/min allows to reach the equilibrium since only the more stable polymorph is observed. While increasing the cooling rate, the crystallization of β' -form and then α -form (the less stable form) starts to appear.

At 0.2°C/min, the melting of β' -form is observed and with a cooling rate of 0.5°C/min, the melting of the α -form is measured.

The transition $\beta' \rightarrow \beta$ is shifted to lower temperatures when the cooling rate increases.

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