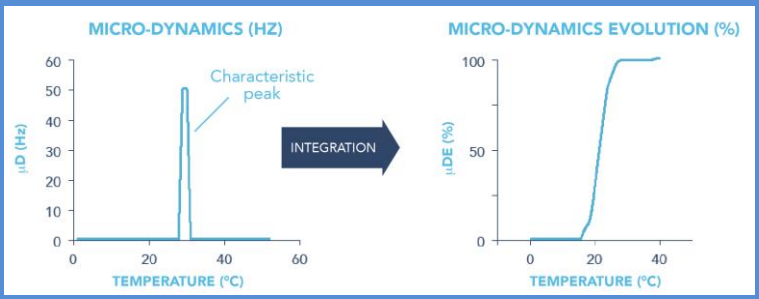
Quality control of raw materials thanks to thermal microrheology

Introduction

Crystallization and melting temperatures of natural products may vary in function of origin, climate or in function of the blending and process. It's then important to control easily these parameters before using the raw materials.
In this note, we will compare two palm oils with different origins, during crystallization and melting.

Reminder on the technique

RHEOLASER Crystal uses the DWS principle. Light is scattered by the particles, creating an interference pattern (Speckle Image). The variation of this image is related to the motion of the particles. By a mathematical analysis of this variation, decorrelation functions can be computed and then processed, to obtain a characteristic time τ as a function of time or temperature.



Values of 1/τ or Micro-Dynamics (Hz), are then plotted against time or temperature, resulting in characteristic peaks when the product shows a microstructural evolution, such as a phase transition or any other physical event. The signal can then be integrated for an easier visualisation, obtaining the so-called Micro-Dynamics Evolution (%).

Method

Two types of palm oils were analyzed, differing by their origin.
For crystallization, samples were cooled from 75°C to 4°C, with a ramp of 5°C/minute. For melting, samples were heated from 4°C to 75°C at 5°C/minute. Analyses were performed with a RHEOLASER Crystal.

Experimental results

1) Crystallization

Samples were cooled down from 75°C to 4°C with a ramp of 5°C/min.

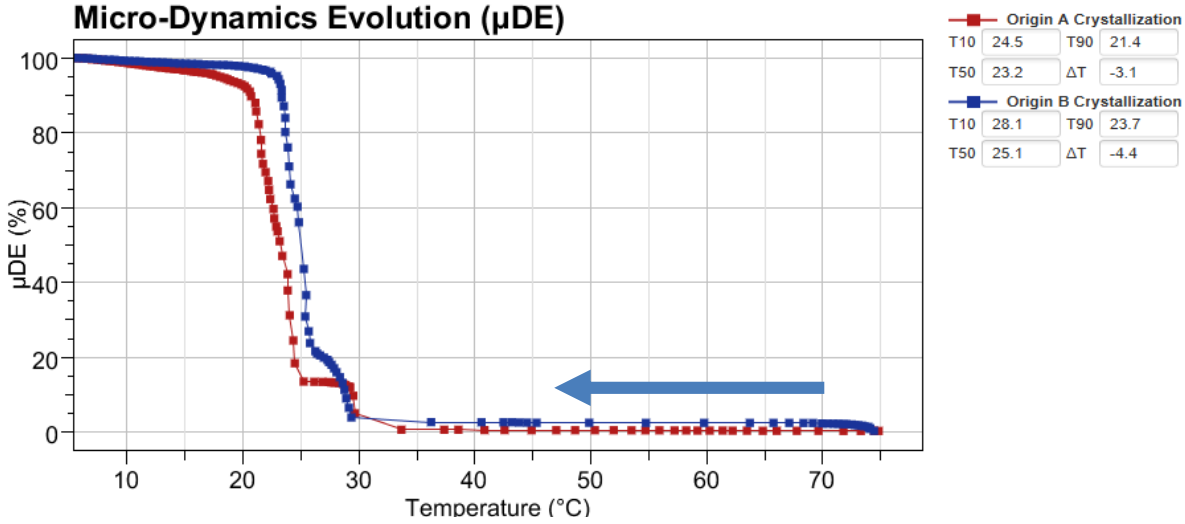


Figure 1 Micro-Dynamics Evolution in function of temperature, 75°C to 4°C, 5°C/min

Even if both palm oils began to crystallize at the same temperature (approximately 30°C), Origin A shows an average transition temperature (T_{50}) lower than Origin B (about 2°C lower) (figure 1).
At 30°C, samples were still completely liquid and at 10°C both of them were completely crystallized (Micro-Dynamics Evolution reached 100%).

2) Melting

For melting, the same parameters were applied for the ramp (4°C to 75°C, 5°C/min).

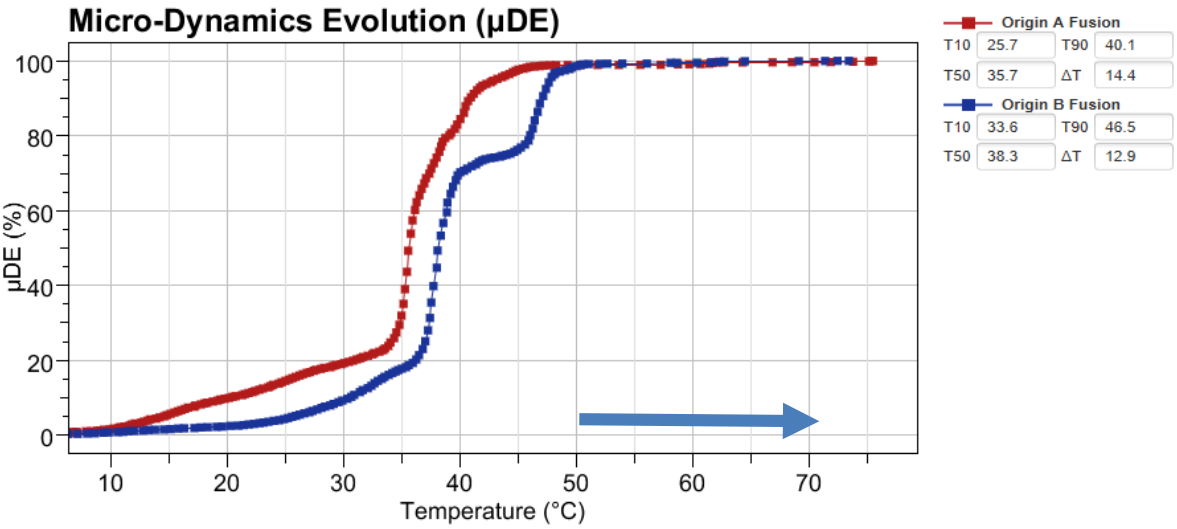


Figure 2 Micro-Dynamics Evolution in function of temperature, 4°C to 75°C, 5°C/min

Origin B have a higher average transition temperature than Origin A (figure 2).
At 50°C both samples were completely melted, but only sample A presents an increase of signal as early as 15°C (melting of some lipids), while sample B does not change until it reaches more than 20°C.

Variances between melting and crystallization temperatures of the two samples are due to disimilarity in lipid profile (more unsaturated triglycerides for Origin A than Origin B).

It is crucial for manufacturers to control polymorphic temperatures at reception of products, to validate the quality, or to adjust the process. Indeed, differences in origin of oils may have a huge impact on the transition temperatures.

Conclusions

The Rheolaser Crystal can differentiate melting and crystallization temperatures of raw products like palm oil.
The sampling system enables to work with "macroscopic" samples, preventing any risk of denaturation and allowing analysis of heterogeneous products, or with small volume for rare products.
As polymorphic transition temperatures vary in function of origin, climatic conditions, blending or process, it is very important for manufacturers to control easily raw materials, ensuring a great quality of the final products.

Key Benefits

VERSATILE – Monitor any transition phenomenon, any sample form (liquid, gel, paste…)
ACCURATE - nano-scale sensitivity, macroscopic sample, temperature range 4-90°C
EASY - 1-click set-up and data treatment, direct sampling without denaturation