Application Note
Melting of Chocolate

Melting different chocolate varieties in a variable sample temperature setup for T2 relaxometry

Summary

- Variable Temperature (VT) probe 30 °C - 100 °C
- T2 measurement during heating
- Observation of the melting and crystalization of cocoa butter

Abstract

In this study, we conducted measurements using the Pure Devices Time-Domain Nuclear Magnetic Resonance (TD-NMR) system on various types of chocolate. The TD-NMR system was equipped with a variable temperature setup to control the sample temperature. We observed changes in T2 relaxation times as a function of temperature. Notably, around approximately 36°C, a distinct inflection point was detected in the relaxation time curve. This phenomenon may be attributed to the melting of cocoa butter, a primary component in chocolate, which undergoes a phase transition around this temperature. Interestingly, upon cooling, this inflection point was no longer present, and the relaxation time curves exhibited a continuous trend. Additionally, different types of chocolate displayed varying relaxation time profiles across the temperature range, highlighting the influence of composition and processing on their thermal behavior.

Used hardware:

For the experiments the following hardware components were used (Figure 1):

- MRI control unit drive-L
- Magnet magspec 20 mm
- VT system 30 °C - 100 °C

Method

The study examined the following chocolate varieties:

- Cheap dark chocolate (no-name)
- Milk chocolate (Schogetten, Ludwig Schokolade)
- Aerated chocolate (Milka Luflee, Mondelez)

As reference sample, a vegetable oil was used.

The chocolate was cut to shavings and pressed into a 15 mm glass tube to ensure good thermal contact to the tube.

The sample was put into the variable temperature (VT) probe which was set to room temperature (25 °C) . For each measurement step, the temperature was increased by 2 °C to a maximum

Figure 1: Magspec 20 mm and VT Unit

of 100 °C. After passing the interesting point at 40 °C, where most of the changes in relaxation times were expected, the temperature increment was set to 10 °C . After reaching the maximum of 100 °C, the temperature was decreases in the same way. After 5 minutes tempering at the target temperature (waiting time), a T2 CMPG measurement was performed. The acquired data was fitted with a single exponential fitting routine.
For further evaluation, the resulting T2 values as well as the amplitudes of the exponential fit, extrapolated to the point at time t = 0 (axis section) were used.

Results T2 values

Figure 2 shows the T2 values for the increasing temperature profile. One can see the melting of the cocoa butter occurs between 28 °C and 36 °C, which agrees with literature values (*Ullmann's Food and Feed. Vol. 2, Wiley, 2017, ISBN 978-3-527-33990-7, S. 655 f, 706.)* The temperature dependency of the T2 values for the different varieties of chocolate is also clearly different.
Figure 3 shows an enlarged and selected illustration from Figure 2, additionally showing the descending temperature profile. It is clearly visible that the process that is leading to the steep transition to higher T2 values is irreversible. Further experiments could investigate whether this irreversibility occurs only above a critical temperature or already after passing 36 °C.

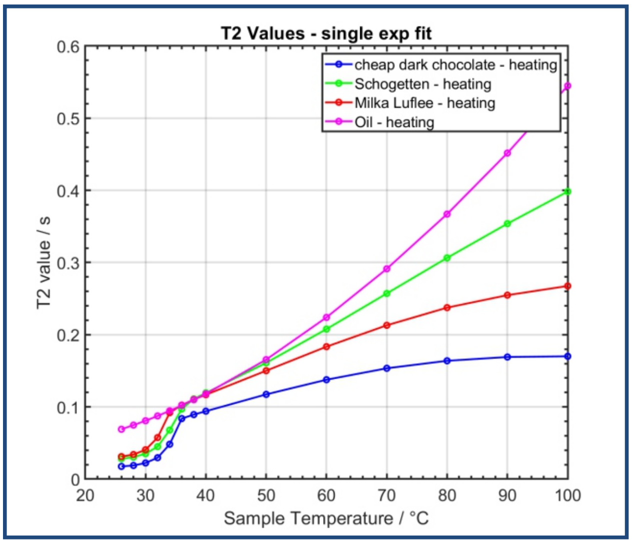

Figure 2: T2 values of different chocolate variations.

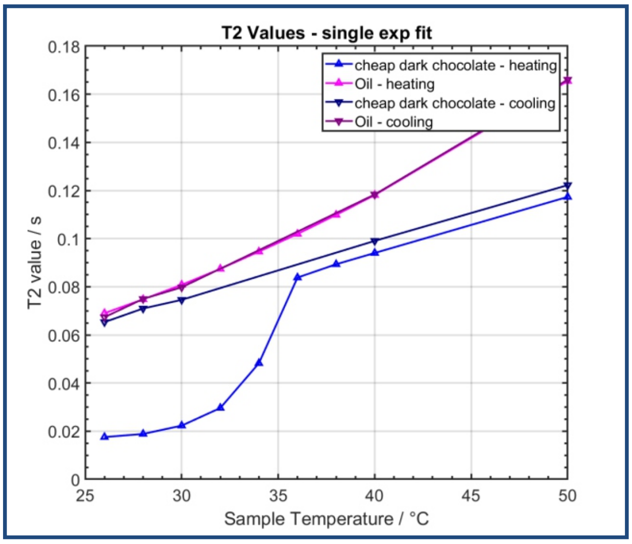

Figure 3: Enlarged and selected illustration from Figure 1. Increasing and descending temperature profile showing hysteresis for chocolate.

Temperature offset

The VT system controls the temperature of the sample by a hot air jet. To determine the offset between the target temperature (air jet) and the sample temperature, the sample temperature was measured directly by an optical fiber thermometer after the waiting time. The results can be found in Figure 4. One can see that the differences between the target temperature and the actual sample temperature are negligible for the used temperature profile. However, if a precise absolute temperature in a small range is required, one should use a optical fiber to control the temperature. This is natively supported in the control software of the VT system.

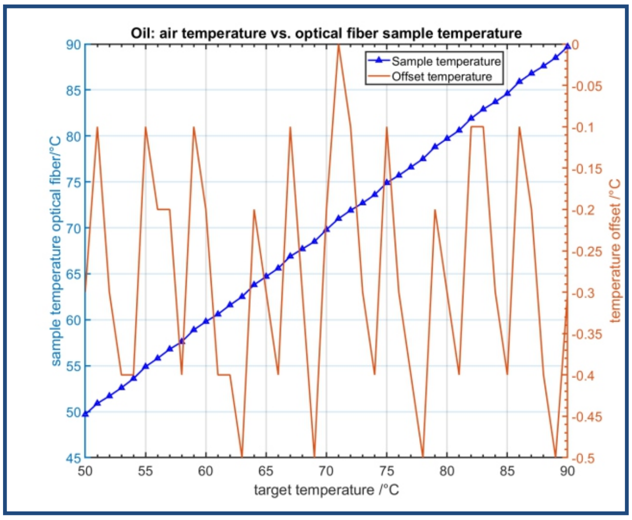

Figure 4: Temperature offset target to sample temperature measured using a optical fiber thermometer with the reference oil sample.

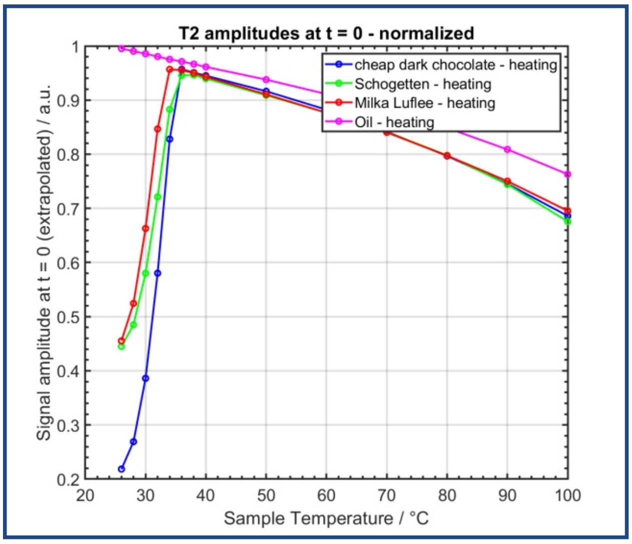

Figure 5: Measured T2 amplitudes of different chocolates extrapolated from the T2 fit to t = 0; normalized.

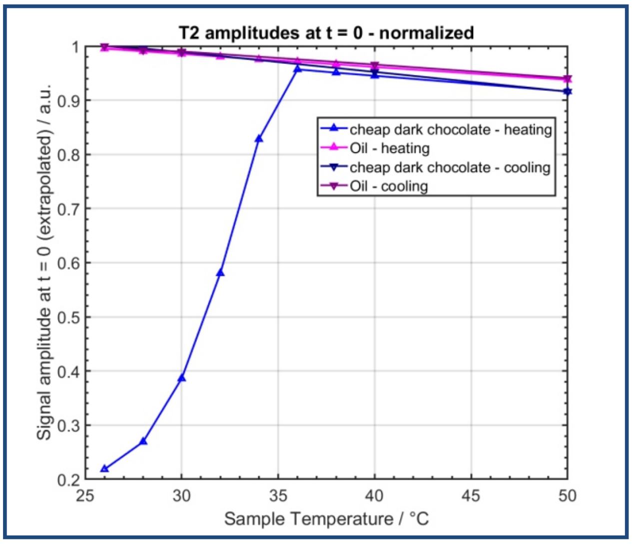

Figure 6: Enlarged and selected illustration from Figure 4. Increasing and descending temperature profile showing hysteresis for chocolate.

Results T2 amplitudes

Figure 5 shows the T2 amplitudes at time t = 0 (axis section) which were normalized to the last point of the decending profile. One again can see the melting of the cacao butter between 28 °C and 36 °C. Different behaviours of different kinds of chocolates are also clearly visible at the lower temperatures before melting. At higher temperatures, the curves converge to each other.

Figure 6 shows the enlarged illustration showing also the descending temperature profile. A irreversibility for chocolate is clearly visible as previously seen for the T2 values.

Boltzmann calibration

Due to the Boltzmann distribution, which dictates the population difference between the lower and higher energy states, signal decreases as temperature increases. At higher temperatures, more nuclei occupy the higher energy state, reducing the net magnetization. This results in less signal at higher sample temperatures. This effect can be compensated by measuring a sample which shows no phase transitions or melting effects over temperature like vegetable oil. The Boltzmann-compensated T2 amplitudes are shown in Figure 7. These kinds of measurements can for example be used for the determination of the solid fat content (SFC) of the chocolate sample.

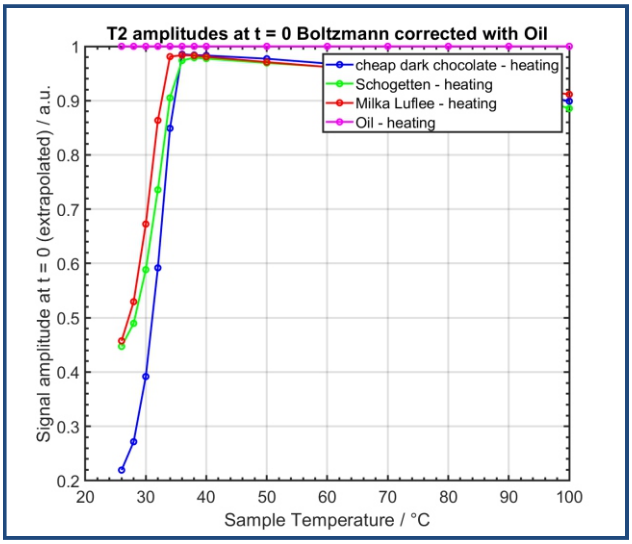

Figure 7: Measured T2 amplitudes at t = 0 after Boltzmann calibration

Conclusion

Both the relaxation times itself as well as the extrapolated amplitudes at t = 0 show interesting effects at different temperatures. The effects can be correlated to the phase transition of the cocoa butter. Boltzmann calibrated measurements can for example be used for SFC determination of chocolates.

Advantages

The Pure Devices Variable Temperature setup is a flexible tool to investigate the temperature dependency of materials during heating as well as cooling. The possibility of running arbitrary temperature profiles enables the researcher to study a wide variety of materials and effects.

The measurement is not limited to relaxation time measurements and can be combined with any MR sequence resulting in endless possibilities.

pure devices
Pure Devices GmbH
Kettelerstr. 5-11
97222 Rimpar
GERMANY
www.pure-devices.com
info@pure-devices.com
Tel.: +49 (0) 9365 2069490
Fax: +49 (0) 9365 2069486