Rheometers

Flow behavior of chocolate melts

Working according to ICA standard 46

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Figure 1: HAAKE Viscotester iQ Rheometer with Peltier temperature module and coaxial cylinder measuring geometry.

Introduction

The flow behavior of molten chocolate is a crucial property for many reasons. During production, the various steps of transportation, filling, dipping, coating, or dosing depend on a precisely defined viscosity and yield stress. The properties of the finished chocolate, such as the appearance of its surface or its mouthfeel, are also directly related to the viscosity behavior of the chocolate. Viscosity testing is therefore a standard quality control (QC) procedure for companies that manufacture chocolate or use chocolate for its own production of, for example, chocolate-coated cookies.

The International Confectionery Association (ICA) introduced a standard method (method 46) for the proper determination of the viscosity of molten chocolate at a temperature of 40 °C using a rotational rheometer with a coaxial cylinder measuring geometry.[1]

In this study, the rheological properties of two chocolates were determined using the ICA method 46 and the results are compared.

Materials and methods

Rheometry

The Thermo Scientific™ HAAKE™ Viscotester™ iQ Rheometer equipped with Peltier temperature control module for coaxial cylinders geometries (Figure 1) was used to measure the rheological behavior of chocolate samples at a temperature of 40 °C. All measurements were conducted using a CC25 DIN Ti coaxial cylinders geometry with a sample volume of 16.1 ml. A sample cover was used to further improve temperature homogeneity and minimize gradients.

Materials

Two commercially available chocolate samples, a milk chocolate, and a dark chocolate, were prepared according to ICA method 46 by placing chocolate pieces into glass containers, sealing the containers, and leaving them in a temperature chamber at 52 °C for between 45 and 60 minutes. Meanwhile the cup and rotor of the measuring geometry were preheated to 40 °C in the Peltier temperature module of the HAAKE Viscotester iQ Rheometer.

Test protocol

The test protocol according to the ICA method 46 was converted into a HAAKE RheoWin™ Software Job (Figure 2) that consists of three parts: sample conditioning, testing and data evaluation. Sample conditioning should always be part of the test method, to ensure that it is not skipped and always performed in the same way. This improves the reproducibility of the measurement results and the evaluation results calculated from them. During the conditioning step (Figure 2, Job element 3), the sample is kept at rest with the upper part of the measuring geometry already in measuring position, while the temperature is adjusted to 40 °C. During this time any mechanical stresses caused by sample loading and closing the geometry should completely dissipate, while at the same time, the whole sample should reach the temperature at which the test is to be performed.

The middle section contains the actual viscosity measurement (Job elements 6–10). After a 10 min pre-shear period at 5 s^{-1}, the shear rate is first increased from 2 to 50 s^{-1} within three minutes in a ramp that increases either continuously or in steps.

The shear rate is then maintained at a constant 50 s^{-1} for one minute, after which it is lowered again to 2 s^{-1} within three minutes by means of a downward ramp (continuous or stepwise). During the three stages, viscosity and shear stress data are recorded continuously.

The complete shear rate profile of this methods is shown in Figure 3.

In the final steps of the procedure (Figure 2, Job elements 11–12), the data evaluation is performed automatically by the HAAKE RheoWin Software. This part includes the determination of viscosity values at specific shear rates highlighted in the ICA method 46 via interpolation. For the additional calculation of a yield stress or other rheological parameters, a Windhab[2] or Casson model fit can be added to the data evaluation. In a simpler approach, Servais suggested using the shear stress value at 5 s^{-1} (taken from the upward curve) divided by 10 as a measure for the yield stress.[3] If this method is preferred, the corresponding viscosity value is obtained via the interpolation step in the HAAKE RheoWin Software Job.

For comparison, a steady-state shear test at 40 °C was performed with both samples in addition to the determination of the rheological properties according to ICA method 46. Compared to transient viscosity data from shear rate ramp tests, the steady-state viscosity is independent of any time-dependent rheological effects due to viscoelastic properties or the speed of the applied shear rate ramp. For the comparison of absolute viscosity data, the steady-state shear test is the preferred choice, because it is independent of any instrument or measurement artefacts.

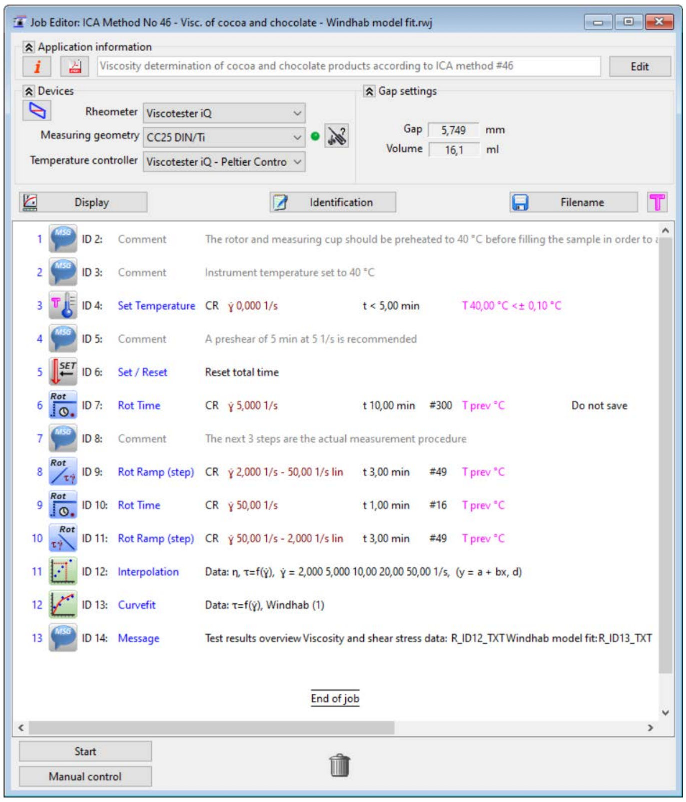

Figure 2: The HAAKE RheoWin Software job composed to run the test according to ICA method 46. The test consists of sample conditioning (elements 1–5), the rheological test (elements 6–10), and data evaluation (elements 11–12).

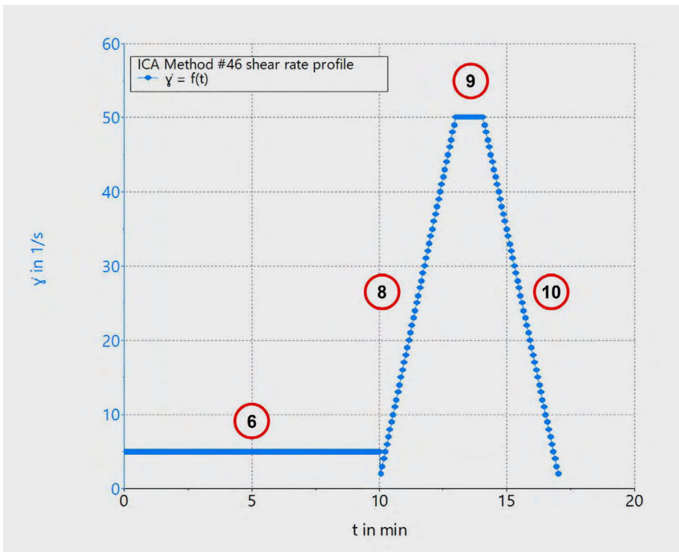

Figure 3: Shear rate profile applied according to ICA method 46. The numbers 6 to 10 represent the Job element number of the HAAKE RheoWin Software Job shown in Figure 3.

Results

A typical representation of the results from a test according to ICA method 46 is shown in Figure 4. The red curves depict the shear viscosity and the blue curves the shear stress. It clearly shows that the milk chocolate has the higher viscosity by a factor of two or more. This is also evident from the results of the steady-state shear test, as shown in Figure 5.

Furthermore, the viscosity curves from the increasing and the decreasing shear rate ramps are almost identical for the dark chocolate. In contrast to that, the milk chocolate shows a pronounced thixotropic behavior with a significant difference between the two viscosity curves.

The green parabolic lines extrapolating the flow curves down to a shear rate of 0 s^{-1} represent the results of curve fittings according to the Windhab and Casson models. The vertical green lines indicate the interpolation according to Servais at 5 s^{-1} and 40 s^{-1}. The yield stress according to Servais is calculated from the Stress value at 5 s^{-1} divided by 10. The results of the different methods to determine the yield stress of the two chocolate melts have been summarized in Table 1.

The first and probably most important result from Table 1 is the insight that even based on the same data, different mathematical models lead to different results. Therefore, only yield stress values calculated with the same mathematical model can be compared.

Independent of the model chosen, the milk chocolate in this example shows the higher yield stress, the higher viscosity and the stronger thixotropy.

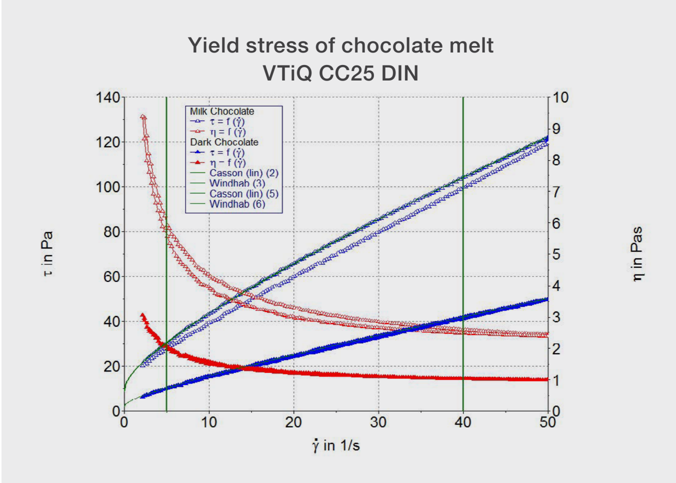

Figure 4: Test results for a milk chocolate (open symbols) and a dark chocolate (filled symbols). The milk chocolate shows the higher viscosity values (red curves), stronger thixotropy and a higher yield stress. The extrapolation of the flow curves (green curves) to 0 s^{-1} were calculated according to the Windhab and Casson model. The green vertical line at 5 s^{-1} represents the stress value needed for the determination of the yield stress according to Servais.

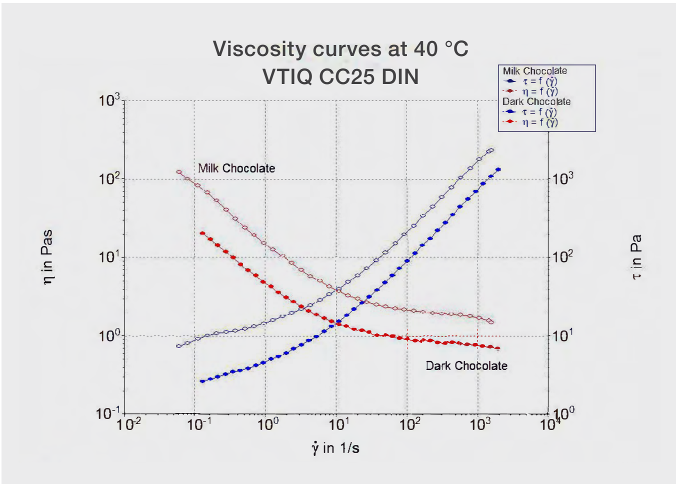

Figure 5: Viscosity curves of milk chocolate and dark chocolate at 40 °C. The milk chocolate shows a significantly higher viscosity.

	Milk chocolate	Dark chocolate
τ_0 Casson / Pa	8.9	2.1
τ_0 Windhab / Pa	14.7	4.0
τ_0 Servais et al. / PA	3.0	1.0

Table 1: Determination of yield stress based on the data from Figure 4 using different models.

Conclusions

ICA method 46 is a frequently used test procedure for determining the rheological behavior of chocolate melts with rotational rheometers. The two chocolate samples tested for this report showed significantly different behavior in terms of their shear viscosity, thixotropy and yield stress. With the HAAKE Viscotester iQ Rheometer, measurements according to the standard can be carried out easily, with high precision and reproducibility and with various evaluation methods. Further measurements, e.g., over a larger shear rate range, are also easy to realize with this device.

References

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3. C. Servais, H. Ranc, I. D. Roberts: "Determination of Chocolate Viscosity", Journal of Texture Studies 34 (5-6): 467-497 (2003)