Flow Behaviour of Chocolate Melts-Working according to ICA Standards

Klaus Oldörp, Thermo Fisher Scientific, Material Characterization, Karlsruhe, Germany

Introduction

The flow behaviour of molten chocolate is a crucial parameter for many reasons. During production the transport, filling, dipping, coating or dosing steps depend on a well defined viscosity and yield stress. Likewise, the properties of the final chocolate like the look of it surface or its mouth feeling are directly related to the chocolate's viscous behaviour.

Testing the viscosity is therefore one of the standard quality control (QC) test methods for any company producing chocolate or using chocolate for their own production of e.g. chocolate-coated cookies.

To make viscosity testing in QC easier and more reliable, the Thermo Scientific™ HAAKE™ Viscotester™ iQ (Figure 1) has been developed. This unique viscometer has some new features especially designed for QC applications. For example, due to its improved sensitivity it is possible to use smaller measuring geometries, which reduces sample volume, time for temperature equilibration and cleaning effort. Also, smaller shear rates are accessible due to the improved sensitivity, which improves the reliability of yield stress calculations [1] with extrapolation methods like the Casson model.

Preparations

Two chocolate samples, a milk chocolate and a dark chocolate, have been prepared according to ICA method 46 [2] by putting chocolate pieces into glass containers, sealing the containers and leaving them in an oven at 52 °C for between 45 and 60 minutes. Meanwhile the cup and bob of the measuring geometry are preheated to 40 °C in the Peltier temperature control unit of the Viscotester iQ.

The HAAKE RheoWin Job

For the tests done for this report, the CC25 DIN Ti measuring geometry has been selected. This small cylindrical system with only 16.1 ml sample volume fits into the Peltier cylinder temperature control and is easy to disassemble and clean.

The test method itself has been taken from ICA method 46 and has been translated into a Thermo Scientific™ HAAKE™ RheoWin™ job. The shear rate profile is shown in Figure 2.

The HAAKE RheoWin Job (Figure 3) consists of three parts: Sample conditioning, testing and evaluation. The sample conditioning should always be part of the test method itself to ensure that it is not forgotten and always performed in the same way, which improves the reproducibility of the results. During the conditioning part (job element 1-4) the sample is kept at rest with the cylindrical upper part of the measuring geometry already in measuring position. During this time any mechanical stress caused by sample loading and closing the geometry should relax completely while at the same time, the whole sample should reach the temperature, the test is going to be performed at.

In the final part (job element 11-13), the data evaluation is performed automatically by HAAKE RheoWin. To calculate the yield stress of a chocolate melt, the traditional Casson model and the modern Windhab model [3] can be selected from a long list of fit models. In a more simple approach Servais [4] suggested to use the shear stress value at 5 s^{-1} as the yield stress. If this method is preferred, a simple interpolation calculation in HAAKE RheoWin will do the job.

Fig. 1: The Thermo Scientific HAAKE Viscotester iQ

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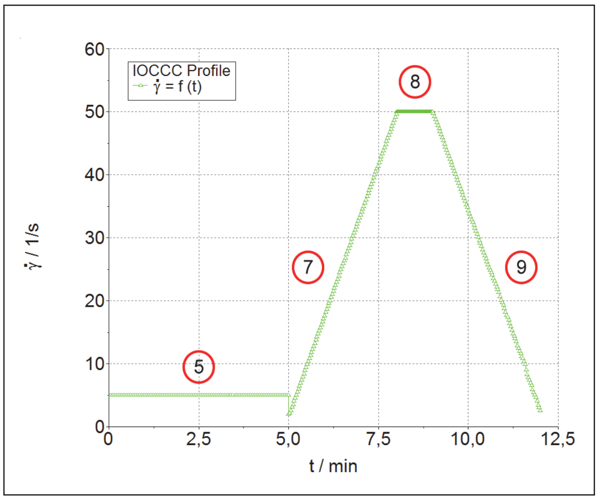

Fig. 2: Shear rate profile applied according to ICA method 46. The numbers 5 to 9 represent the job element number of the HAAKE RheoWin job shown in Figure 3.

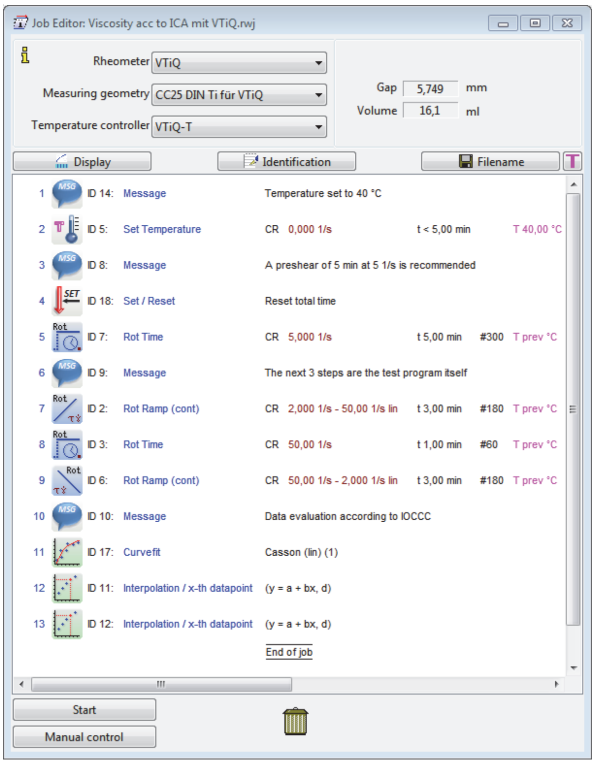

Fig. 3: The HAAKE RheoWin Job composed to run the test according to ICA method 46. The test consists of sample conditioning (elements 1-4), the rheological test (elements 5-9) and data evaluation (elements 10-13).

In addition a steady-state viscosity curve at 40 °C has been recorded for both samples. Compared to transient viscosity data from shear rate ramps, the steady-state viscosity is independent from time-dependent effect and the slope of the shear rate ramp. For comparison of viscosity data the steady-state viscosity is the best choice, because it is independent of the instrument used and can be directly correlated with the shear rate applied.

The 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 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.

The viscosity curves for the increasing shear rate ramp and the decreasing shear rate ramp are almost identical for the dark chocolate. In contrast, the milk chocolate shows a pronounced thixotropic behaviour with significant differences between the two viscosity curves.

The green parabolic curves extrapolating the flow curves to a shear rate of 0 s^{-1} represent the Casson fit. The vertical green lines indicate where the interpolation according to Servais has been calculated. The results of the different methods to determine the yield stress of the two chocolate melts have been summarized in Table 1.

	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	30.0	10.4

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

The first and probably most important result from Table 1 is the insight that even from the same data, different models give different results. Therefore, only yield stress values calculate 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.

Summary

In QC the rheological characterization of chocolate mainly focuses on its viscosity and yield stress. The HAAKE Viscotester iQ is a compact instrument with the right combination of sensitivity and strength to successfully test chocolate melts over a wide range of shear rates.

The commonly accepted test method according to ICA method 46 can easily be performed using only a small sample. The same is true for steady-state viscosity curves. The very good quality of the results shown in this report is an excellent base for a reliable data analysis with a variety of available methods and models.

Literature

[1] J. F. Steffe, "Rheological Methods in Food Process Engineering", Freeman Press 23 (1996)

[2] "Analytical Method 46 - Viscosity of Cocoa and Chocolate Products", ICA (2000)

[3] E. Windhab, Bericht: IV. Tagung Lebensmittelrheologie Detmold (1993)

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

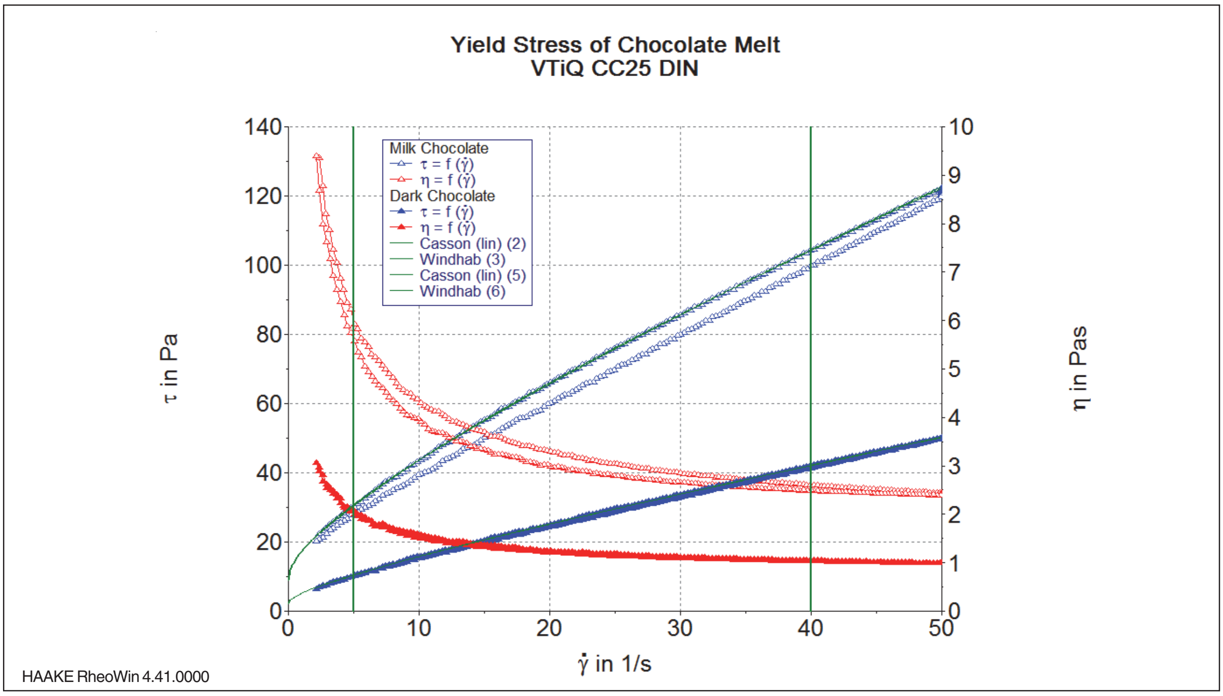

Fig. 4: Test results for a milk chocolate (open symbols) and a dark chocolate (filled symbols). The milk chocolate shows the higher viscosity values (red curves), stronger thixotropy and a higher yield stress. The extrapolation of the flow curves (blue curves) to 0 s^{-1} has been calculated according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

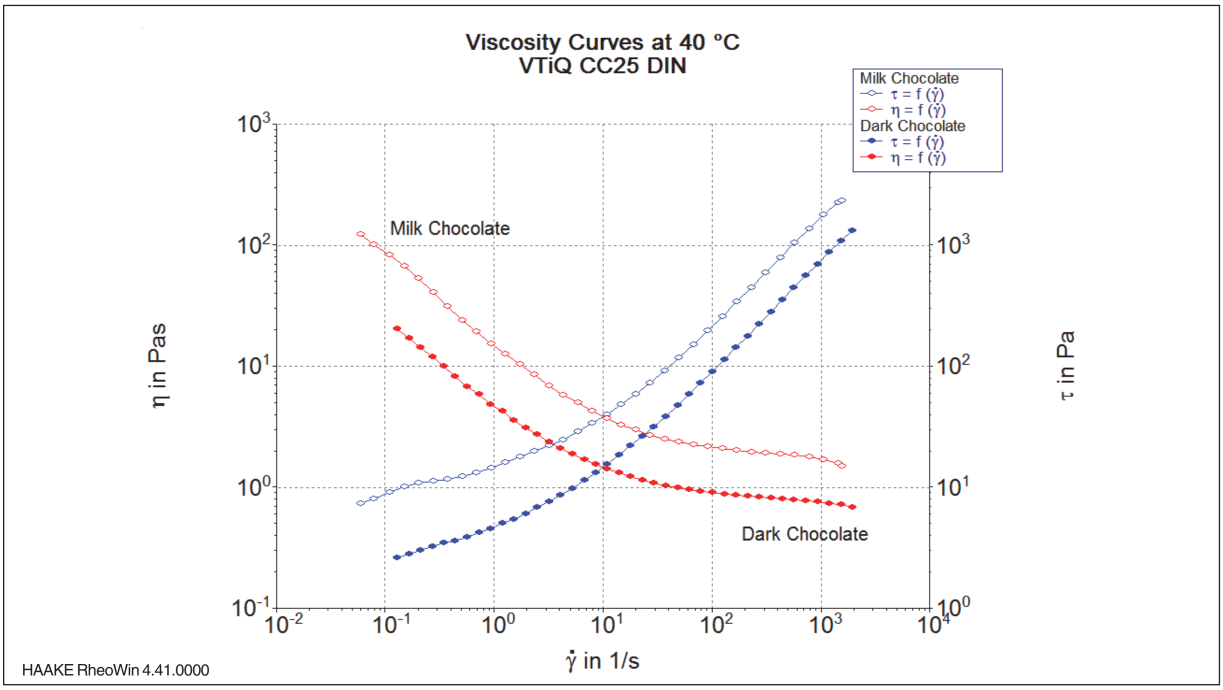

Fig. 5: Viscosity curves of milk chocolate and dark chocolate at 40 °C. The milk chocolate shows a significantly higher viscosity.

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Benelux
Tel. +31 (0) 76 579 55 55
info.mc.nl@thermofisher.com

China
Tel. +86 (221) 68 65 45 88
info.mc.china@thermofisher.com

France
Tel. +33 (0) 1 60 92 48 00
info.mc.fr@thermofisher.com

India
Tel. +91 (20) 6626 7000
info.mc.in@thermofisher.com

Japan
Tel. +81 (45) 453-9167
info.mc.jp@thermofisher.com

United Kingdom
Tel. +44 (0) 1606 548 100
info.mc.uk@thermofisher.com

USA
Tel. +1 603 436 9444
info.mc.us@thermofisher.com

International/Germany
Dieselstr. 4
76227 Karlsruhe
Tel. +49 (0) 721 4 09 44 44
info.mc.de@thermofisher.com

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