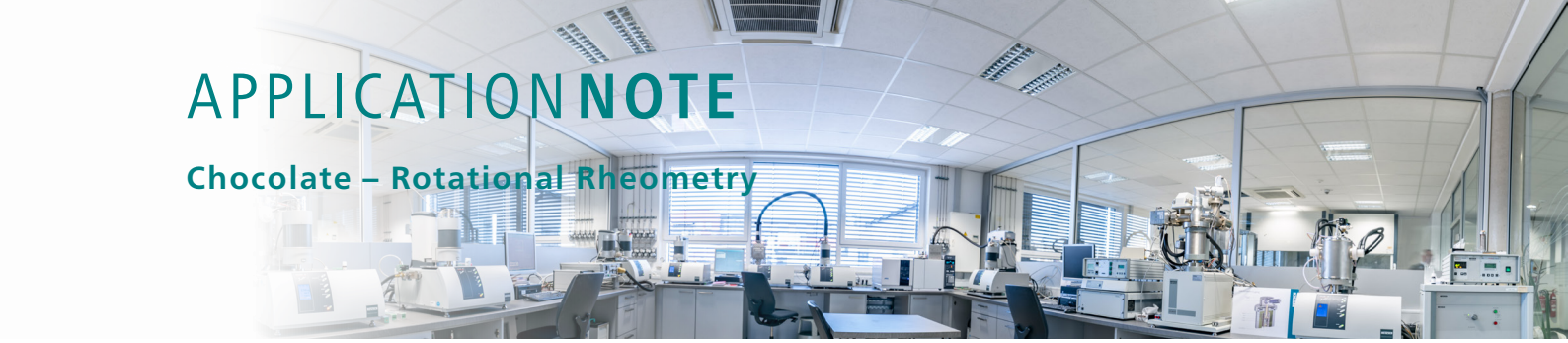

APPLICATION**NOTE**

Chocolate – Rotational Rheometry

Quality Control of Chocolate in No Time by Means of the Kinexus

Claire Strasser, Applications Laboratory Selb

Introduction

The shear viscosity of chocolate is a key feature for ensuring consistent quality and optimizing industrial processes. To standardize the measurement of this critical property, the International Office of Cocoa, Chocolate, and Sugar Confectionery (IOCCC) updated Analytical Method 46 in 2000. This method outlines a protocol for determining the viscosity of chocolate and cocoa-based products. It describes meticulously the sample preparation and measurement procedure, tailored to different types of cocoa products.

In the following, two chocolates (milk and dark chocolate) are measured with the Kinexus rotational rheometer following this SOP (Standard Operating Procedure). Then, the Casson analysis is applied to the curves. It is a mathematical model describing the flow behavior of chocolate, particularly its yield stress. This value is relevant in the chocolate industry, for example, for pumping processes, as it represents the minimum force required to induce flowing.

The **Casson model** is used to characterize the flow behavior of fluid that exhibits yield stress behavior such as chocolate, blood, or printing inks. It is expressed as:

$$\sqrt{\sigma} = \sqrt{\sigma_0} + \sqrt{k\dot{\gamma}}$$

σ: Shear stress [Pa]
σ_0: Casson yield stress (stress required to initiate the flow) [Pa]
k: Terminal shear viscosity (viscosity at high shear rate) [Pa·s]
$\dot{\gamma}$: Shear rate [s^{-1}]

Measurement Parameters

Each chocolate was stored at 60°C and then manually homogenized with a spatula before it was introduced into the geometry, which was maintained at 40°C. Table 1 summarizes The measurement conditions.

Table 1 Measurement conditions

Instrument	Kinexus Prime ultra+
Cartridge	Cylinder
Test	Rotation
Geometry	Cup and bob: 25 mm
Gap	9.15 mm
Temperature	40°C
Share Rate	• Pre-shear: 5 s^{-1} (15 min) • 2 to 50 s^{-1}, 10 samples per decade • 50 to 2 s^{-1}, 10 samples per decade

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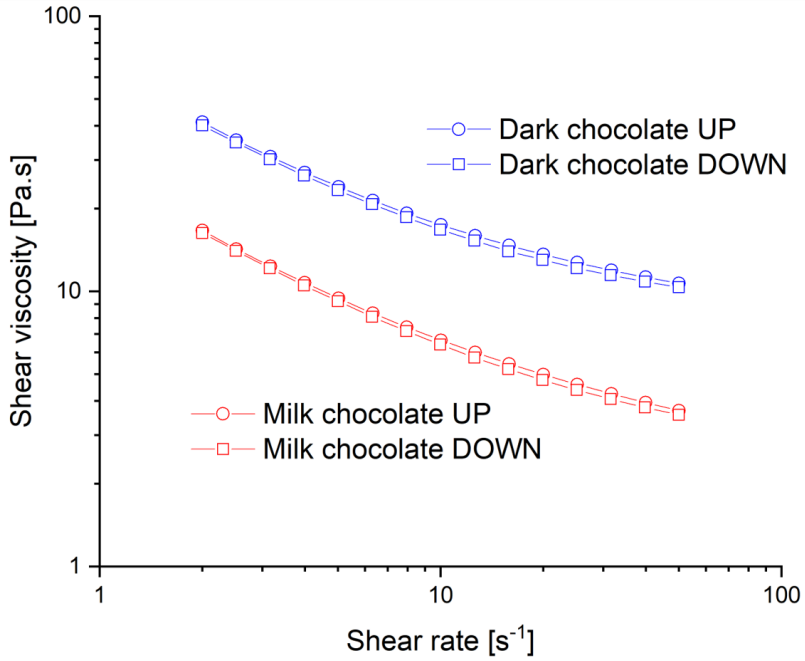
Resuts and Discussion

Figure 1 depicts the graph of the shear viscosity curves for the milk and dark chocolates over the course of the shear rate progression between 2 and 50 s^{-1} (UP, circles) and between 50 and 2 s^{-1} (DOWN, squares). The two samples show shear-thinning behavior with good accordance between the up and down ramps: The shear viscosity decreases with increasing shear rates. This is advantageous for processing because reducing the shear viscosity at the shear rates needed for operations like pumping decreases the resistance to flow. This way, the transfer of chocolate through pipes during manufacturing is easier and requires less energy.

The dark chocolate has a higher shear viscosity than the milk chocolate across the entire measured shear rate range. This makes it more suitable for coating applications, in which the shear viscosity is a key feature for achieving a uniform layer on candies without dripping and spreading. Furthermore, this property is also related to the mouth-feel of the consumer: The higher the shear viscosity, the creamier the chocolate is perceived to be.



1 Shear viscosity curves of the milk and dark chocolates

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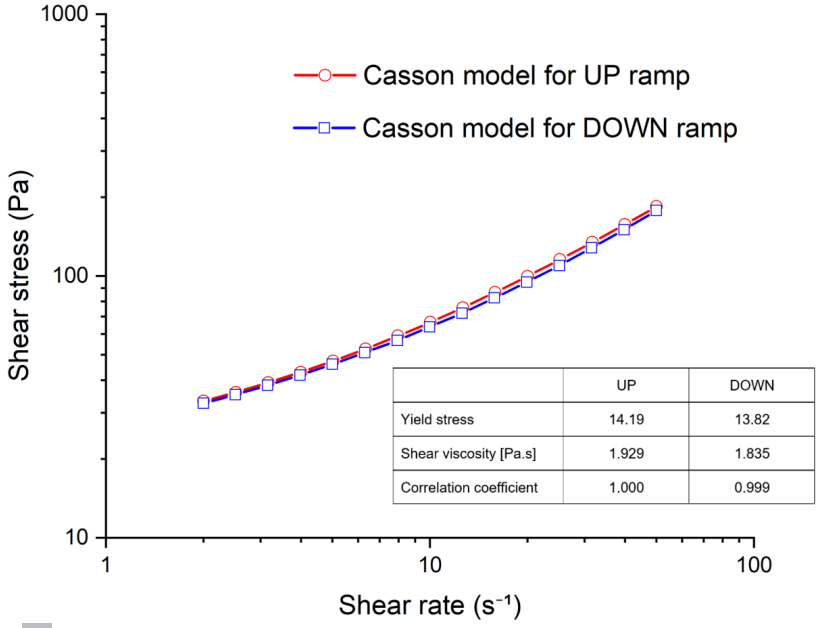
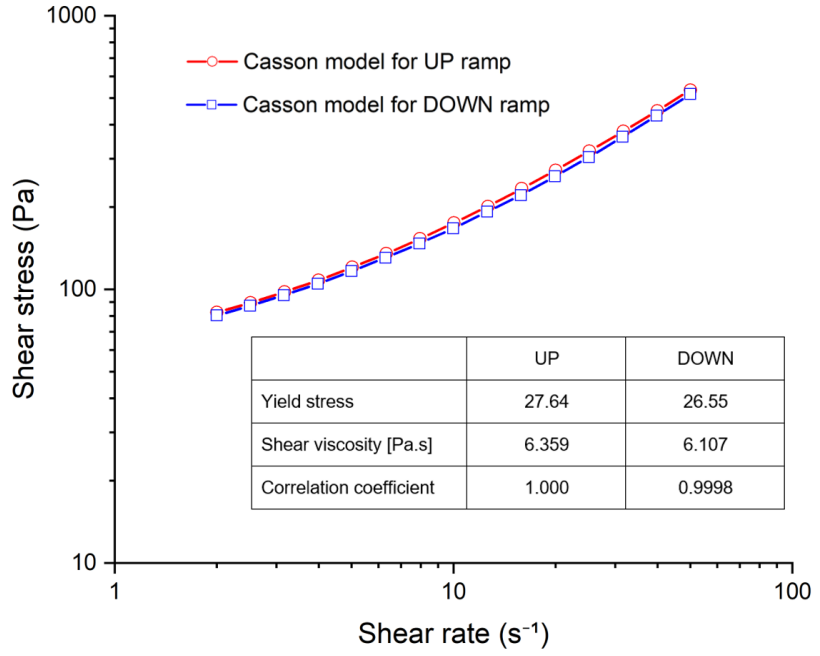
Figures 2 and 3 display the results of the Casson analysis for the shear rate range between 5 and 50 s-1 for the milk and the dark chocolates, respectively. The analysis is performed automatically in the rSpace measurement and evaluation software as soon as the measurement is finished.

Both the up and down tables of shear rate exhibit a good correlation between the measured flow curves and the Casson model. The yield stress of the dark chocolate is much higher than that of the milk chocolate, meaning that pumping would be more difficult, i.e., would require a higher force.



	UP	DOWN
Yield stress	14.19	13.82
Shear viscosity [Pa.s]	1.929	1.835
Correlation coefficient	1.000	0.999

2 Milk chocolate. Casson analysis for up and down tables of shear rates between 5 and 50 s^{-1}



	UP	DOWN
Yield stress	27.64	26.55
Shear viscosity [Pa.s]	6.359	6.107
Correlation coefficient	1.000	0.9998

3 Dark chocolate. Casson analysis for up and down tables of shear rate between 5 and 50 s^{-1}

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How to Check the Test Repeatability: Shear Rate- and Shear Stress-Controlled Measurements

A reliable method for checking the repeatability of the measurements is to perform them either with another geometry or with a different kind of control. This is because a shear viscosity at a specified temperature and shear rate is an absolute value. It is independent of the geometry used. Also, whether shear rate-controlled or shear stress- controlled, the same value must be obtained.

To illustrate this, a measurement was performed on dark chocolate in almost the same way. The only difference was that instead of an up and down table of shear rate, an up and down table of shear stress was applied. The resulting curves displayed in figure 4 exhibit good repeatability.

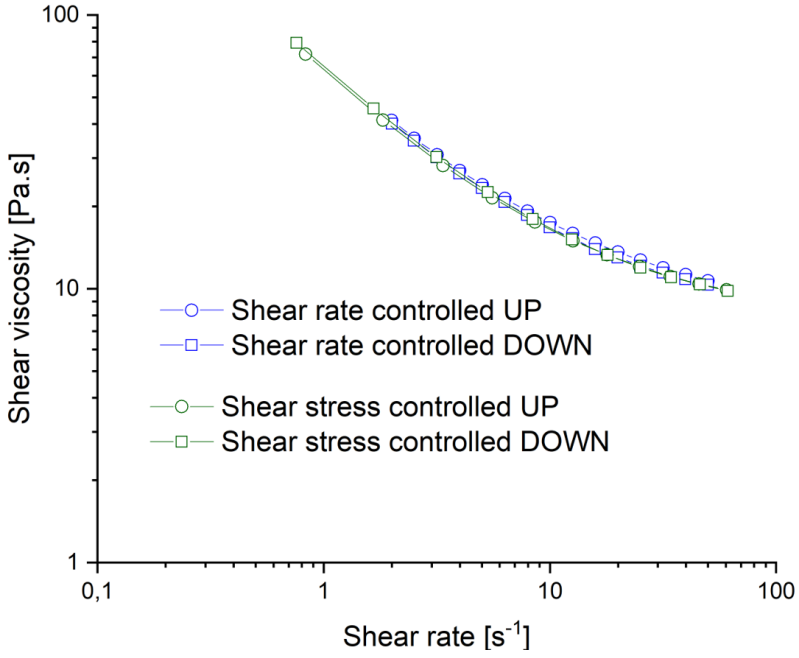
Conclusion

Shear viscosity curves performed with the Kinexus rotational rheometer are used not only to ensure consistent quality across batches, they also help predict the chocolate's behavior during processing as well as its mouth feel. So, a rheological understanding of chocolate is crucial for process optimization and product design.

The sequences as well as the evaluation of the Casson analysis which follows automatically after each measurement turns an initially complex method into a routine activity.

Literature

[1] International Office of Cocoa (IOC) (2000), Viscosity of cocoa and chocolate products, Analytical Method 46



4 Dark chocolate. Shear rate- and shear stress-controlled measurements.

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