Viscosity Measurements on Liquid Chocolate with the VT550

Eva-Maria Kutschmann, Gebrüder HAAKE GmbH, Dieselstrasse 4, D-76227 Karlsruhe

Conches are a suspension of solid particles (sugar, milk powder, cocoa particles) in a liquid fat phase, the cocoa butter. In addition, small quantities of water and lecithin may also be included.

For the processing properties and the final quality of chocolate products the flow behavior of this rheologically complicated system is of decisive importance. Today, modern instruments allow a systematic application of rheology in the processing of the raw material as well as the process design in order to produce final products of constant high quality at a low price.

Already in 1973 an expert commission of the OICCC (Office International du Cacao, du Chocolat et de la Confiserie) has proposed a working regulation for viscosity measurements on chocolate. According to this method which is mainly used in the chocolate industry today the flow curve is evaluated in a shear rate range of 5 to 60 s^{-1} with a curve fitting according to Casson.

Experts often criticized that the Casson parameters η_{CA} (Casson viscosity) and τ_{CA} (Casson yield point) obtained with this method do not describe the real flow behavior of conches accurately enough. The Casson parameters differ considerably from the real yield point τ_0 and the equilibrium viscosity η_∞. Especially in the shear rate range from 0.1 to 5 s^{-1} which is important for the bar forming process the Casson values can deviate from the real flow properties by more than 30%. Therefore, it must be emphasized that a Casson-fitting will only deliver meaningful results with the required accuracy of ±5% within the range of 5 – 60 s^{-1}. Fig. 1 shows a typical flow and viscosity curve of a milk chocolate coating obtained with the Casson evaluation. The values for the Casson yield point t_{CA} are within the range from 0 to 20 Pa , the Casson viscosity typically between 0.5 and 2.5 Pas. For chocolate used for bars the resp. values are higher. The literature states the following Casson parameter:

τ_{CA} = 10 – 200 Pa η_{CA} = 1 – 20 Pa

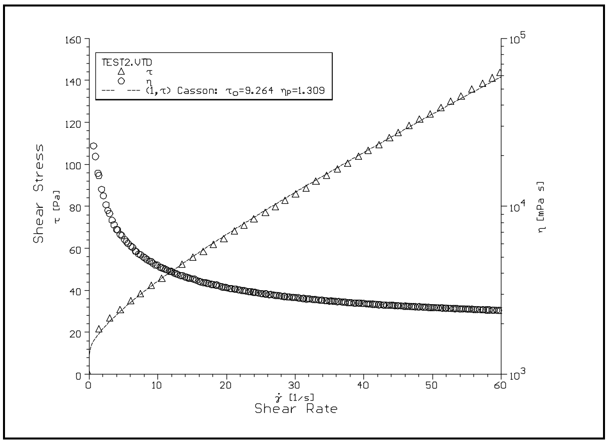

Fig. 1: Typical Flow and Viscosity Curves of a Milk Chocolate Coating at 40°C

Another frequently used method in the quality assurance is the indication of the viscosity at two (or more) exactly defined shear rate values. In the past, those shear rates resulted partly from the pre-defined fixed speed values of older HAAKE rotational viscometers; they remained unchanged and were used over many years. This procedure has proved to be of advantage if comparable statements regarding the quality of the products were needed between different production plants or between customer and supplier.

This thought of the comparability of measuring results between different laboratories was followed up again by the OICCC leading to a proposal for a new work regulation which is supposed to replace the old regulation of 1973 in the future.

The following procedure is proposed by the OICCC experts: The measurement should be performed with a rotational viscometer based on the Couette or Searl principle with cylindrical measuring systems at 40°C (±1°C). The used measuring system should have a ratio of radii (radius of rotor vs. radius of cup) of max. 1.2 and measuring gaps (difference of radii) between 1 and 2 mm. Using the HAAKE viscotester VT550 with measuring systems according to DIN 53019 will fulfill those requirements. The ratio of radii at those measuring systems is 1.0847 with gap widths of 1.64 mm (MV-DIN) resp. 0.9 mm (SV-DIN). From the measuring systems according to DIN 53018 the geometries of MV1, MV2 as well as SV1 and SV2 fulfill the OICCC requirements. A calculated correction of the shear rate is usually not necessary since at DIN rotors the shear rate will automatically be calculated according to the representative method in the center of the shear gap (see DIN 53019, part 1) by the HAAKE software. In this connection, however, it should be mentioned that when using different geometries especially in the low shear rate range (i.e. below 2 s^{-1}) the measuring results for the same sample can differ significantly. In this case it is recommendable to perform the measurements with the VT550 at higher shear rates or to calculate the shear exactly according to the correction formula given by the OICCC.

After filling the preheated cup (40°C) a pre-shearing of the sample should be performed, e.g. five minutes at a shear rate of 5 s^{-1}. On one hand the suspension can be homogenized once again that way, on the other hand this pre-shearing will lead to an accurate tempering.

In accordance with the recommendation from the OICCC we recommend to define the program with the VT 550 application software as follows:
1st segment CR time curve, 5 min at 5 s^{-1}
2nd segment CR-ramp, 3 min, 0 – 60 s^{-1}
3rd segment CR time curve, 1 min at 60 s^{-1}
4th segment CR-ramp, 3 min, 60 – 0 s^{-1}

Whether a flow curve can also be obtained at higher shear rates with the VT550 depends on the viscosity of the product and the measuring system used. Usually dark chocolates (coatings) can be measured with the MV-DIN-system up to abt. 120 s^{-1}; for higher viscous chocolates (e.g. milk chocolate, fillings) and measurements at higher shear rates the SV-DIN-system is preferable in order to be able to work in the optimum measuring range of the VT550.

With an interpolation the software will deliver viscosity values at defined shear rates. Special attention should be paid to the fact that for some conches the results of the up- or down-ramp may differ.

The following tables show some typical experimental data for different chocolate types:

Table 1: Dark chocolate coating, MV-DIN, measuring data from the up-ramp

Shear rate / s^{-1}	**Viscosity / Pas**
2	5.5
5	3.7
10	2.5
20	1.9
50	1.5

Table 2: Milk chocolate, SV-DIN, measuring data from the up-ramp

Shear rate / s^{-1}	**Viscosity / Pas**
2	20.3
5	13.4
10	9.5
20	7,3
50	5.7

Gebr. HAAKE GmbH, Tel. +49 721 4094-444
Dieselstr. 4, D-76227 Karlsruhe, Germany