Determining the yield point and viscosity of chocolate

Introduction
This report describes a measuring method for determining the yield point of chocolate. The yield point influences the way the chocolate levels out and how it melts in the mouth and on hands. Chocolate makers are also very interested in the spatial distribution of the cocoa butter molecules. They aim to produce a Beta V crystal structure, which melts on the tongue at temperatures between 29 and 33 °C. Without this crystal structure the chocolate tastes waxy or is too soft at room temperature. (Source: Windhab).

History
Cocoa was already used more than 3000 years ago by the Olmecs. This knowledge was transferred to the Mayas and Aztecs, who used cocoa beans to prepare a highly nutritious drink called Xocolatl, the root of today’s word ‘chocolate’.

Keywords
Beta V crystal structure, yield point, flow curve, ISO 3219 for cylinder measuring systems, chocolate, viscosity

Samples
All types of chocolate have one thing in common: A high calorie count. A 100 g bar contains around 40 g fat and 50 g sugar. The remaining 10 g are made up of flavorings.

Three commercially available sorts of chocolate were investigated: One white chocolate, one milk chocolate and one bittersweet chocolate.

All percentages given in this table are minimum values.

<table>
<thead>
<tr>
<th>Type</th>
<th>Cocoa paste</th>
<th>Cocoa butter</th>
<th>Milk content</th>
<th>Milk fat</th>
</tr>
</thead>
<tbody>
<tr>
<td>White</td>
<td>-</td>
<td>20 %</td>
<td>14 %</td>
<td>3.5 %</td>
</tr>
<tr>
<td>Milk</td>
<td>30 %</td>
<td>-</td>
<td>18 %</td>
<td>4.5 %</td>
</tr>
<tr>
<td>Bittersweet</td>
<td>50 %</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 1: Composition of types of chocolate

Test procedure
All measurements were performed with an Anton Paar RheolabQC rheometer with cylinder measuring system CC27 according to ISO 3219. This measuring system is an excellent substitute for the old systems (with a flat bottom design and an air bubble between the sample and cylinder bottom) which do not conform to ISO. One problem with the old measuring systems is the difficulty in positioning due to the very high axial forces in highly viscous chocolate mass.

ISO 3219 describes the construction of the cylinder geometry and defines the ratio of measuring cup diameter to measuring bob diameter as 1.0847. This guarantees an industrial standard for shearing in the measuring gap, independent of the measuring system size and manufacturer.

To keep cleaning to a minimum, disposable aluminum cylinders can be used. This means the measuring cup no longer requires cleaning. Many users clean their disposable cups in a dishwasher. High sample throughput with minimum required cleaning is guaranteed.

The measuring system is heated or cooled either in a water bath or directly in the instrument via a temperature-controlled measuring cell. The temperature of the measuring system can be controlled using the RheoPlus software. The use of defined temperature profiles allows diverse options for investigating the melting and crystallization properties of chocolate.

Test conditions
The measuring profile consists of 4 intervals.

1. Preshearing for 500 s at 5 s⁻¹ to homogenize and control the temperature of the sample. No measuring points are recorded in this interval.
2. Shear rate ramp of 2 s⁻¹ to 50 s⁻¹ with 18 points in 180 s. This is the main measuring interval. Interval 2 is analyzed according to method IOCCC2000.
3. Constant shearing at 50 s⁻¹, duration 60 s.
4. Shear rate ramp of 50 s⁻¹ to 2 s⁻¹, duration 180 s.

(*) IOCCC: International Office of Cocoa, Chocolate and Sugar Confectionery

The test is controlled manually at the instrument or via the RheoPlus software.

The measuring temperature is always 40°C (+- 0.1 °C), as the sample viscosity and determined yield point have a high temperature dependence of 5 % to 10 % per 1°C. The chocolate sample must be attemperated before filling it into the measuring cup. It is also recommended to attemperate the measuring system before the first measurement.
Results and discussion

Continuous quality control is essential in order to achieve consistently high quality despite the immense production volume. The viscosity of the chocolate is a particularly important parameter, especially during production. Emulsifiers are added to chocolate products. They are used to adapt the flow properties of the chocolate mass to defined requirements, influence the crystallization behavior and increase the storage stability. Emulsifiers decrease the yield point value. They work by reducing the interfacial tension between the hydrophilic solid particles (sugar) and the hydrophobic cocoa butter phase.

The investigated chocolates show significant differences in the viscosity and shear stress function (Fig. 1a/b). The viscosity values are calculated at high shear rates. The term „infinite-shear viscosity“ or ETA,inf is applied here. It is assumed that the viscosity approaches a constant limit value at high shear rates. The white chocolate sample (1a) shows by far the highest viscosity values.

The flow properties at „almost rest“, i.e. when the chocolate levels out, can be determined at low shear rates. This is usually called the yield point of the sample. White and milk chocolate show yield points which are almost identical whereas the calculated yield point of the bittersweet chocolate is considerably lower (1b).

<table>
<thead>
<tr>
<th>Type</th>
<th>Yield point at almost rest</th>
<th>Viscosity at high shear rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>White</td>
<td>24 Pa</td>
<td>4.2 Pas</td>
</tr>
<tr>
<td>Milk</td>
<td>23 Pa</td>
<td>2.9 Pas</td>
</tr>
<tr>
<td>Bittersweet</td>
<td>17 Pa</td>
<td>2.2 Pas</td>
</tr>
</tbody>
</table>

Table 2: Comparison of the yield points and viscosity values

Summary

It was shown that the RheolabQC with the ISO3219 measuring system CC27 is very suitable for characterizing chocolate. Besides measuring the flow and viscosity curves, the yield point can also be calculated according to the Casson model or according to IOCCC 2000 (Windhab Model). Consistently checking the yield point and viscosity value guarantees a constantly high chocolate quality.

Fig. 1 a/b: Viscosity curve (a) and shear stress as a function of the shear rate, with analysis of the yield point according to method IOCCC 2000 (b).

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