WHITE PAPER

NETZSCH

# Analytical Characterization of Mouthfeel in Chocolate

## The Particle Size and Rheology of the Chocolate are Critical Factors in Determining and Predicting Mouthfeel

### Introduction

Chocolate is one of the world's favorite snack foods: over $100 billion were spent on chocolate in 2015 [1]. The unique appeal of chocolate lies in its taste, aroma and mouthfeel, or texture. These three attributes combine into the complex flavor of chocolate. As the natural ingredients of chocolate vary according to growing conditions, chocolate manufacturers go to some lengths to ensure the flavor of their chocolate products is consistent with their signature flavor. As with many food products, consumers are intensely loyal to their favorite brands and resist any changes to the flavour they expect [2].

Ensuring that the signature flavour is replicated across batches requires correlation of analytical techniques with expensive sensory testing, since it is not feasible to taste test every batch that emerges from a chocolate factory, however desirable that job might be!

### The Importance of Particle Size and Rheology in Chocolate Manufacture

Several factors are considered important for increasing the appeal of chocolate. These include:

- Melting temperature of 37°C, so that it melts in the mouth
- Shine, so that it looks appealing
- Smooth texture, which gives a pleasant mouthfeel
- Snap, so there is an initial "bite" [3]

The textural component is critical: as consumers, we prefer a smooth chocolate to a "gritty" one, and we tend to assume a smooth chocolate is a more luxurious product. Extensive consumer testing by chocolate manufacturers over decades has established that particles of cocoa solids, sugar and milk are detected as a gritty mouthfeel at sizes over 30 µm. However, the particle grinding processes in chocolate manufacture are expensive, lengthy and energy intensive so largescale manufacturers optimize their processes to achieve the required particle size as efficiently as possible. This optimization is underpinned by regular particle size measurements, which are increasingly performed by laser diffraction instruments.

While particles affect grittiness, the flow properties of the fat phase (cocoa butter, which may be mixed with other fats) control how the chocolate coats the mouth and influences the perception of flavor. The flow, or rheological, properties of the chocolate also have significant impact on the chocolate manufacturing process. Reducing the particle size increases viscosity, potentially causing blockages as the liquid chocolate is piped through the factory. The final product may be a bar, or tablet, of solid chocolate, or the chocolate may be used in an enrobing process to surround a filling center. Chocolate for enrobing processes is often optimized to achieve good coverage and may have a different recipe than chocolate for tablets.

### Laser Diffraction

Laser diffraction enables rapid measurements of particle size across a wide dynamic range, conveniently and close to the production line. Samples can be obtained from intermediate steps in the process, such as after grinding, and from the final product to ensure that the particle size distribution meets the manufacturer's requirements.

The particles in chocolate are suspended in a matrix of fat. For measurement by laser diffraction (Mastersizer, Malvern Panalytical), shavings of chocolate are immersed in a miscible oil or alcohol and agitated to release the particles and suspend them. The suspended particles can then be circulated through the measurement cell of a laser diffraction system, where they will scatter the laser light. The spacing and intensity of the rings in the diffraction pattern is related back to the particle size distribution by use of optical models defined by Fraunhofer, the industry standard, or Mie. The particle size result is obtained within minutes of obtaining the sample, enabling constant monitoring of the production process and the product quality.



NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

### Particle Size in Different Types of Chocolate

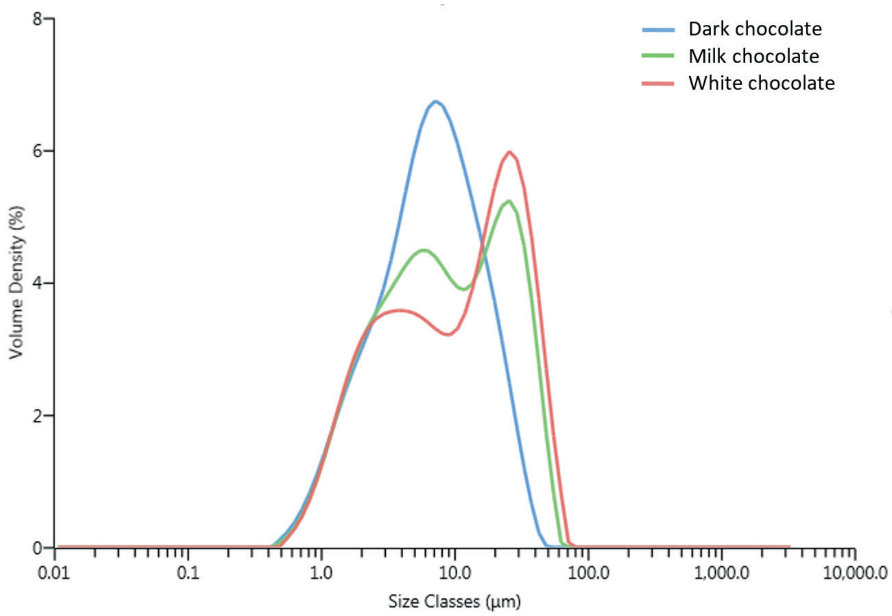
Dark, milk and white chocolates from a single range and brand of chocolate were selected for measurement to minimize variations in the chocolate manufacturing process and ingredients. The particulate ingredients of dark, milk and white chocolates are shown in table 1.

There are clear differences in the shape of the particle size distribution between the three types of chocolate (figure 1). There are common features: a mode of large particles at approximately 30 µm and a mode of smaller particles at approximately 5 µm. Note that the 30 µm mode is present as a slight shoulder in the dark chocolate. The proportion of these modes varies with the type of chocolate and may be related to the presence and proportion of the different ingredients. For example, the large mode appears to correspond to the increasing sugar content from dark to milk to white chocolate. As there is a much smaller proportion of these large particles in the dark chocolate, it will be perceived by consumers as having a smoother, and more luxurious, texture than the others.

The increase in particle size from dark to milk to white chocolate can be followed seen in Dv50 and Dv90, while Dv10 does not change significantly (table 2). As the main interest for chocolate manufacturers is detecting the presence of large particles, the critical parameter is often Dv95 or even Dv98, where we can detect significant differences between the chocolate types. Alternatively, the proportion above 30 µm can be obtained directly from the analysis.

**Table 1** The particulate ingredients in different types of chocolate.

| | Cocoa solids | Sugar | Milk powder |
|---|---|---|---|
| Dark | Y | Y | |
| Milk | Y | Y | Y |
| White | | Y | |




**1** The particle size distributions of different types of chocolate made by one manufacturer.

**Table 2** The percentiles and proportion of particles above 30 µm for different types of chocolate.

| | Dv10 [µm] | Dv50 [µm] | Dv90 [µm] | Dv95 [µm] | Dv98 [µm] | %>30 µm |
|---|---|---|---|---|---|---|
| Dark | 1.74 | 6.62 | 19.5 | 24.5 | 30.3 | 2.13 |
| Milk | 1.77 | 8.32 | 32.5 | 39.2 | 46.0 | 12.6 |
| White | 1.77 | 10.8 | 37.0 | 44.4 | 52.2 | 17.3 |



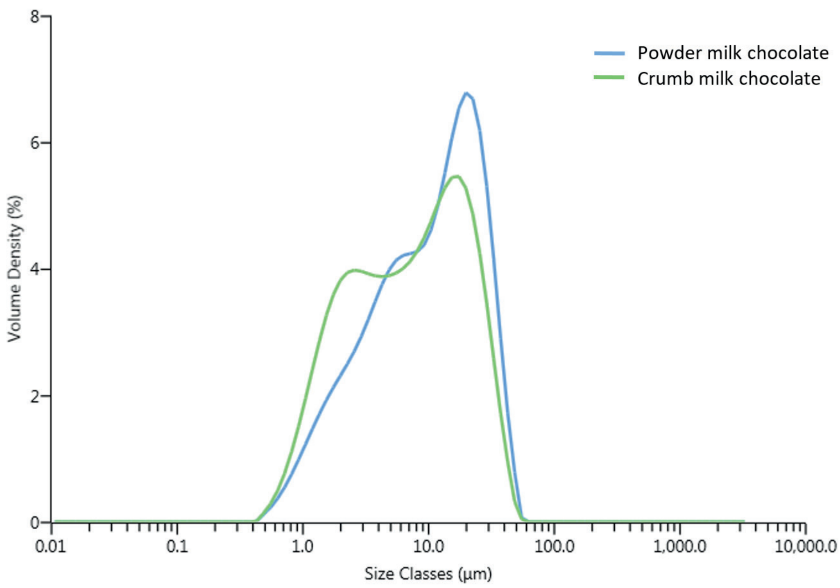
NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

**Particle Size in Powder and Crumb Milk Chocolates**

In the early days, the addition of milk was problematic because milk was a seasonal product and was out of season when the manufacturers wanted to make chocolate to sell at Christmas. Two solutions were found to this problem: the use of dried milk powder and the use of crumb. Crumb is made by drying milk, sugar and cocoa solids together, creating composite particles [4]. The dried crumb powder can then be stored for use throughout the year. The crumb process is more expensive and fresh milk is now available throughout the year. However, crumb gives a different flavor profile from the powder process and it is still used to maintain the signature flavor of particular brands.

The particle size distributions from powder and crumb chocolates made by the same manufacturer are shown in figure 2 and the particle size statistics are shown in table 3.

The mixed material nature of chocolate is again apparent with a multimodal particle size distribution. The two main modes are much closer together in powder chocolate than in the crumb chocolate. There is a tail of fines in the powder chocolate that isn't present in the crumb. These features give the crumb and powder chocolates each a distinctive profile in the particle size distribution and a distinctive texture. The source of these differences lies in the different recipe and processing routes. The crumb chocolate has a lower Dv90 than the powder chocolate and so will be perceived as being the finer chocolate by the consumer. This may be a true reflection of the quality of the chocolate, or it may be related to the flavor profiles and textures associated with each brand. In this case, it is likely that the crumb chocolate had longer, and more expensive, grinding stages than the powder chocolate, as the composite particles in crumb enter the process with a larger particle size than milk powder.




2 The particle size distributions of powder and crumb milk chocolates made by one manufacturer.

Table 3 The percentiles and proportion of particles above 30 μm for powder and crumb milk chocolate made by one manufacturer.

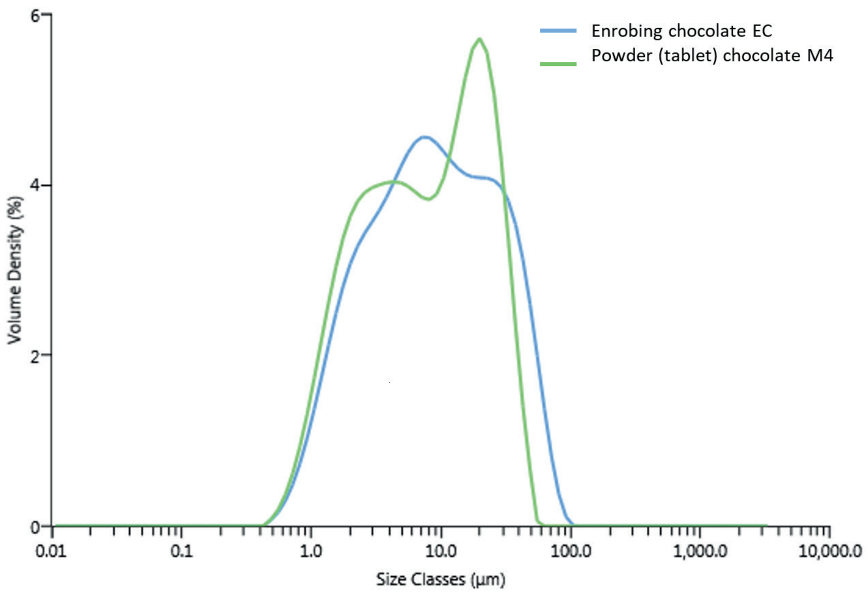
| | Dv10 [μm] | Dv50 [μm] | Dv90 [μm] | Dv95 [μm] | Dv98 [μm] | %>30 μm |
|---|---|---|---|---|---|---|
| Powder | 1.93 | 10.5 | 29.3 | 34.7 | 40.4 | 9.21 |
| Crumb | 1.48 | 7.22 | 25.0 | 30.6 | 36.8 | 5.43 |



NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

### Particle Size in Enrobing and Tablet Chocolates

Enrobing chocolate should flow well enough to easily encase the centers, but also be viscous enough to form a complete shell as the chocolate cools. A broader particle size distribution (see figure3 and table 4) generally results in a less viscous fluid and consumers are less concerned with a smooth mouthfeel when they are focused on the center of the chocolate. A chocolate with flow properties optimized for the enrobing process may have a different composition of fats as well as containing larger particles than a tablet chocolate.




**3** The particle size distributions of a powder (tablet) milk chocolate and an enrobing milk chocolate.

**Table 4** The percentiles and proportion of particles above 30 µm for enrobing and tablet milk chocolate.

| | Dv10 [µm] | Dv50 [µm] | Dv90 [µm] | %>30 µm |
|---|---|---|---|---|
| Enrobing | 1.77 | 8.56 | 37.9 | 15.7 |
| Tablet | 1.58 | 7.72 | 27.4 | 7.45 |



NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

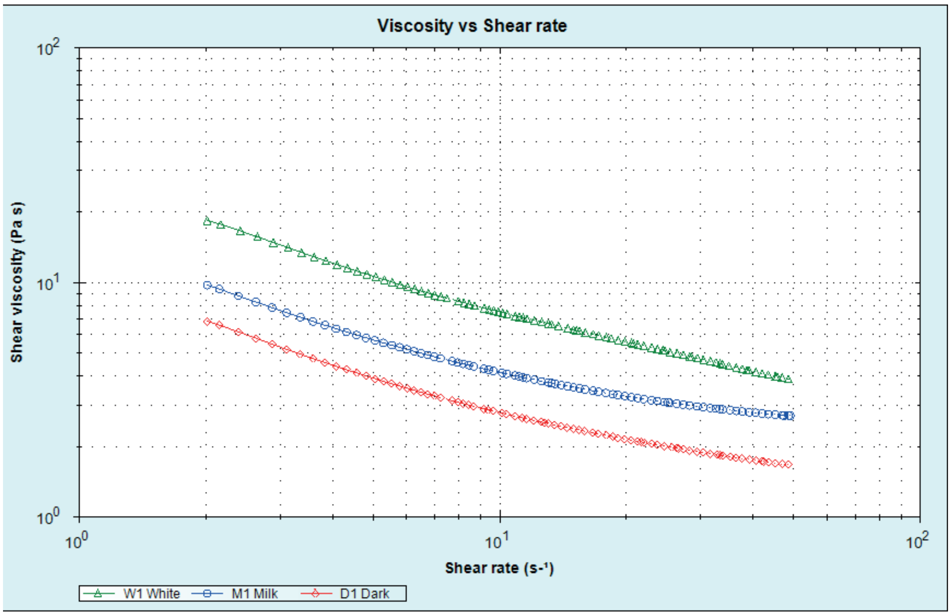
## Rheology

The most fundamental rheological measurement made on chocolate is a viscosity measurement. A small quantity of sample is sheared at a fixed rate (speed), and the stress (force) required to achieve this shear rate is measured. The shear viscosity can then be calculated by dividing the shear stress by the shear rate.

### Viscosity Versus Shear Rate

Dark, milk and white chocolates from a single range and brand of chocolate were selected for measurement to minimize variations in the chocolate manufacturing process and ingredient variation. The particulate ingredients of dark, milk and white chocolates are shown above in table 1.

The dark chocolate has the lowest viscosity across the measured shear rate range and will be easiest to pipe around the plant (figure 4). Interestingly, the milk chocolate has a higher viscosity but a similar yield stress to the dark chocolate (table 5). This suggests the milk chocolate and dark chocolate will have similar "slump" properties and will fill molds in a similar way. The white chocolate has the highest viscosity over the range and the highest yield stress by some margin.




**4** Viscosity vs shear rate for dark, milk and white chocolates produced by the same manufacturer.

**Table 5** Yield stress and shear viscosity for dark, milk and white chocolates produced by the same manufacturer.

| | Yield stress (Pa) | Shear viscosity (Pa· s) |
|---|---|---|
| Dark | 5.03 | 0.94 |
| Milk | 5.71 | 1.66 |
| White | 18.9 | 1.86 |



NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

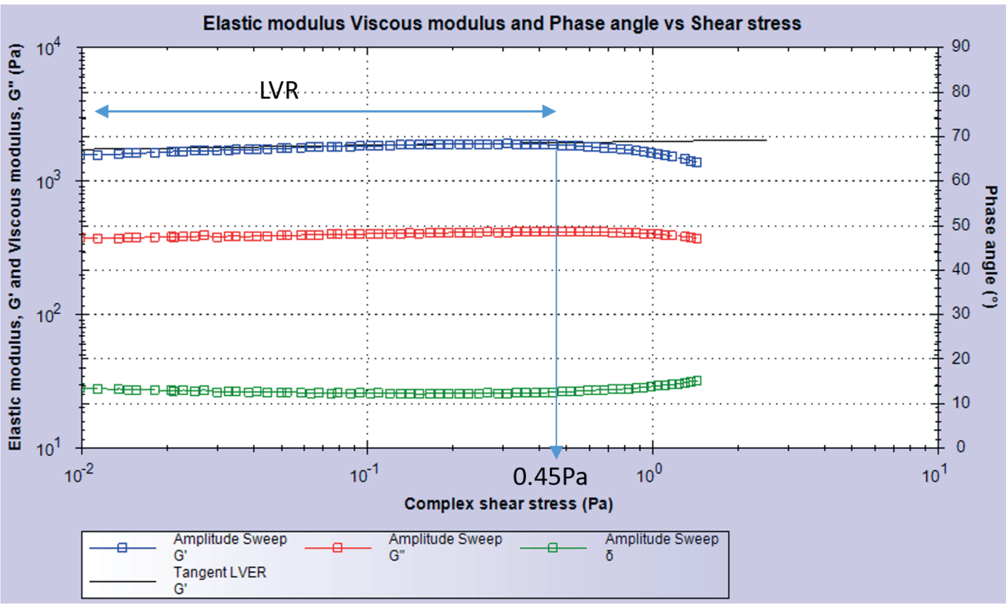
**Probing Microstructure and Onset of Flow by Oscillation Testing**

Further insights in to the rheological properties of chocolate can be determined using oscillatory testing on a rotational rheometer. This provides additional information about the viscoelastic properties of chocolate through the elastic modulus (G'), viscous modulus (G'') and phase angle (δ). It also provides an alternative method to steady shear testing for determining the yield stress.

The elastic and viscous moduli relate to the microstructural characteristics of the chocolate and can be used to probe component interactions and melting characteristics of chocolate by evaluating the solid-like and liquid-like properties.

The yield stress, which is the stress is required to break down the solid structure and make it flow, influences how the chocolate will coat the molds and how well the chocolate will cling to the walls of the mold or slump before it sets.

Oscillatory measurements are non-destructive tests which show how the material behaves under small deformations or forces – before the material yields and starts to flow. The stress or strain region in which this behavior occurs is known as the linear viscoelastic region (LVR). By measuring G' as a function of shear stress, the yield stress of the structure can be determined – this is generally taken as the stress at which G' starts to drop and the LVR ends [5]. It is important to note that different yield stress methods may give slightly different answers. For example, Casson and Winhab models are commonly used for determining yield stress of chocolate by extrapolating a steady shear plot of shear stress vs shear rate to zero shear rate. Although oscillatory testing is less common it has been shown to be able to better resolve differences between chocolates [6]. For the milk chocolate tested, the yield stress was found to be 0.45 Pa (figure 5).




5 Amplitude sweep for milk chocolate showing the LVER and yield point at 0.45 Pa.



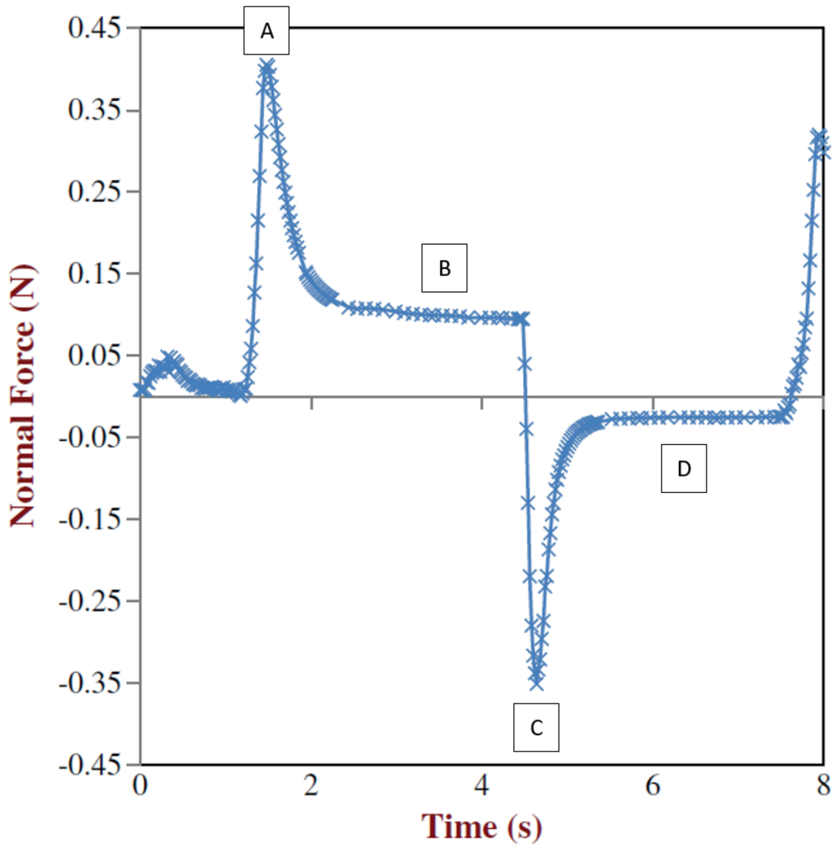
NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

### Simulation of Mastication by Normal Force Measurements

Extending conventional rotational rheometry by employing the axial capabilities of the Kinexus enables mastication, or the action of chewing, to be simulated [7]. From this test, the hardness, yield stress, coating of the roof of the mouth and stickiness of the chocolate can be objectively assessed.

Monitoring the normal force generated during a measurement gives an indication of how the chocolate will melt, flow and coat the inside of the mouth as it is eaten. In this cyclical test, the material experiences both shear and normal (squeeze) forces. As the gap is compressed, the normal force increases (figure 6, point A) and we might call this the hardness or consistency of the sample. The residual force after compression is the yield stress of the chocolate (figure 6, point B).

The area under the curve as the normal force reaches zero indicates how long the chocolate remains in contact with the upper plate. This mimics how the chocolate coats the roof of the mouth. As the gap is increased once again, a negative normal force is recorded which could be classed as the adhesiveness (figure 6, point C). The residual force after decompression is the "stickiness" of the chocolate (figure 6, point D).



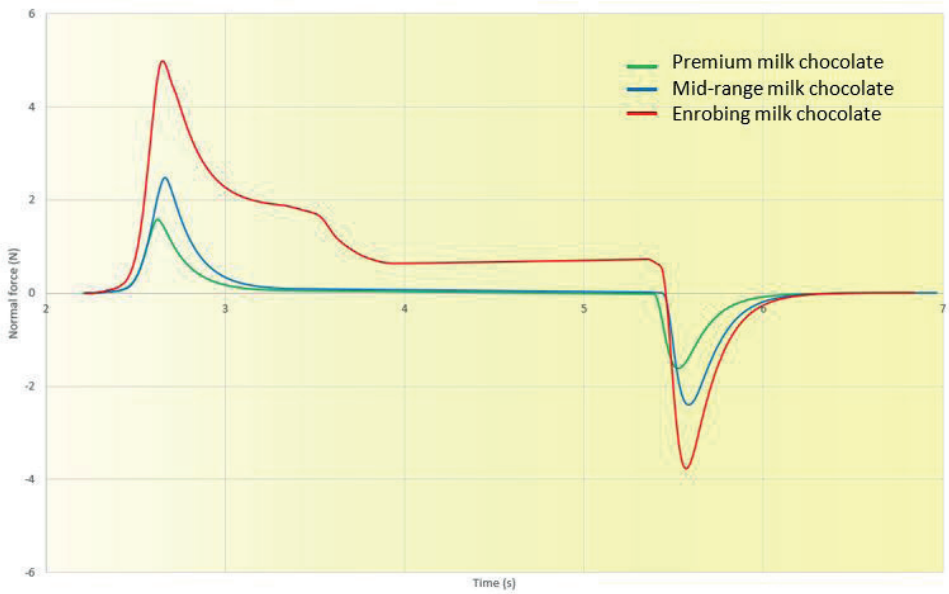

6 The mastication test. A = consistency, B = yield stress, C = adhesiveness, D = stickiness [6].



NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

Three different milk chocolates were subjected to mastication simulation by conducting a normal force and shear measurement on the Kinexus (figure 7). The premium chocolate has the softest consistency and the enrobing chocolate is significantly harder. The yield stress of the enrobing chocolate is also high and this chocolate will coat the mouth for longer than both the tablet chocolates, giving the consumer a satisfying chocolate hit despite the relatively low proportion of chocolate in the product (this product will contain a center). The premium chocolate has the least adhesiveness and stickiness. This is desirable as high adhesiveness or stickiness can be associated with a cloying mouthfeel.

As there is less chocolate present in the consumption of an enrobed chocolate product, these parameters have less influence on the consumer experience than in a chocolate tablet.




**7** Normal force vs experiment time for milk chocolates during a mastication simulation test

## Summary

The texture, or mouthfeel, of chocolate is critical for the consumer perception of product quality. By correlating expensive sensory testing with analytical results, it is possible to finely control the textural aspects of the signature flavor of chocolate brands. The mouthfeel can be characterized through the determination of particle size, by laser diffraction, and flow, by rheology. It is even possible to use rheological methods to simulate mastication and thus predict or control the structural changes in the chocolate as it is consumed.

## References

[1] www.theguardian.com/news/datablog/2015/jul/19/which-country-spends-themost-on-chocolate-bars
[2] www.independent.co.uk/news/uk/home-news/creme-egg-easter-cadburymondelez-recipe-sales-a6950221.html
[3] www.howitworksdaily.com/why-is-chocolate-so-tasty/
[4] Beckett, S. (2008) The science of chocolate (2nd edition) RSC Publishing
[5] Chan, F. and De Kee, D. (1994) Yield stress and small amplitude oscillatory flow in transient networks. *Industrial & engineering chemistry research*, 33(10), p2374-2376
[6] De Graef, V., Depypere, F., Minnaert, M., & Dewettinck, K. (2011). Chocolate yield stress as measured by oscillatory rheology. Food Research International, 44(9), 2660–2665.
[7] https://www.malvernpanalytical.com/en/products/product-range/mastersizer-range





NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Phone.: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com