Thermo Fisher SCIENTIFIC

Food Rheology Applications

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The world leader in serving science



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Rheology

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Loaf bread slices (TPA and firmness)

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Tribology

Butter Milk Products

Rheology & Texture

Cheese (milk-based and analogue vegan)

Plate/plate (large gap)

Plate

Plate

Ball-on-3-pins

Serrated plate/plate, vane



Bread spread, cream cheese, mayonnaise (spreadability)

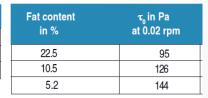
CR-Rotation vane rotor testing in original container holder

Chocholate spread	τ _o in Pa	t in s
Α	364	117
В	722	73

Tab. 2: Comparison of FL 22 vane rotor yield stress measurements with 0.05 rpm for chocolate hazelnut spread products

Rasberry jam	τ _o in Pa	t in s
With seeds	590	100
Sieved	967	104
Sieved (measurement 2)	977	104

Tab. 1: Comparison of FL 22 vane rotor yield stress measurements with 0.05 rpm for a sieved raspberry jam and one with seeds



mayonnaises with three different fat contents

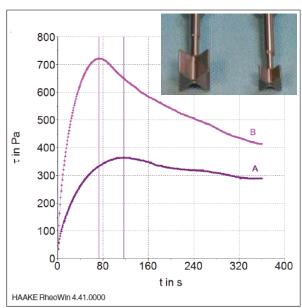


Fig. 5: FL 22 vane rotor yield stress measurements with 0.05 rpm for chocolate hazelnut spread products A and B

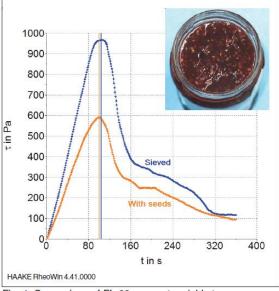


Fig. 4: Comparison of FL 22 vane rotor yield stress measurements with 0.05 rpm for a sieved raspberry jam (blue curve) and raspberry jam with seeds (orange curve, inserted image)

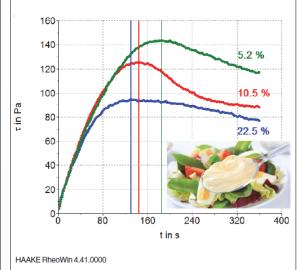


Fig. 6: Vane rotor yield stress measurements with 0.02 rpm for mayonnaises with three different fat contents

PLICATION NOTE

Spreadability of cream cheese -Influence of temperature and fat content

APPLICATION NOTE

Investigation the effect of fat content on the yield stress of mayonnaise measured with HAAKE Viscotester iQ Rheometer and vane rotor

APPLICATION NOTE

Yield stress of jam, chocolate spread and peanut butter measured with HAAKE Viscotester iQ Rheometer and vane rotors



Thermo Scientific Rheology Application Note V238 Applied Food Rheology Using Fast Speed Control and Axial Measurements Thermo Scientific Rheology Application Note V268 Spreadability of Cream Cheese - Influence of Temperature and Fat Content Thermo Scientific Rheology Application Note V271 Investigation of the effect of fat content on the yield stress of mayonnaise Thermo Scientific Rheology Application Note V272 Yield stress of jam, chocolate spread and peanut butter

Chocolate spread, mayonnaise (viscosity curve, stress plateau)

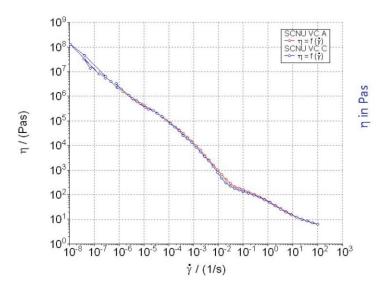
CR-Rotation equilibrium steps 🔀 (are more exact than ramps 🎇), air bearing rheometers



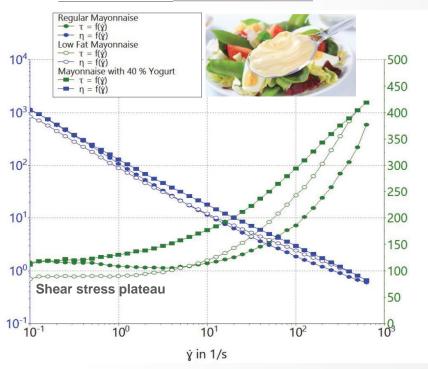


- Plate/cone measuring geometry temperature-controlled
- Viscosity decreases by 7 or 3 orders of magnitude, resp.

Viscosity curve of a chocolate spread. MARS 60 with plate/cone C35/1° at 35 °C



Mayonnaise type	Steady-state step-test stress plateau
Regular	116 Pa
Low fat	89 Pa
40% yogurt	119 Pa



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APPLICATION NOTE

Rheological and textural properties of various food formulations analyzed with a modular rheometer setup

Thermo Fisher Scientific, Karlsruhe, Germany

Rheology, food, viscosity, yield stress, texture analysis

and textures, the range of rheological methods used to

Rheological and texture properties play an important role during the entire life cycle of liquid or solid food formulations. Starting with simple single point viscosity measurements in the original containers for batch release in production. over the determination of classical rheological parameters like shear viscosity or yield point for quality control purposes, the mechanical testing of foods reaches a certain level of complexity with comprehensive rheological investigations for the development of new formulations in the research and development department.



Figure 1: HAAKE MARS IQ Rheometer with Peltier plate temperature control for use with parallel-plate and cone-and-plate geometries



While some methods rely on classic rheometer geometrie like parallel plates, cone & plate or coaxial cylinders, some other methods try to emulate a certain application by application is studying the texture of food products, which designed probes a rotational rheometer can test various important food properties such as softness, stickiness or spreadability, and can even be utilized for performing

In this application selected rheometer accessories and reviewed using a modular rheometer designed for product measuring "classic" rheological properties such as viscosity prehensive mechanical investigation of food formulations

ed using the Thermo Scientific™ HAAKE™ MARS™ iQ Rheometer with a mechanical bearing (Figure 1). The tests to be performed included viscosity and yield stress measurements, axial bending, breaking and squeezing tests as



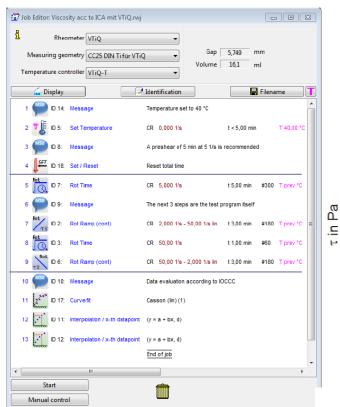
Thermo Scientific Rheology Application Note V238 Applied Food Rheology Using Fast Speed Control and Axial Measurements Thermo Scientific Rheology Application Note V292 Rheological and textural properties of various food formulations analyzed with a modular rheometer setup

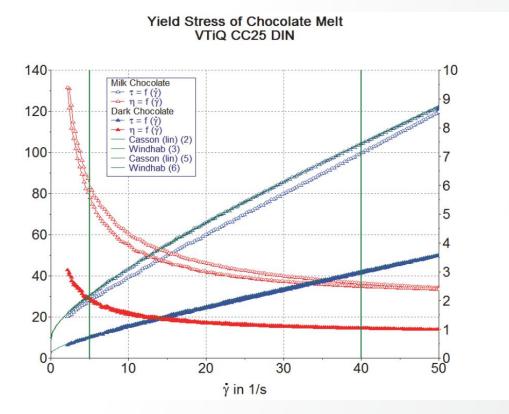
Chocolate (ICA standard method 46 International Confectionery Association)

CR-Rotation ramp, **thixotropy** loop area, curve fits and interpolation for **yield stress** determination

- Concentric Cylinder measuring geometry @ 40°C
- **Push Button** for sample conditioning, measurement, evaluation & documentation

	Milk Chocolate	Dark Chocolate
τ ₀ Casson / Pa	8.9	2.1
τ _o Windhab / Pa	14.7	4.0
$τ_0$ Servais et al./ Pa	30.0	10.4





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APPLICATION NOTE

Flow behaviour of chocolate meltsworking according to ICA standards

Author: Klaus Oldorp

Chocolate, QC, ICA standards, viscosity, yield stress

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™ HA AKE™ Viscotester™ iO Rheometer (Figure 1) has been developed. This viscometer includes features especially designed for QC applications. For example, due to its improved sensitivity it is possible to



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 the HAAKE Viscotester iQ Rheometer in the Peltier temperature

volume, time for temperature equilibration and cleaning

effort, Also, with improved sensitivity smaller shear rates

are accessible, which improves the reliability of yield stress calculations [1] with extrapolation methods like the Cassor



Thermo Scientific Rheology Application Note V269 Flow Behaviour of Chocolate Melts – Working according to ICA Standards

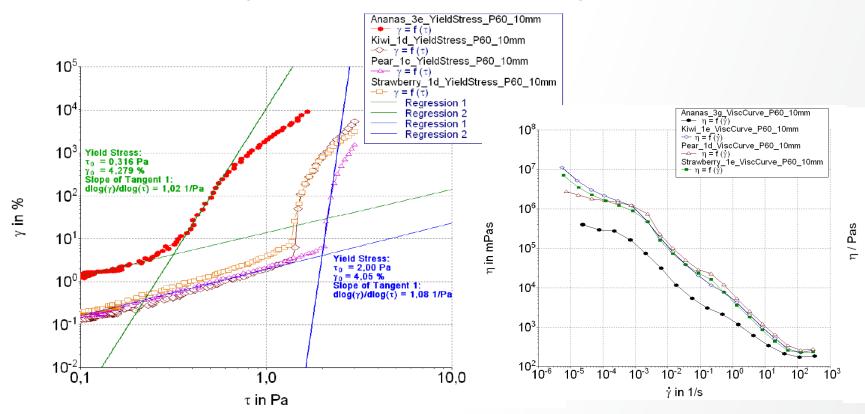


Fresh fruit juices, tomato sauce with tomato & onion pieces

CR-Rotation viscosity curve: large gap plate/plate meas. geom. TMP60 DC (10 mm rim) + P60 rotor

CS-Rotation ramp for yield stress measurement and stability prediction

- Due to large particles (several mm) absolute measurements are not possible
- Fresh pineapple juice with fruit particles (4 mm gap)
- Tomato sauce with larger tomato & onion pieces (10 mm gap)





^γ/ 1/s

10⁴

10³

 10^{2}

10¹

10°

Lab Report Thermo Fisher Scientific Rheology AppLab, Karlsruhe, Germany

Thermo Fisher

Starch (temperature sweeps)

CR-Rotation with vane in temperature-controlled cup

CD-Oscillation plate/plate temperature sweeps with polarization light microscope

Temperature induced transition crystallin/amorphous

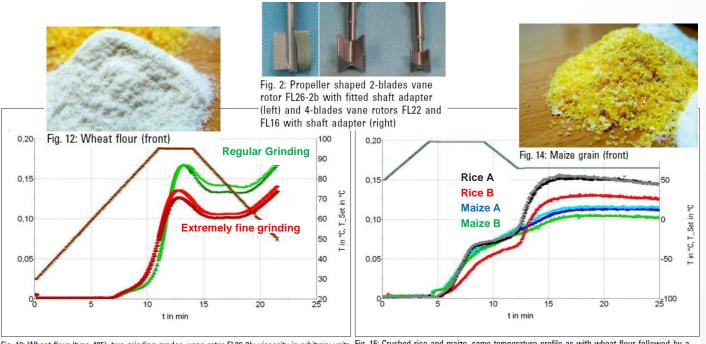


Fig. 13: Wheat flour (type 405), two grinding grades, vane rotor FL26-2b; viscosity in arbitrary units Fig. 15: Crushed rice and maize, same temperature profile as with wheat flour followed by a constant temperature of 65 °C, FL22 vane rotor; viscosity in arbitrary units

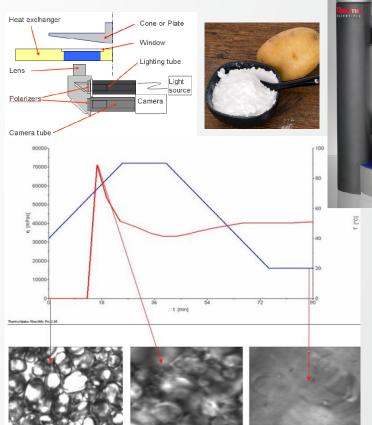


Figure 5: Native potato starch (5 % in water): Photos show the starch crystals in the beginning, the swollen crystals at peak viscosity and the inhomogeneous solution after cooling down.

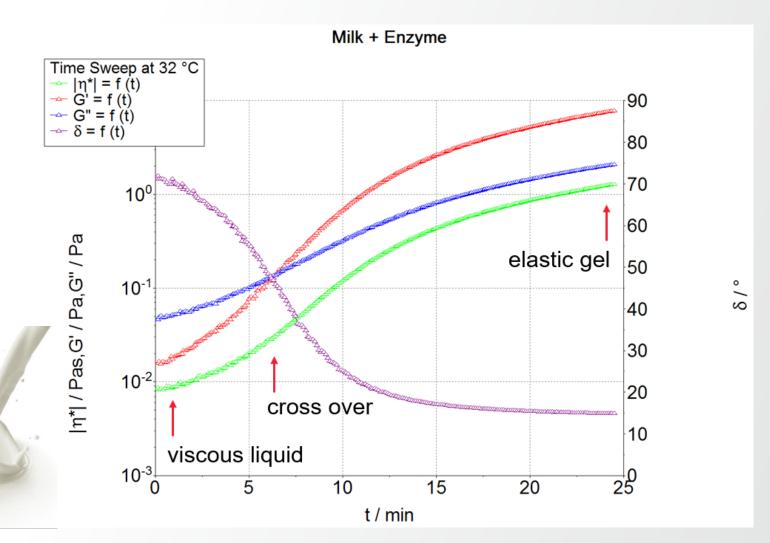
Thermo Scientific Rheology Application Note V228 Rheooptical Tests during the cooking of starch and the crystallization of fats Thermo Scientific Rheology Application Note V255 Rheological investigation of starch swelling, gelatinization and retrogradation



Milk, yoghurt, cheese, fat, dough (gelation, baking, crystallization)

CD-Oscillation time sweeps or temperature sweeps, plate/plate measuring geometry

- Oscillation
 - does not affect the development of the structure
 - Yields valuable information about the structure (e.g. strength)
- Gelling of protein (see graph)
- Baking of dough (e.g. flour batter)
- Crystallization of fat (cooling run)
- Solid elastic samples (e.g. cheese)



Marshmallows (chewing simulation)

in mm

Thermo Fisher

Texture analysis with a **plate**

AXIAL h-controlled lift movement

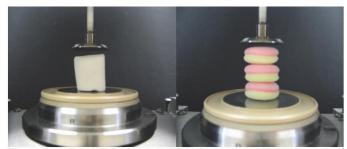


Fig. 1: Samples M (left) and S (right) before measurement.

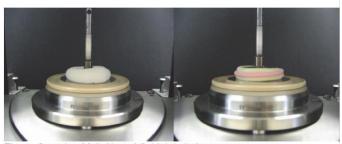
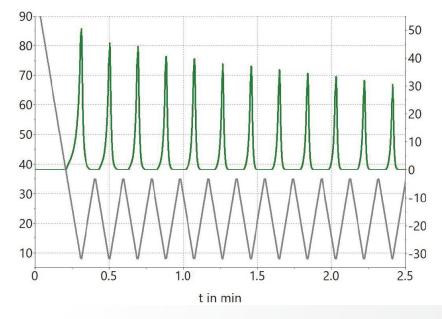


Fig. 2: Samples M (left) and S (right) during measurement.

Repeated Compression of a Marshmallow



A Rheometer with Bite - Marshmallows in the Rheometer

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

We use many of our senses to experience our food. For a food product to be popular and therefore successful, it must meet many requirements: it has to look appealing, smell good, have a pleasant texture in the mouth - or "mouth feel" - and of course taste good. As soon as a food product is perceived as being "unpleasant" during even one of these subjective "tests," it can quickly lead to consumer rejection.

When regarding natural products like bread, meat or cheese, certain fluctuating or individual "bad" qualities are sometimes accepted, such as the smell of certain cheeses. However for industrially-produced foodstuffs, and particularly when dealing with treats such as sweets, all of the above criteria have to be met to gain consumer acceptance. No matter how good something looks or tastes, the "mouth feeling," i.e. the texture in the mouth, has to be right, otherwise the pudding will remain on the shelf in the refrigerated section and the cookies will never be purchased.

Marshmallows are another example of food where not only the flavour and sweetness, but also the "bite", is critical for enjoyment. Using a rheometer or a testing machine such as a texture analyzer, this consistency or texture can be described using objective parameters and thus making quality control or targeted improvement possible. A modern rheometer allows various options for characterising marshmallows and similar products. in an oscillatory test.

By combining a sensitive normal force sensor with a highprecision lift drive, the Thermo Scientific™ HAAKE™ MARS™ provides the additional option of stressing samples axially, i.e. by exerting a vertical force on them from above at a maximum of 50 N (which corresponds to a weight of 5 kg) by either pressing on or pulling the required to compress sample S; the sample is thus felt to marshmallow sample. In this type of situation, the Thermo Scientific HAAKE MARS measures the axial force and position of the measurement geometry precisely while the the second cycle to 40 N during the fifth cycle. sample is being squeezed, or the penetration of a probe into the sample is tracked.



Two products were compared during the experiment. Marshmallows (M) of about 30 mm in height and a sugar foam product (S) of about 10 mm in height. In order to compare the absolute values discs were cut from product S matching the diameter of the marshmallows and were stacked three high (Fig. 1), A 35 mm-diameter plate as used for rotational and oscillation measurements was run down It also allows the viscosities of the starting materials to be to a height of 7 mm (Fig. 2) at a speed of 1 mm, and then measured, and the finished product can be characterized unun back up to 30 mm at 1 mm/s. This process was repeated on the samples five times in a row to simulate chewing in

When compressing first sample M, the force increased to about 45 N before dropping again. During the following cycles the force only reached about 40 N but remained nearly constant through each cycle. A force of 50 N is be somewhat harder. The maximum applied force decreases throughout subsequent cycles, dropping from 44 N during



Thermo Scientific Rheology Application Note V243 A Rheometer with Bite – Marshmallows in the Rheometer Thermo Scientific Rheology Application Note V292 Rheological and textural properties of various food formulations analyzed with a modular rheometer setup

Loaf bread slices (TPA and firmness)

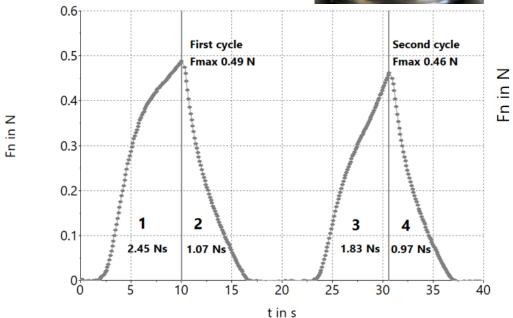
Texture analysis with a **plate**

AXIAL h-controlled lift movement

Texture profile analysis (TPA)

Two-bite test (2 compression cycles of a cylindrical sample by 4 mm (30 %) with 0.4 mm/s)

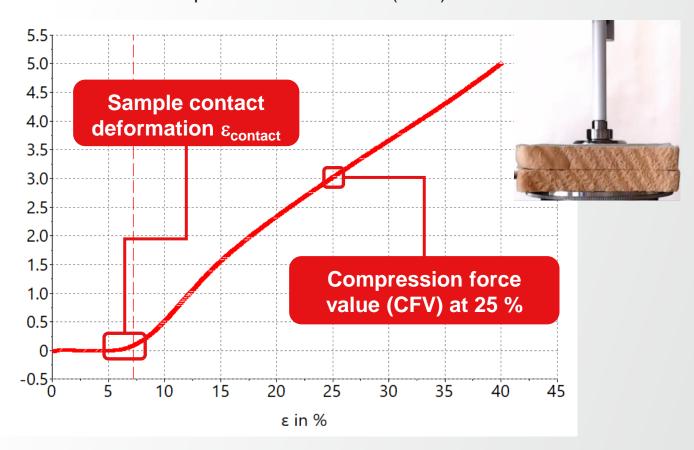




Lab Report Thermo Fisher Scientific Rheology AppLab, Karlsruhe, Germany

Firmness test based on AACC 74-09

Compression ϵ of two slices of loaf bread with 100 mm/s up to 40 % of the initial gap height and evaluation of compression force value (CFV) at ϵ = 25 %





Bread roll, apple, margarine (penetration, indenter test)

Texture analysis with an indenter

AXIAL h-controlled lift movement

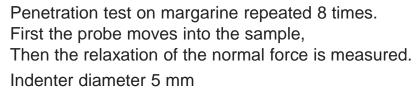
Maximum force	t in s	F in N
Upper half	8.2	6.0
Lower half	5.8	4.6

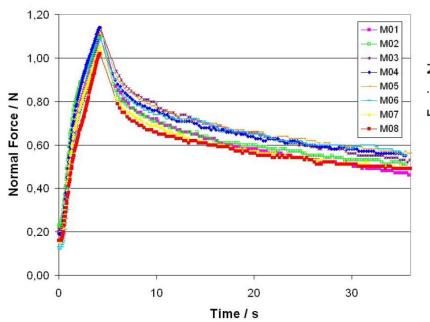
Crust : $F_{Upper \ half} > F_{Lower \ half}$

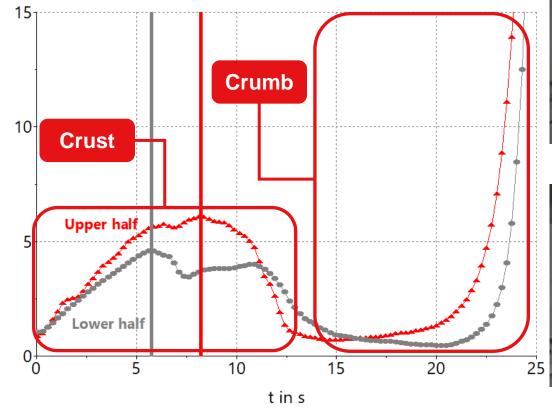
Crumb: Compression of the inner part

Indenter diameter 6 mm

Lift speed 1.2 mm/s







Upper half



Lower half

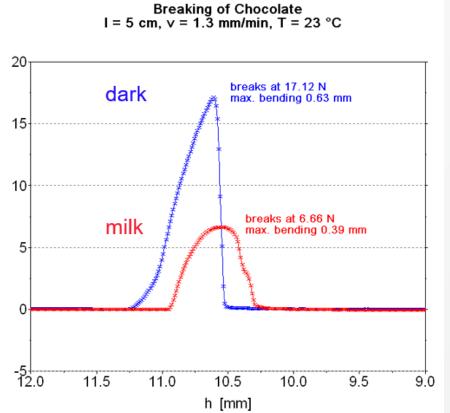
Thermo Scientific Rheology Application Note V238 Applied Food Rheology Using Fast Speed Control and Axial Measurements Lab Report Thermo Fisher Scientific Rheology AppLab, Karlsruhe, Germany

Chocolate (breaking test)

Texture Analysis - Chocolate experts can judge the quality of a chocolate bar by breaking off a piece

- AXIAL h-controlled lift movement with 1.3 mm/min, T = 23°C
- Indenter diameter 5 mm
- Force for breaking correlates with sound of empirical breaking test





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APPLICATION NOTE

Breaking strength of chocolate

Authors: Sabine Dohmen, Klaus Oldörp, Hans-Michael Petri

Chocolate appeals to all of our senses. We see the colour and the glossy surface of the bar, we smell the elusive flavours, we weigh it in our hands and feel how it melts, we hear it break and sense its resistance, and savour the taste. With so much "sensuality", it is easy to forget how quickly the pleasure can be destroyed, if even just one of these properties is not as we expect it or are accustomed to.

Even unconsciously, the breaking behaviour of chocolate plays an important role in influencing the consumer's impression. Chocolate experts can even evaluate the quality of a sample by breaking off a piece.

Rheology plays an important role in several steps of chocolate production. The liquid chocolate formulation and the fats used can be characterized by their viscosities, yield stresses and solidification behaviours. These parameters are important for quality control and processing, and can be determined using rotational or oscillation measurements.



process, or "mouth feeling", can be described with the viscosity curves and the yield stress. However, these rheological parameters contain no information about the breaking strength of the final chocolate bar—and therefore a new method and new equipment are necessary to assess the resistance to break.

Sensory properties as experienced during the melting

The Thermo Scientific™ HAAKE™ MARS™ Rheometer, manufactured by Thermo Fisher, features a highly sensitive normal force sensor and a very precise lift motor which allows the customer to apply controlled axial forces to the sample, pushing or pulling it, and to analyze its axial deformation.

For example, with a new measuring geometry (Figure 3), chocolate bars can be positioned on the rheometer and submitted to an increasing axial force until they break. This measuring geometry [1] consists of two parallel support bars which can be mounted onto a base plate in a variable



Figure 1: The HAAKE MARS Rheometer.

Thermo Scientific Rheology Application Note V222 Breaking Strength of Chocolate
Thermo Scientific Rheology Application Note V238 Applied Food Rheology Using Fast Speed Control and Axial Measurements

Cookies (breaking test, crunchiness)

Texture Analysis

- **AXIAL** h-controlled lift movement, indenter
- Fresh cookies (reference)
- Aged cookies (exposed to air/humidity) more elastic
 - Good Reproducibility
 - 22 % higher force Fnmax
 - 8 % more bending ∆h_{max}

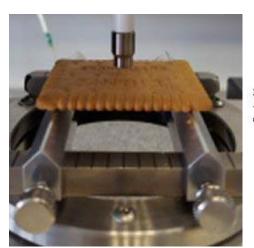
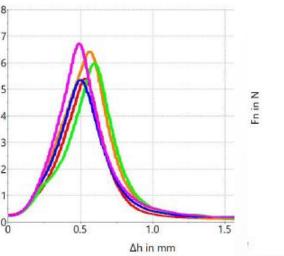
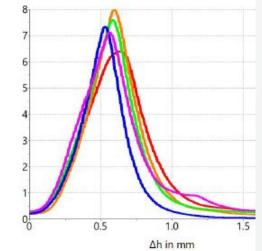


Table 1: Amount of bending and force at curve maximum of fresh and aged biscuits.

	Fresh biscuits		Biscuits aged for 14 day	
No.	Δh / mm	Fn / N	Δh / mm	Fn / N
1	0.532	5.384	0.633	6.395
2	0.563	6.395	0.596	7.927
3	0.596	5.943	0.584	7.545
4	0.501	5.332	0.530	7.289
5	0.490	6.684	0.562	7.065
Mean value	0.537	5.948	0.581	7.244
StdDev	0.039	0.536	0.034	0.512
rel. StdDev	7.31 %	9.02 %	5.90 %	7.07 %







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APPLICATION NOTE

Crunchiness of biscuits - Texture analysis on a rheometer

Thermo Fisher Scientific, Karlsruhe, Germany

Cookies, biscuits, crunchiness, texture analysis, bending

Introduction

Biscuits or cookies, no matter what they are called, they need to have the right taste and the right bite. While the first is a rather subjective matter and can lead to lengthy discussions, the latter can be tested in a very objective way with a rheometer.

Apart from the classic measuring parameters shear rate, deformation and stress, a modern rheometer comes equipped with an additional sensor to detect normal force. Thus, combined with its precise lift a rheometer can do texture analysis as well.





For testing biscuits, a Thermo Scientific™ HAAKE™ MARS™ iQ Rheometer (Figure 1) was equipped with the 3-point bending-accessory [1], using the 8 mm plate as the probe (Figure 2). Other probes can easily be adapted to the measuring head using the universal adapters U1 with a 6 mm bore or U2 with a 4 mm bore.

The measuring protocol starts with zeroing the normal force and lowering the probe to a position still high enough to allow a quick placement of a fresh biscuit onto the lower supports of the 3-point-bending accessory. For the tests discussed here, a position about 2 mm above the biscuits upper surface has been used.

The lower supports of the 3-point-bending-tool were positioned 5 cm apart from each other to support the biscuit approximately 0.8 cm from its edges. The biscuit was placed onto the lower supports with its centre under the probe (Figure 2).



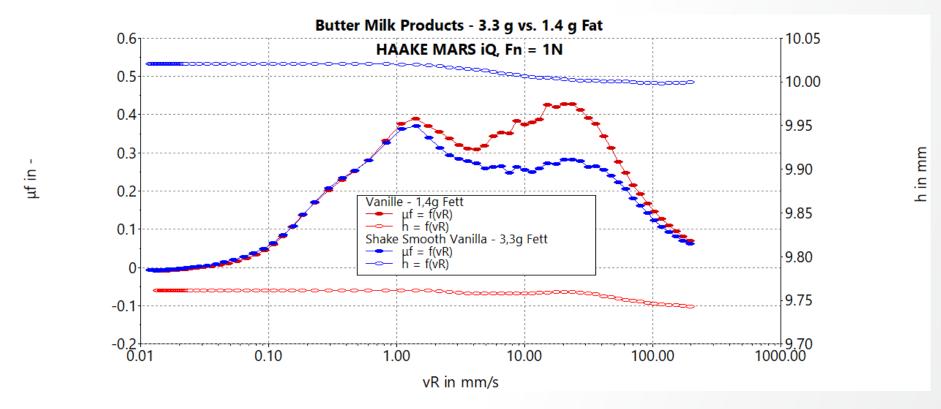
Thermo Scientific Rheology Application Note V292 Rheological and textural properties of various food formulations analyzed with a modular rheometer setup Thermo Scientific Rheology Application Note V293 Crunchiness of biscuits – Texture analysis on a rheometer

Tribology



Different Tribology measuring geometries available – here: **Ball-on-3 Pins** geometry

- 3D printed (rapid prototyping), machined version available
- Rotor with ceramic shaft, metal spring and holder for a ½" ball (here: glass)
- **Lower holder** for **3 Pins** (here: PDMS, made in a 3D printed mould)







Thermo Scientific Rheology Application Note (in prep.) Influence of fat content on the tribological properties of butter milk products Shewan H.M., Pradal C, Stokes J.R.: Tribology and its growing use toward the study of food oral processing and sensory perception. J. Texture Stud. 2020-51 7–22

Thermo Fisher

Cheese – one rheometer for rheology and texture testing

Comparison milk-based cheese vs. vegan cheese analogue

Serrated plates temperature controlled

- Normal force relaxation (3 N initially)
- CD-oscillation amplitude sweep
- CD-oscillation temperature sweep
- Texture serrated plate

Vane rotor

Cutting/biting test with vane rotor blades

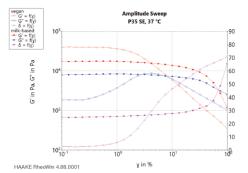


Figure 7: Results from the amplitude sweeps at 37 $^{\circ}\text{C}$ plotted over the deformation γ in %.

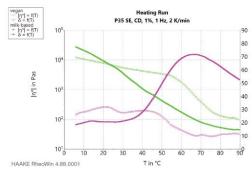


Figure 8: Development of viscosity and phase angle during heating up to 90 $^{\circ}\text{C}\text{.}$

Figure 2: Upper and lower serrated plate (left) and measuring geometry with cheese sample (right).



Figure 3: 4-blade vane rotor used to simulate cutting or biting the product.

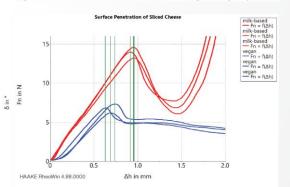


Figure 9: Results of penetration test of both products.

Thermo Scientific Rheology Application Note V300 Rheology and texture of cheese and a vegan cheese analogue



Rheology and texture of cheese and a vegan cheese analogue

Authors

Annika Hodapp and Klaus Oldörp, Thermo Fisher Scientific, Karlsruhe, Germany

Keywords

Cheese, vegan cheese analogue, food analogue, texture analysis, rheology, rheometer

Introduction

What is cheese? Cheese is termented milk with the right taste and the right taxture. While the taste is far beyond its capabilities, a rheometer can definitely help to analyse the texture. This becomes especially helpful when trying to create a vegan substitute for cheese, since the consumer expects a similar look and feel compared to a milk-based cheese.

The texture is an important part of a cheese's properties. Depending on the kind of cheese and its age this can range from creamy to solid. Fiheological measurements can be used to quantify the viscoelastic properties of cheese and cheese formulations. On top of that, a modern rheometer is equipped with a normal force sensor and therefore offers capabilities beyond the classic rheological measurements. In combination with its automatic lift, a rheometer can also be used to run texture analysis tests. In the case of a cheese this could be used to quantify e.g. its bite or its cutting properties.

This report summarises the results of various rheological measurements and a penetration test on a milk-based cheese and a vegan cheese substitute.

Materials and methods

For the measurements described here, a sliced butter cheese and a sliced vegan alternative were used. Both products are commercially available and were purchased at a local grocery shop. The milk-based cheese consisted mainly of protein and fat (25 % and 24 % respectively). The main components of the vegan alternative were fat and starch (20 % and 17 % respectively) while its protein content was below 1 %.



Figure 1: The Thermo Scientific HAAKE MARS iQ Rheometer

The rheological tests were performed using a Thermo Scientific* HAAKE* MARS* IQ Air Rheometer (Figure 1) equipped with a Petiter temperature module for cone-plate- or paralle-plate geometries. To be able to adapt to the thickness of the cheese slices, a parallel-plate-geometry was used. To prevent slippage, serrated plates were used on both sides (Figure 2).

To get a general idea about the properties of both products, amplitude sweeps at 20 °C and 37 °C were performed over a strain range from 0.01 % and 100 % at a frequency of 1 Hz. The measurements at 20 °C were intended to show the sample properties at room temperature, where chesse is cut or a piece is bitten off, for example. At 37 °C the behaviour during chewing or swallowing was investigated.

Conclusion



Rheology, Texture, Tribology ... (possible with one rheometer)

Rheology

Bread spreads, cream cheese, mayonnaise (spreadability)

Vane

Chocolate spreads, mayonnaise (viscosity curve, stress plateau)

Plate/cone

Chocolate melt (ICA standard method 46 International Confectionery Association)
 Concentric cylinder

Fresh fruit juices, tomato sauce with tomato & onion pieces
 Plate/plate (large gap)

Starch (temperature induced transition crystallin/amorphous)

Vane, RheoScope

Texture

Marshmallows (chewing simulation) Plate

Loaf **bread** slices (TPA and firmness)

• Bread roll, apple, margarine (penetration, indenter test) Indenter

Chocolate, Cookies (breaking test, crunchiness)
 Indenter + Lower

Tribology

Butter Milk Products
 Ball-on-3-pins

Rheology & Texture

• Cheese (milk-based and analogue vegan) Serrated plate/plate, vane



Literature



Thermo Scientific Food Rheology Application Notes, White Paper and Compendium

- V136 Measurement and evaluation on liquid chocolate according to the IOCCC Standard
- V187 Rheological behavior of hydrogels dependent on concentration of the polymer and type of neutralizing agent
- V218 Testing instrument accuracy with Newtonian standard fluids
- V222 Breaking strength of chocolate
- V223 HAAKE RheoWin software features for quality control and routine measurements
- V228 Rheooptical Tests during the cooking of starch and the crystallization of fats
- V238 Applied food rheology using fast speed control and axial measurements
- V243 A rheometer with bite marshmallows in the rheometer
- V248 Well prepared good results
- V255 Rheological investigation of **starch** swelling, gelatinization and retrogradation
- Product **stability** or shelf life what rheology has to do with it
- Image acquisition with the **RheoScope** module at very high shear rates (stroboscope, contrast enhancing illumination)
- V268 Spreadability of cream cheese influence of temperature and fat content
- V269 Flow behaviour of **chocolate** melts working according to ICA Standards
- Investigation of the effect of fat content on the yield stress of mayonnaise
- V272 Yield stress of jam, chocolate spread and peanut butter
- V275 How much squeezing power is required to get a paste out of a tube?
- V292 Rheological and **textural** properties of various food formulations analyzed with a modular rheometer setup
- V293 Crunchiness of biscuits **texture** analysis on a rheometer
- V297 Investigating cocoa butter crystallization using simultaneous rheology and Raman spectroscopy (RheoRaman)
- V298 Investigating heat-induced gelation of whey protein using simultaneous rheology and FTIR spectroscopy
- V300 Rheology and texture of cheese and a vegan cheese analogue
- White Paper WP04 An evaluation of meat analog product characteristics by the combination of microscopy and rheological tools.
- Compendium C001.2 Guide to skilled food rheology & extrusion



Thank you

