APPLICATION NOTE

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

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Key words

Rheology, food, viscosity, yield stress, texture analysis, tribology

Introduction

Since foodstuff comes in such a vast variety of structures and textures, the range of rheological methods used to characterize its mechanical properties is even wider.

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 geometries like parallel plates, cone & plate or coaxial cylinders, some other methods try to emulate a certain application by utilizing special rotors and/or measuring fixtures. One such application is studying the texture of food products, which has to match the consumer's expectations. With specially designed probes a rotational rheometer can test various important food properties such as softness, stickiness or spreadability, and can even be utilized for performing tribological tests.

In this application selected rheometer accessories and physical measurements of various food products will be reviewed using a modular rheometer designed for product development and quality control purposes. This includes measuring "classic" rheological properties such as viscosity and yield stress, as well as customized setups for a comprehensive mechanical investigation of food formulations

Materials and methods

Different commercially available food products were examined 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 well as tribological measurements. Shear rate-dependent viscosity was determined by steady-state shear-rate step tests in a range from 0.1 to 100 s^{-1}. Yield stresses were determined by shear stress ramp tests. Different evaluation methods were used to determine distinct values for the yield stress. All viscosity and yield stress tests were performed with a 60 mm parallel plate geometry. In order to avoid sample slippage during the measurement, parallel plates with a serrated (crosshatched) surface profile were used. For the investigation of the tribological profile of different food products, friction coefficients were measured as a function of the circumferential velocity (sliding speed) in a range from 0.001 to 1000 mm/s. The tribological measuring fixture used was based on the ball-on-3 plates principle (Figure 2). Both, the ball as well as the three plates were made of hardened stainless steel. A detailed description about this setup can be found in the corresponding product report [1]. Bending and breaking tests were performed using a 3-point bending tool as shown in Figure 3. With this setup axial ramp tests at a defined set value for the lift speed were performed. The resulting normal forces were recorded and analyzed. Axial squeeze tests were performed with a 35 mm diameter parallel plate setup.

Figure 2: Tribology setup for HAAKE MARS iQ Rheometer based on the ball-on 3-plates principle.

Figure 3: 3-point-bending-tool for performing bending and breaking testswith the HAAKE MARS iQ Rheometer with 8 mm plate rotor.

Results and discussion

Viscosity and yield stress measurements

Liquid and semi-solid food formulations are exposed to a wide range of shear conditions during their entire life-cycle. During storage for instance, when only gravitational forces are present, very low shear rates are applied. During production (mixing, pumping or stirring) and consumption (oral processing) medium-to-high shear rates are observed. Thus, when performing only single point viscosity measurements at one rotational speed, an incomplete picture of the viscous properties is obtained that does not reflect the true nature of the tested material. Only a complete flow curve over a wide shear rate range provides the information required to estimate how a specific food product will behave under different shear conditions. Figure 4 shows the results of steady- state shear rate step tests of three commercially available mayonnaise products.

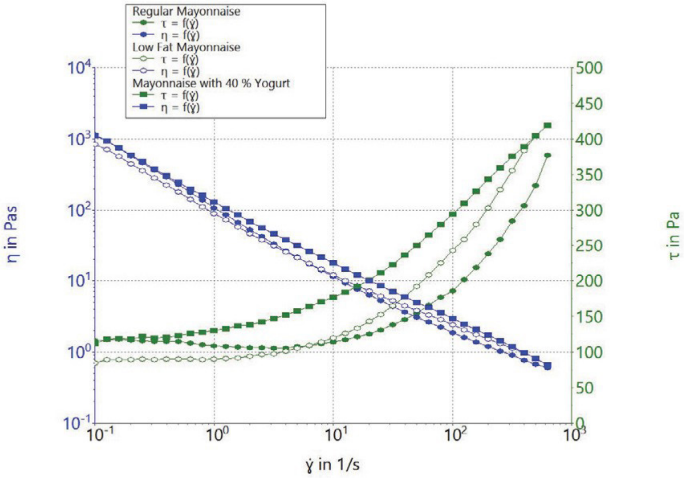


Figure 4: Steady-state-viscosity η (blues symbols) and shear stress τ (green symbols) as a function of shear rate γ for three different mayonnaise products.

As expected for emulsion-based food products, all mayonnaises exhibited significant shear thinning behavior in the investigated shear rate range. Starting at a viscosity of around 1000 Pas at 0.1 s^{-1} all three samples drop to values below 1 Pas at around 800 s^{-1}. At shear rates higher than 800 s-1 sample was ejected out of the geometry leading to a dramatic drop in viscosity as well as in shear stress. That faulty data was removed in Figure 4. Exhibiting such a shear thinning profile is a desired behavior for many semi-solid food products. A high viscosity at low shear rates prevents phase separation of multi component foods and contributes to the overall stability of a product. However, too high of a viscosity at higher shear rates is, in general, less desired. Since it has disadvantages for the application (spreadability, spoonability) and consumption (chewing, swallowing).

Figure 4 also shows that a steady behavior of the shear stress signal at low shear was present for all samples, indicating a yielding behavior. For the regular mayonnaise and the version that contains yogurt, the plateau was occurring almost at identical shear stress values of 120 Pa. The low fat version yielded at a lower shear stress of 90 Pa.

Yield stresses are considered an important parameter in research as well as in quality control to describe the “flow” behavior of many structured fluids and semi-solids. A yield stress can improve the stability of dispersed systems by preventing sedimentation or as an example simply keeping a ketchup from sinking in too much into French fries instead of forming a nice thick layer on top of the fries. Furthermore, yield stresses are connected to certain food properties that are deemed important during oral consumption such as initial firmness [2].

However, the measured value of a yield stress is strongly dependent on sample handling, the chosen rheological measuring method, the data evaluation and even the measuring geometry selected for testing. A common way to investigate the yielding behavior of a sample very precisely is to perform a shear stress ramp test where a linearly increasing shear stress is applied and the deformation or the viscosity is monitored. The results of stress ramp experiments performed with the same mayonnaise samples used for the steady-state shear tests are shown in Figure 5.

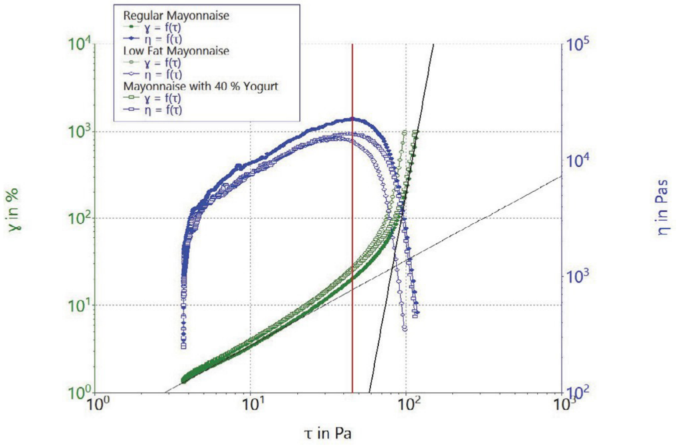


Figure 5: : Deformation γ (green symbols) and viscosity η (blue symbols) as a function of shear stress τ for three different mayonnaise products. The red line indicates the yield stress according to the maximum in viscosity for the regular mayonnaise. The black lines are the tangents applied to the different regions of deformation (elastic deformation and steady flow). The intersection of both tangents represents an alternative method for the yield stress determination.

The deformation stress curves shown in Figure 5 exhibit three distinct regions. In the first region (at low stresses below the yield stress threshold) the samples underwent an elastic deformation. Here the slope of the deformation stress curve is not much larger than 1 in the double logarithmic plot. As the stress increased and approached the yield stress value of the sample, the deformation started to change more rapidly and the slope increased. At higher shear stresses a second linear region with a significantly higher slope was observed. In this region steady flow occurred and the microstructure was altered by these higher shear forces. A common way to calculate the yield stress out of a deformation curve is to apply tangents to the two linear regions. The yield stress then corresponds to the stress value at the intersection of the tangents.
In Figure 5 this method was used to determine the yield stress of the regular mayonnaise sample (black lines). The corresponding yield stress value was at 82 Pa. The tangent method provides a yield stress that is located more in the middle of a transition range between elastic deformation and steady flow. An alternative way to determine the yield stress from a shear stress ramp test is to use the maximum in viscosity as a measure. This method was also applied and is also shown in Figure 5 (red line). The corresponding yield stress value was at 45 Pa and clearly lower than the value derived from the tangents method. It can be seen in Figure 5 that the maximum in viscosity occurred more at the beginning of the transition and deformation just left the region of almost pure elastic deformation behavior.

Table 1 gives an overview of the yield stress values derived from the different evaluation methods. It also includes the shear stress plateau values from the steady-state shear-rate step tests. It can be seen that this value provided the largest value for the apparent yield stress, which corresponds to the fact that the data has been collected at the beginning of a rotational test and therefore the sample is already at the end of its transition between elastic and viscous behavior.

Table 1. Yield stresses of different mayonnaise obtained from different rheological tests and evaluation methods.

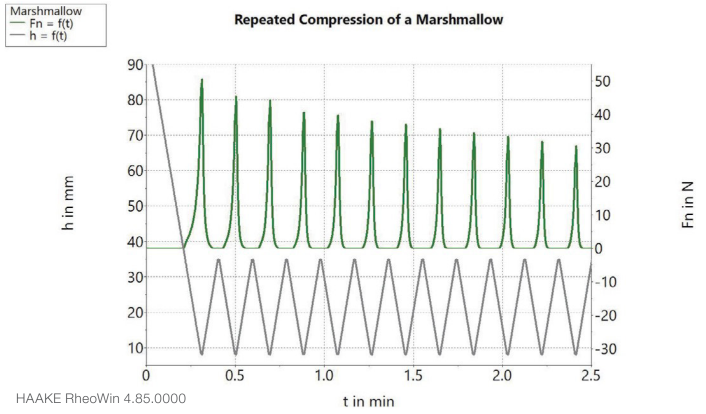
	Test / evaluation method		
Mayonnaise type	Stress ramp maximum viscosity	Stress ramp tangent intersection	Steady-state step-test stress plateau
Regular	45 Pa	82 Pa	116 Pa
Low fat	37 Pa	70 Pa	89 Pa
40% yogurt	45 Pa	83 Pa	119 Pa

In general it can be said that yield stresses of different materials can be compared when the experimental setup and the evaluation method are identical.

Texture analysis

Apart from the classic rheological test methods described above, food formulations are often also characterised regarding their textural properties. A texture analyzer tries to simulate a real-world treatment of a food such as scooping, chewing, spreading or breaking. This is accomplished by moving a measuring geometry onto or into a food formulation with either a defined speed while recording the force necessary to do so or with a defined force recording the resulting deformation. Precise lift movement and sensitive axial force control are inherent capabilities of a modern rheometer. Consequently, it is rather easy to use a rheometer for texture analysis. In cases, where the normal measuring geometries like cones, plates or cylinders are not suitable for such a texture test, almost any kind of special measuring geometry can be adapted using a universal adapter fixture.

Marshmallows are a typical example for sweets, where the desired mouthfeel during chewing is an important part of their success. To simulate the chewing behaviour, marshmallows were placed onto the lower plate of the rheometer's measuring geometry and a 35 mm plate was used to squeeze them down to 8 mm height with a speed of 5 mm/s. Then the top plate went up again with the same speed and afterwards the squeezing step was repeated several times to simulate chewing. The results of this testing are shown in Figure 6.



HAAKE RheoWin 4.85.0000

Figure 6: 12 cycles of compression and relaxation of a marshmallow. The black Curve shows the down and up movement of the upper geometry. The green curve shows the corresponding changes in the force needed to compress the sample.

The maximum force at each compression step went down from cycle to cycle indicating that the marshmallow became softer simply due to repeated compression without any influence of liquid (saliva).

The breaking behaviour is another important property for certain types of food like biscuits or chocolate. In the latter case it needs again to fulfill the consumer's expectation. For example a milk chocolate is expected to be softer whereas a dark chocolate is expected to be harder, maybe even brittle. Regarding the texture of biscuits, it gets a bit more complex since starch-based products usually change their texture over time due to the influence of air humidity. So, apart from their initial properties the aging of biscuits is also a topic for investigation where usually the force needed to break the biscuit, and the amount of bending before the biscuit breaks, were evaluated.

To test the breaking behaviour, biscuits were placed onto the 3-point-bending-tool (Figure 3). A plate rotor with a dia- meter of 8 mm was selected as the upper part of the measuring geometry. The starting position of the upper geometry was chosen sufficiently high enough to allow the convenient positioning of the biscuits. The upper geometry moved downwards with 0.1 mm/min to detect the surface of the biscuit with a sensing force of 0.1 N. From that point on, the upper geometry continued downwards with 1 mm/min, bending and breaking the biscuit.

Afterwards, using the loop function in the Thermo Scientific™ HAAKE™ RheoWin™ Software, the measuring geometry was lifted up again to make way for positioning the next biscuit. The results of multiple tests on fresh biscuits, tested immediately after opening the package, showed some scattering in maximum bending and breaking force as expected when testing samples based on natural raw materials (Figure 7).

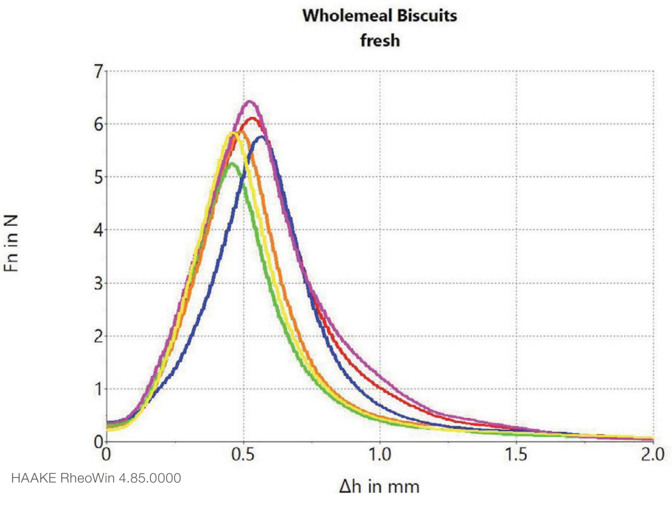


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Figure 7: Multiple breaking tests with fresh wholemeal biscuits.

The same tests were repeated 14 days after the biscuit package was opened (Figure 8). The maximum of the force curves became broader and the amount of bending slightly increased compared to the fresh biscuits, indicating that slight aging had occurred over this time period.

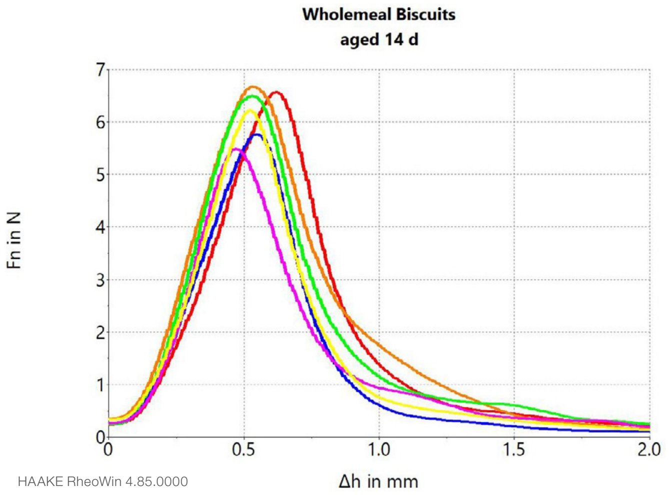

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Figure 8: Multiple breaking tests with aged wholemeal biscuits.

Tribological tests

Tribology is a field of materials science and mechanical engineering that deals with the properties of interacting surfaces in relative motion. It includes the study and application of the principles of friction, lubrication and wear. Tribological measurements have raised interest in the field of food science as an additional technique to describe the complex concept of texture and mouthfeel.

Surfaces of different materials and roughness have been used to simulate the complex interaction of tongue, food (saliva) and palate during oral processing [Ref 2]. The ultimate goal is to correlate tribological parameters, such as the friction coefficient, with textural mouthfeel properties (e.g. creaminess or fattiness) usually derived from sensory panels.

Tribological data is commonly presented in the form of a Stribeck curve where the friction coefficient is displayed as a function of the sliding speed. The general form of a Stribeck curve is presented in Figure 9 and can be divided in three regions. At low sliding speeds where no lubricating film is present, the behavior is dominated by direct solid/solid contact. This part is commonly referred to as the boundary lubrication range and the resulting coefficients of friction are high. At medium sliding speeds the increasing hydrodynamic pressure of the lubricating sample is causing the development of a lubricating film between the two surfaces and the coefficient of friction will start to drop. At high sliding speeds the lubricating film has separated the two surfaces completely and no solid/solid interactions are present anymore. In this hydrodynamic lubrication range the coefficient of friction will start to increase again. In general one can say that the lower the coefficient of friction, the better the lubricating properties of a liquid/ semi-solid surface system. Figure 10 shows the comparison of Stribeck curves obtained from tribological tests with two different chocolate spreads and an olive oil on a ball-on-3-plates setup.

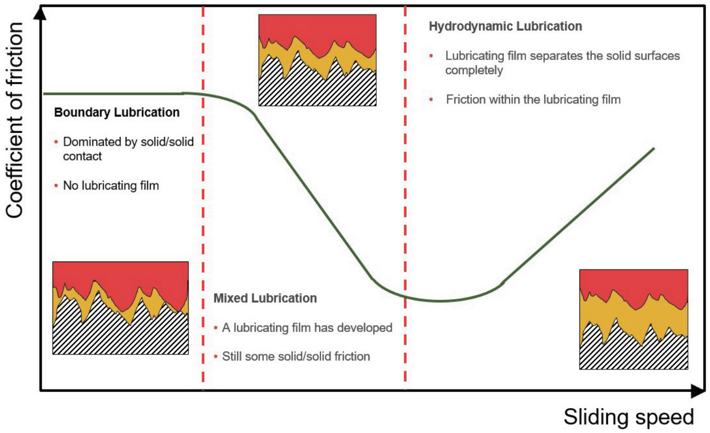

Figure 9: General shape and regions of a Stribeck curve for tribology measurements.

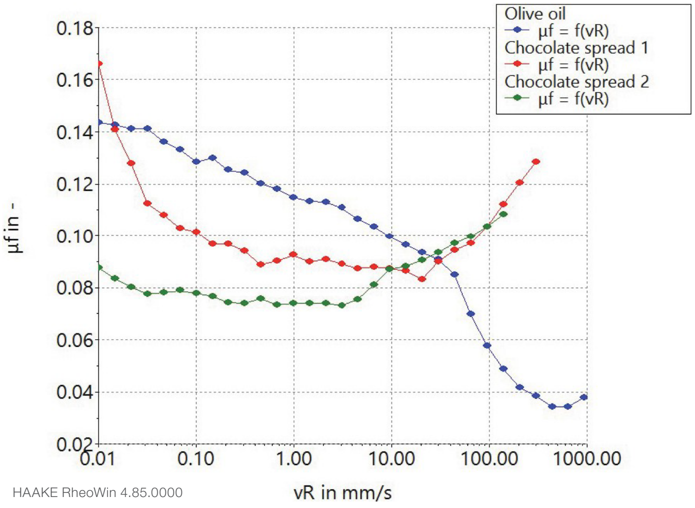

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Figure 10: Stribeck curves (friction coefficient µf as function of sliding speed vR) of two different chocolate spreads and an olive oil.

The graph reveals the different lubrication properties of the olive oil compared to the two chocolate spreads. Due to their higher viscosity and more paste-like structure, the chocolate spreads formed a more stable lubricating film at lower speeds. At higher sliding speeds, the lower viscosity of the olive oil had a clear advantage, and it showed the best lubrication properties with the lowest friction coefficients in that range. In addition, the Stribeck curves revealed differences between the two spreads. While at higher sliding speeds the friction coefficients were almost identical, chocolate spread 2 showed better lubrication properties at low and medium sliding speeds.

Conclusions

The HAAKE MARS iQ Rheometer with mechanical bearing is a versatile and modular instrument ideal for investigating the mechanical properties of liquid and semi-solid food formulations. Due to a large number of measuring geometries and other accessories, it is capable of performing standard rheological tests to measure shear-rate-dependent viscosity over a wide shear range and yield stresses in stress-controlled measuring mode as well as more comprehensive texture analysis and tribological tests. While viscosity and yield stress are important parameters for predicting the behavior of food products during processing, transport and storage, texture and tribological properties allow for a more comprehensive study of oral processing and the general concept of mouthfeeling. The HAAKE MARS iQ Rheometer allows food scientists in R&D to characterize all stages of a product's life cycle from its raw materials to its consumption. Rheological tests developed by R&D can then be easily transferred over to quality control departments using the same HAAKE MARS iQ Rheometer for batch release testing.

References

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