Axial Measurements and Fast Speed Control Used for Food Characterization

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ABSTRACT

The characterization of food requires more information than just the usual viscoelastic properties accessible with any standard rheometer. This article shows some classical rheological data and some results, which can now be collected with the new capabilities of a modern rheometer.

INTRODUCTION

Food comes in a huge number of varieties and the number of rheological methods used to characterize it is even bigger. For example it can be a simple test to check the viscosity of an oil or a molten chocolate or it can become quite difficult when quantifying the texture of a peanut butter.

Some tests rely on classical rheological terms like viscosity or yield stress. Others try to emulate an application by using a special measuring geometry or try to save time by measuring directly in the original container coming from the production.

In this article some special capabilities of the HAAKE MARS are shown. For example, very small and extremely fast controlled deformations have been used to test a food's undamaged structure. The normal force sensor has been used in combination with the lift to expand the range of information accessible with a rheometer.

SMALL DEFORMATIONS

Bread spreads are interesting materials regarding their rheological behaviour. How a bread spread looks like when a fresh container is opened, how easily it can be spread and whether it looks appetizing on a slice of bread is related to its solid-like behaviour at rest and the force necessary to overcome it. Thus the yield stress is an important parameter to characterize the "look and feel" of a bread spread.

When a sample is put into a rheometer the weak part of its structure can already be destroyed by handling or squeezing it. Thus some authors distinguish between the static yield stress, measured on the "undamaged" sample, and the dynamic yield stress, measured after the loss of the weak structure[1].

Therefore and to save time it is often preferred to measure the static yield stress in the original container. Equipped with a special flexible container holder and e.g. a star-shaped vane rotor, the HAAKE MARS can measure in a variety of containers. Due to its unique spacious design it allows measurements in containers up to 10 l buckets (Figure 1).

When measuring with a vane rotor in the original container, the yield stress is measured by applying a constant shear rate to the sample and by determining the initial maximum of the shear stress. To get a yield stress independent of the shear rate applied, the shear rate has to be as low as possible[1]. At the same time, the shear rate has to be constant before the weak structure breaks to get reliable and reproducible results. To combine these two requirements is a very demanding task for a rheometer since it takes the longer to get a constant shear rate the lower the shear rate is.

Figure 1. The HAAKE MARS measuring in a 10 l bucket.

A peanut butter and a chocolate spread were tested with such a low shear rate of 0.001 1/s. Even against the high resistance of the bread spreads the control loop managed to stabilize the shear rate in less than 1 s leading to the perfect reproducibility shown in Figure 2.

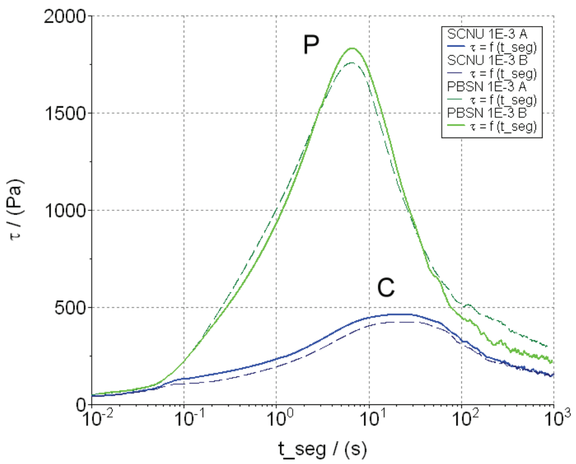

Figure 2. Yield Stress of a peanut butter (P) and a chocolate spread (C) measured with a vane rotor in their original glasses using a shear rate of 0.001 1/s.

FAST SPEED CONTROL

The shear viscosity is probably still the most commonly know rheological property of food products. Its importance can be seen from the huge variety of different thickeners available to stabilize food or to make it appeal to the customer's expectations.

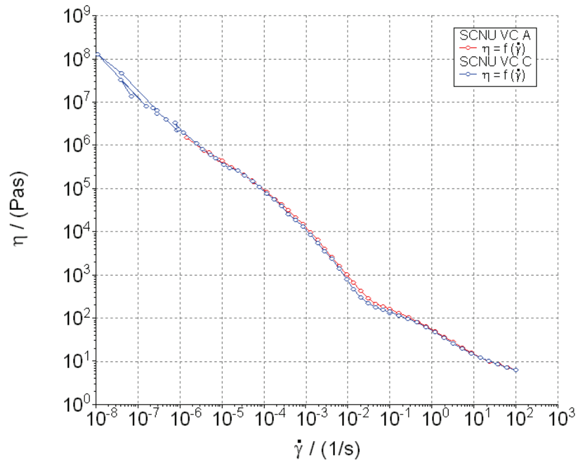

Figure 3. Viscosity curve of a chocolate spread. 2 measurements in one run over 10 decades of shear rate in CR mode with a cone 35 mm / 1 ° at 35 °C.

To completely describe a food's behaviour during e.g. storage, pumping or chewing, its viscosity has to be measured over a wide range of shear rate. In the industry especially the lower shear rates are often ignored simply due to the long time it can take to get stable viscosity data.

Following the industries demands, a new control loop has been developed to significantly shorten the time to get stable data in CR mode. Figure 3 shows the viscosity curve of a chocolate spread measured with a cone plate geometry at 35 °C in one run. In less than 20 min the viscosity has been measured over 10 decades in shear rate.

The combination of the wide dynamic range of the HAAKE MARS and its new CR control loop offer new possibilities for fast and reliable measurements.

AXIAL MEASUREMENTS

The texture of a food determines whether the consumer likes for example its touch or its mouth feeling. For a solid food like chocolate even the force needed to break it and the sound when it breaks add to the impression of good quality, provided they are in the right range.

Apart from test panels where food is tested with the human senses, so-called texture analyzers are used to measure impartial texture-related parameters. These instruments mainly consist of a lift, which drives a probe onto or into the food's surface and a force transducer, which measures the force required for bending, breaking, or penetration.

A modern rheometer like the HAAKE MARS is equipped with a precise lift and an extremely sensitive normal force transducer, a configuration suggesting itself for texture analysis. The owner of the rheometer gains the capability to measure data comparable with the data used by e.g. suppliers or customers using only a texture analyzer.

Bending and Breaking

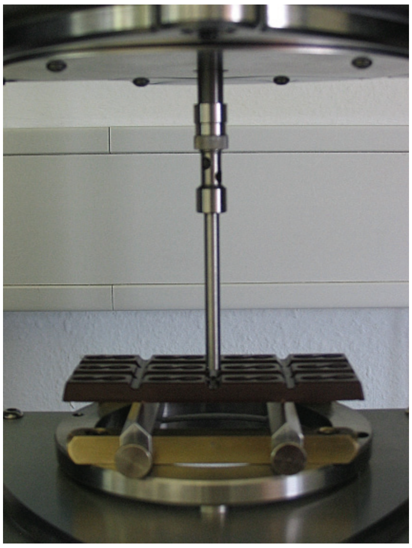

Figure 4. Measuring the breaking resistance of chocolate with the HAAKE MARS using a two-blade-fixture and a 6 mm piston.

For bending and breaking tests, a special sample holder has been developed based on the design of a three-point-bending fixture. The chocolate sample is placed onto 2 blades with adjustable distance. A round piston is mounted to the measuring head using an adapter (Figure 4). During the test the piston drives downwards with a constant speed until the chocolate breaks.

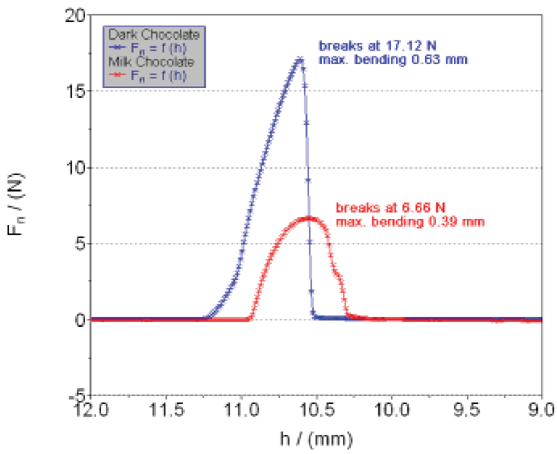

Figure 5. Axial force as a function of piston position for dark chocolate (higher peak) and milk chocolate (smaller peak).

The evaluation of the data makes it possible to quantify the breaking behaviour by the maximum force needed and the slope of the curve when the force returns to zero. As an example, the breaking curves for dark chocolate and milk chocolate are shown in Figure 5. Piston speed was 1.3 mm/min.

Here the dark chocolate breaks sharply when the necessary force has been reached, while the milk chocolate slowly breaks in 2 steps. The typical hard texture of the dark chocolate and the soft creamy texture of a milk chocolate can easily be identified with the test performed.

Penetration

Another common method to determine the spreadability of a bread spread is the determination of its firmness with a penetration test. Different methods and different probes are used.

For tests on margarine the 6 mm piston has been used again. The margarine has been stored in the fridge until just before the measurement and is then placed onto the universal container holder. First the rheometer lowers the probe until the sensitive normal force sensors detects the contact with the margarine's surface. Then the piston is driven some millimetres into the sample with a constant speed. This final position is then held and the relaxation, i.e. the decaying normal force, is measured.

The test has been repeated several times on different spots of the same block of margarine. After every second measurement the sample was put in the fridge again for 5 min.

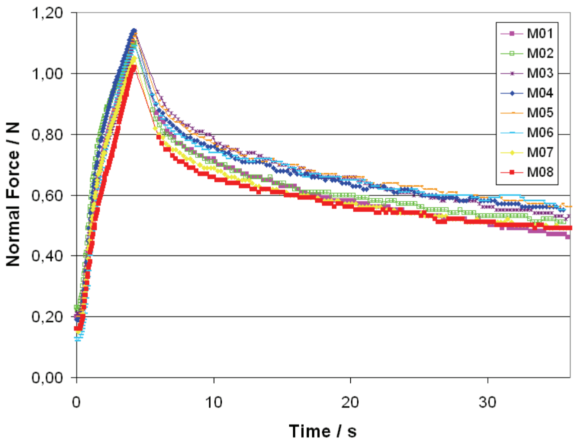

Figure 6. Penetration test on margarine repeated 8 times. First the probe moves into the sample, then the relaxation of the normal force is measured

The curves presented in Figure 6 are the results of 8 measurements done on the same block of margarine and show the good repeatability of this method. For data evaluation the maximum force at the end of the movement and the residual force at the end of the relaxation can be used.

SUMMARY

With its extremely fast CR control loop, the HAAKE MARS is able to stabilize the shear rate extremely fast. This new capability enables the user to measure the strength of weak structures with such a rheometer in a reproducible way before they are destroyed by the rotation.

The same capability satisfies the need of QC for fast measurements covering the wide shear rate range from storage to application within an acceptable timeframe.

A modern high-end rheometer with a very sensitive normal force sensor and a precise lift control can be used like a texture analyzer or a penetrometer, which are commonly used in the food industry. It therefore expands the variety of information accessible using only one instrument.

ACKNOWLEDGMENTS

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2. Oldörp, K. (2007), "What Happens When Rheological Properties Change?", Annual Transactions of the Nordic Rheology Society, Vol. 15, pp. 121-126.