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APPLICATION NOTE

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Yield stress determination of food spreads using the vane rotor method

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Abstract
A standard task in the Quality Control (QC) of typical food spreads – like jam, chocolate spread or peanut butter – is the determination of the yield stress in the original container. For this purpose a vane rotor needs to be moved into the intact product structure in a perfectly vertical movement. Efficient and high-throughput QC measurements require a rheometer with an easy-to-operate lift function and a quickly adaptable universal container holder for different container designs as well as software routines for automated measurement, evaluation and QC documentation. Furthermore, an optionally available temperature sensor mounted parallel to the rotor allows for recording the sample temperature during the measurment without damaging its structure.

The yield stress of a semi-solid food product is a measure of important material characteristics such as stability, mouthfeel, pourability, spreadability and processability and is affected by the food ingredients and their formulation [1 - 3].

Figure 1: The HAAKE Viscotester iQ Rheometer with mounted universal container holder and vane rotor FL26-2 blades (left); universal container holder with three vane rotors: 4-blade rotors FL16 and FL22 as well as FL26-2 blades (right).

In order to investigate the effect of particles and different formulations on the yield stress as well as to study the reproducibility of the method, three typical food spreads were tested.

Introduction
An advantage of vane rotors is that they can be used for testing materials with larger particles. The size of the vane blades needs to be several times bigger than the maximum particle size (e.g. the seeds in a raspberry jam). From a rheological point of view, the solid particles act as passive filler (like glass beads) and therefore do not contribute to the elastic network caused by weak interaction of molecules or larger aggregates. Below the yield point, a sample is showing a linear response to an applied shear stress or deformation. Around the yield point, the applied stresses

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become large enough to alter the microstructure of the material and cause a non-linear viscoelastic response. Above its yield point, a material behaves like a liquid. The results obtained from yield stress determination strongly depend on the rheological method and experimental settings used [1 - 3].

Chocolate spreads show differences in sugar, fat, cacao and protein content as well as the type of emulsifier. This may have a considerable effect on the yield stress. Two different commercially available chocolate spreads were selected for this investigation.

The yield stress of peanut butter is even higher than that of chocolate spread [4]. Therefore, creamy peanut butter was selected to check the reproducibility of the vane rotor measuring curves and yield stress evaluation.

Yield stress is by definition the minimum shear stress required to make a material flow. The yield stress is a measure for pourability, spreadability and spoonability and is also used to predict product stability [2 - 5]. The measured yield stress values τ_0 depend on the one hand on the spread's ingredients and on the other hand on the rheological method and experimental parameters used for yield stress determination. Moreover, the pre-experimental sample handling plays an essential role and determines whether an intact or a disturbed structure is measured [2, 3, 5].

Regarding rheological measuring methods, the most accurate and recommended method to determine absolute yield stress values is the Controlled Stress (CS) ramp with parallel plate measuring geometry. This requires careful sample preparation, handling and loading in order to maintain the intact structure of the material [6]. Extensive stirring or squeezing would lead from the static yield stress of the intact structure to the (lower) dynamic yield stress of a disturbed structure [1 - 3]. Loading a sample properly into a parallel plate, cone and plate (or concentric cylinder) measuring geometry with subsequent equilibration and CS ramp yield stress measurement takes about 10 (or 20) minutes.

For QC, this may be too time-consuming – therefore, relative vane rotor measurements with an intact sample structure in the original container are often preferred, since they can be conducted much faster and are related to the static yield stress [2, 3, 5]. The correct selection of the experimental parameters for vane rotor measurements is fundamental – this is discussed in more detail below and in [7]. In general, Controlled Rate (CR) mode with rotational speeds lower than 1 rpm is recommended [2].

Material and methods

A Thermo Scientific™ HAAKE™ Viscotester™ iQ Rheometer equipped with a 4-blade vane rotor FL16 (vane diameter 16 mm, height 8.8 mm) or FL22 (vane diameter 22 mm, height 16mm) and a universal container holder (Figure 1) was used for the yield stress determination in CR mode. In this investigation, the rotational speed was set to 0.05 rpm for all measurements.

Five commercially available food spreads were investigated with the same method using different vane sizes (see below). The sealed jar was opened and fixed in the universal container holder. Using the manual lift function of the HAAKE Viscotester iQ and the features of the Thermo Scientific™ HAAKE™ RheoWin™ Software (Figure 2), the vane rotor was prevented from rotating (element 1: CR mode $\gamma = 0\ s^{-1}$) and lowered vertically into a well-reproducible position as well as penetration depth (according to the dimensions and shape of the container type; element 2).

Practical experience shows that stiffer products, which are filled into a container in an industrial process line at higher speed, can show different material properties at different regions within the container. In such a case it is mandatory to make the vane measurement always in the same position in each particular container design in order to obtain comparable and reproducible results.

After a short equilibration and recovery time (element 3), the total time t was set to zero (element 4) right before the measurement was started. A low rotational speed (here: 0.05 rpm) was applied and a set number of data points was recorded within the set time (element 5). With an automated evaluation and QC element (element 6: curve discussion), the maximum in the shear stress τ vs. time t plot was determined automatically. The HAAKE RheoWin software provides the option to verify, whether the obtained value for τ_0 falls within a user-defiend tolerance range (pass/fail criteria) [9]. Finally, a report was generated (element 7). It can either be saved in different file formats (e.g. in pdf, jpeg, tiff or emf format) or sent directly to a printer.

For each sample class, the most suitable rotor type and rotational speed need to be determined in a preliminary test. Smaller vane rotors are used for samples with stronger texture and higher yield stress, like peanut butter, while larger vane rotors are more suitable for samples with lower viscosity and lower yield stress, like chocolate spread, jam or mayonnaise [7].

In order to determine one rotational speed, which fits all samples of a class, different rotational speeds need to be tested. A too high rotational speed leads to a sharp peak which cannot be evaluated (red triangles in Figure 3). A too low rotational speed delivers an asymptotic curve with no maximum (green circles).

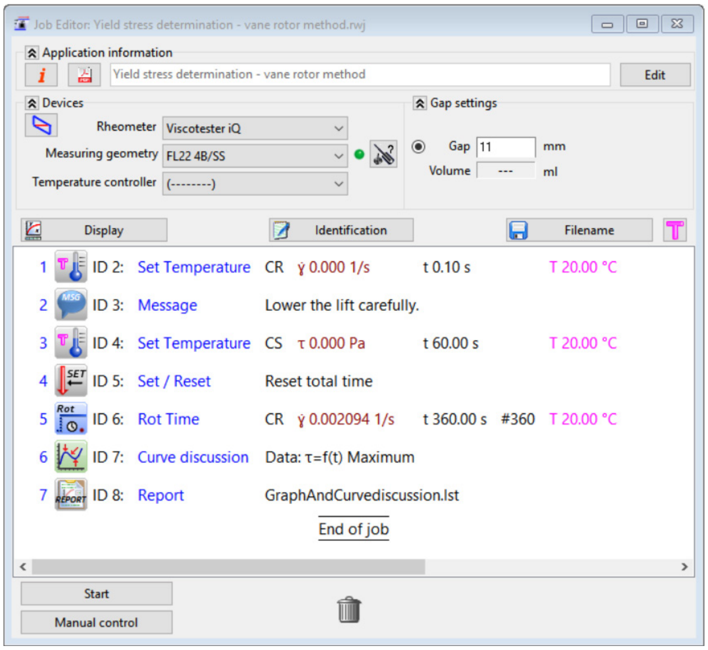

Figure 2: HAAKE RheoWin measuring job for measurement and automated evaluation and documentation.

The goal is to select a rotational speed, which generates a curve with evaluable maximum (blue rectangles). The speed corresponding to the highest evaluable maximum is the best choice for this particular sample [7]. For this investigation, a rotational speed of 0.05 rpm turned out to be a suitable set value.

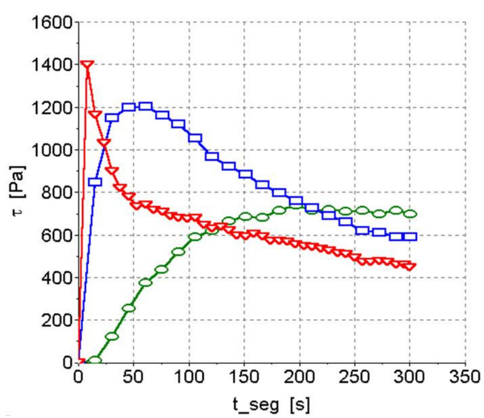

Figure 3: Schematic comparison of vane rotor yield stress measurements with higher (red triangles), medium (blue rectangles) and lower rotational speed (green circles).

Results and discussion

The effect of the composition on the yield stress was subject of several investigations in the past. Different behaviors were observed according to the nature and number of ingredients of the formulation used for the studies [1 - 3].

The rheological characteristics of jams depend strongly on fruit type and jam formulation [8]. Figure 4 shows the vane rotor yield stress measuring curves for two raspberry jams – one with seeds, the other sieved. As expected, the yield stress of the sieved jam is much higher than the yield stress of the jam with seeds, because the seeds behave like hard spheres and do not contribute to the stress bearing elastic structure. On the other hand, the time values for both jams are similar (Tab. 1).

As an example, the reproducibility of the measuring results was checked with the sieved jam – see last line in Table 1. Compared to the average value, the yield stress values differ only by ± 0.5 % and the time value is identical.

After a 4-blade rotor has turned by 85° to 90°, no more intact sample is sensed by the vane rotor anymore. Therefore, the measuring curves exhibited a slight decrease at around 300 s.

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

Raspberry jam	τ_0 in Pa	t in s
With seeds	590	100
Sieved	967	104
Sieved (measurement 2)	977	104

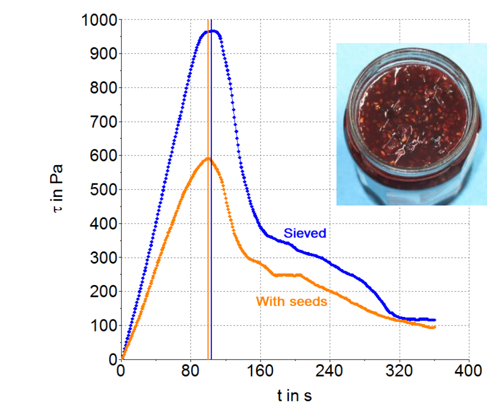

Figure 4: Comparison of FL 22 vane rotor yield stress measurements at 0.05 rpm for a sieved raspberry jam (blue curve) and raspberry jam with seeds (orange curve, inserted image).

Figure 5 and Table 2 show the results of the yield stress measurements for the tested chocolate spread products A and B. Product A contains more cacao, more protein (7 %) and more sugar (56 %) than product B (6 % protein; 50 % sugar). On the other hand, the fat content of product A (32 %) is lower than in product B (35 %). Furthermore, different emulsifiers were used – soy-based (A) or from sunflower (B). The yield stress value of product B is more than twice as high as of product A.

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Table 2: Comparison of FL 22 vane rotor yield stress measurements at 0.05 rpm for two chocolate hazelnut spread products.

Chocolate spread	τ_0 in Pa	t in s
A	364	117
B	722	73

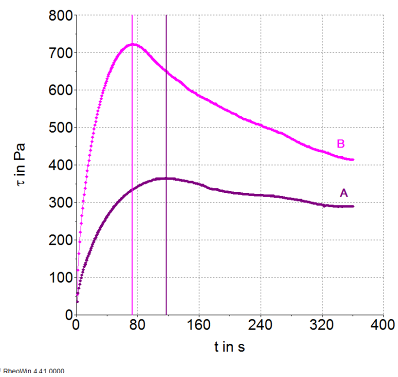

Figure 5: FL 22 vane rotor yield stress measurements at 0.05 rpm for chocolate hazelnut spread products A and B.

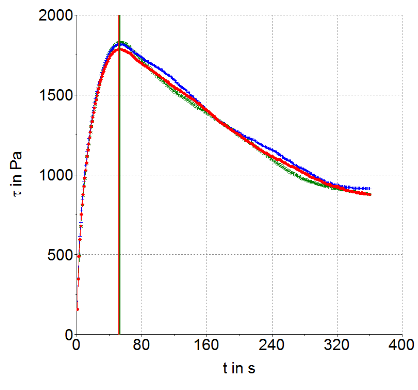

Figure 6: Three independently conducted vane rotor (FL16) yield stress measurements in CR mode with 0.05 rpm for creamy peanut butter.

Table 3: Three independently conducted vane rotor (FL16) yield stress measurements in CR mode at 0.05 rpm for creamy peanut butter.

Peanut butter	τ_0 in Pa	t in s
Measurement 1 (blue)	1818	52.1
Measurement 2 (red)	1785	52.1
Measurement 3 (green)	1829	53.2

The reproducibility of CR vane rotor yield stress determination was tested with creamy peanut butter. Three independently conducted measurements with a FL16 rotor in CR mode with 0.05 rpm show excellent correlation – see Figure 6 and Table 3. Compared to the average value, the yield stress values differ by less than ± 1.5 % and the time values are nearly identical.

Conclusions

The HAAKE Viscotester iQ Rheometer equipped with the universal container holder and a vane rotor allows for efficient, high-throughput measuring routines yield stress testing of food spreads in an QC enviroment.

The instrument's easy to operate lift function ensures convenient and fast handling. In combination with the universal container holder, it allows for a well-controlled and perfectly vertical positioning of the vane rotor in the particular container type – a key to reproducible results.

The HAAKE Viscotester iQ Rheometer can be operated either as a standalone unit with pre-defined or customized measurement and evaluation routines or with the HAAKE RheoWin measurement and evaluation software. Its evaluation functions offer fully automated QC routines including pass/fail criteria and various options for the documentation of the measuring results. [9].

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