

Yield Point Determination-a Critical Discussion of Different Methods

Eva-Maria Kutschmann, Gebrüder HAAKE GmbH, Dieselstrasse 4, D-76227 Karlsruhe

ABSTRACT

In many practical applications the yield point of a product is of interest. Methods and possibilities on different instruments are discussed and compared presenting practical examples of food and cosmetic products.

INTRODUCTION

In a simple definition the yield point is the minimum stress that is required to make a material flow. If it exists at all, it is still in the discussion stage. Some people argue that it is only a question of observation time. If you wait millions of years you can even see mountains flow. But this point of view is not relevant for our daily work, thus we are going to discuss acceptable time scales when we talk about yield values¹.

In general, cosmetic and pharmaceutical formulations, such as O/W or W/O-emulsions, show plastic flow behaviour. In order to characterize the product quality the yield point is used as an important criterium. New product formulations are developed with respect to the final use of the product, to its typical application and the consumer's expectations².

In the food industry the yield value is very important for a product's stability. For example, a salad dressing should not show phase separation or sedimentation of solid ingredients such as herbs and spices³.

The engineers responsible for the processing technologies must know the rheological behaviour of the products. Depending on the yield stress, different filling and packing machines are used, the final products are sold in bottles, tins, boxes, tubes or other special containers.

Last but not least there is nowadays an increasing interest in being able to empty containers completely before the packing material is recycled. Here the yield value is

important information for the recycling company.

TYPICAL YIELD VALUES

The following table shows some typical yield values for well known every day products:

Substance	Yield Point
Body Lotion	2-10 Pa
Skin Creme	20-50 Pa
Ketchup	15-20 Pa
Mayonnaise	20-30 Pa

Table 1: Typical Yield Values

TRADITIONAL METHODS

Traditionally, in many industries the yield value was extrapolated from controlled rate flow curves or calculated using regression models such as Casson or Hershel-Bulkley.

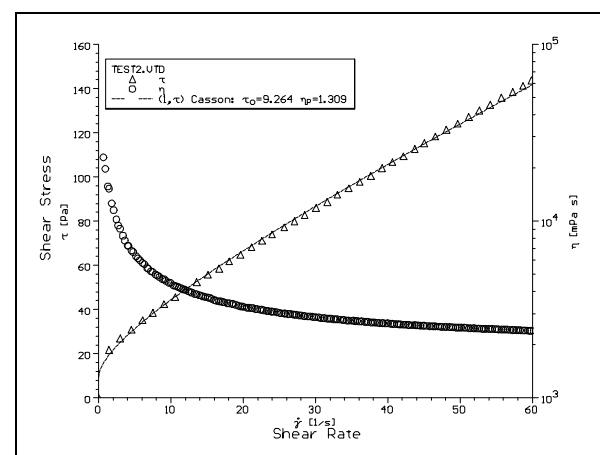


Fig. 1: Flow and Viscosity Curve of chocolate at 40°C

Unfortunately, these results depend strongly on the experimental conditions and the shear rate range used in the regression model. Comparable results are obtained if a well defined and general accepted procedure exists.

One of these worldwide accepted procedures is used in the chocolate industry when testing liquid chocolates at 40°C according to the OICCC recommendations^{3,4}.

CONTROLLED DEFORMATION

A simple but accurate method for yield point determination is the CD-test (controlled deformation) which can be carried out using the HAAKE Viscotester VT 550. By using special star shaped rotors the sample's structure is not destroyed before the test. In the controlled deformation mode the deformation is stepwise increased until the structure breaks down. The yield stress value is shown on the display. When the instrument is connected to a computer the measured stress is plotted as a function of time. The VT550 application software allows a calculation of the maximum stress that corresponds to the yield stress. This method is a quick but accurate possibility for determination of yield values in quality control.

STRESS RAMPS

As the CD test is not sensitive enough for low yield stresses (e.g. below 10 Pa), the preferred method is a stress ramp performed on a controlled stress rheometer. There are different opinions on how to evaluate the test results. Some people plot the viscosity as a function of stress and calculate the yield value from the maximum viscosity. Another possibility is plotting deformation γ versus stress τ in a double logarithmic plot. The yield stress can be clearly detected at a pronounced breakpoint in the slope of two power law regressions. The application software allows the exact calculation of the intercept of the two curve fits. This method gives good reproducibility and accurate results even for low yield values.

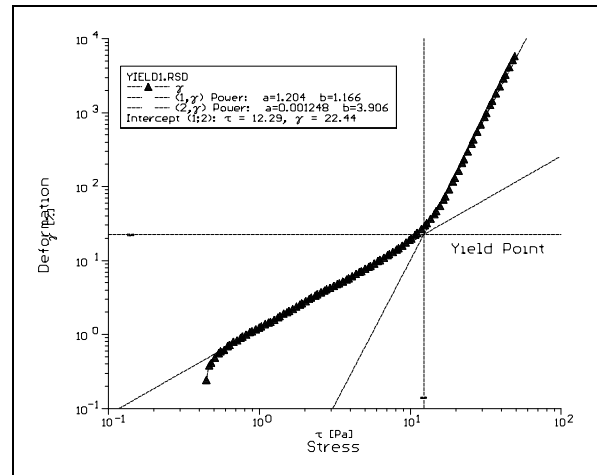


Fig. 2: Yield Point determination from a stress ramp

OSCILLATION STRESS SWEEP

Very often dynamic tests are run in order to characterize the products viscoelastic behaviour. From oscillation stress sweeps we derive in general the linear viscoelastic range of the product. But these tests give us some idea on the yield stress, too. At stresses below the critical stress, the sample material behaves like a viscoelastic solid. At higher stresses, the material starts to flow as its yield value is exceeded. The structure of the sample is then irreversibly destroyed. When testing two different products under the same conditions we can expect the following: Samples showing higher critical stresses in an oscillation stress sweep are expected to possess a higher yield value. Figure 3 shows the results from a stress sweep performed on two toothpastes. Product A (open symbols) has a lower critical stress than product B (filled symbols). As expected, the yield value of toothpaste A is lower. This corresponds to the observation that product A is easier to press out of the container, product B seems to have a stronger gel structure. Please note that in a stress sweep the material is tested only at one frequency (e.g. at 1 Hz), so stress sweep results might be slightly different from static tests.

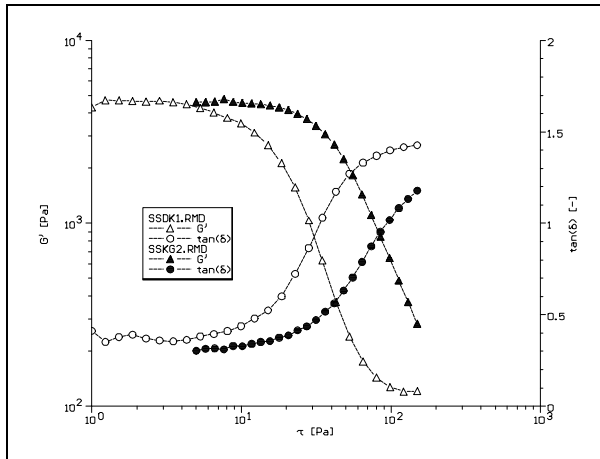


Fig. 3: Oscillation Stress Sweep on 2 different toothpastes

CREEP TESTS

The most accurate but unfortunately a very time consuming method for yield point determination is performing a series of creep tests. When testing a fresh sample under same conditions but applying different stresses, all compliance curves are identical as long as the yield stress is not exceeded^{5,6}.

SUMMARY

Today a general accepted definition of the yield point does not exist. As a consequence many people feel confused about the different possibilities on how to determine the yield point of a product. In this paper methods and their application, advantages and disadvantages are discussed. As mentioned before, in all methods the test results depend on the sample's history and the test conditions (pretreatment, sample loading etc.). It is in the responsibility of the operator to find out the optimum conditions for his special application. Modern instruments, new techniques and software help to find the best solution for reliable results.

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Gehr. HAAKE GmbH, Tel. +49 721 4094-444
Dieselstr. 4, D-76227 Karlsruhe, Germany