How much squeezing power is required to get the toothpaste out of the tube? Yield stress determination using the HAAKE Viscotester iQ.

Cornelia Küchenmeister and Jan Philip Plog, Thermo Fisher Scientific, Material Characterization, Karlsruhe, Germany

Key words
Rheology, Toothpaste, Yield Stress, Customer Expectation

Introduction

Toothpastes are viscoelastic products with a paste-like texture well know in everyday's life. They have a complex structure including many components influencing the rheological properties such as abrasives to support the mechanical cleaning effort of the toothbrush, moisture agents to prevent a drying out of the toothpaste as well as binding agents to prevent the phase separation between the liquid and the solid. Rheology plays an important role in the product development of toothpaste, the production, the filling and packaging processes as well as in fulfilling the expectation of a consumer target group [1].

This article will focus on the importance of the yield stress of different toothpastes. The yield stress characterizes the amount of force that it takes to break down the initial structure of the product and get the fluid moving. Squeezing is required to get the toothpaste out of the tube. There must be enough squeezing power to overcome the yield stress in the material. Two different toothpastes – one for adults and one for children – have been selected for this investigation. For the measurement and evaluation the preferred method described in [2] was applied.

Measuring Setup

For the measurements the Thermo Scientific™ HAAKE™ Viscotester™ iQ rheometer was used. This instrument equipped with an EC motor enables a variety of test methods in controlled rate (CR) and controlled stress (CS) mode in a broad measuring range. As temperature control unit a self-contained Peltier module was used, which enables a fast and reliable temperature control in the range between -5 °C and 160 °C. The tests were carried out by using a parallel plate measuring geometry with a diameter of 35 mm. To avoid slippage a measuring geometry with serrated surface has been selected. The lower measuring plate matches the upper plate in diameter and appearance and therefore ensures ideal measuring conditions and an optimal sample filling [3]. Other advantages of this geometry are a small sample volume, shorter time for temperature equilibrium and reduced cleaning effort. In addition a sample cover was put over the filled measuring geometry to minimize the evaporation and the temperature gradient within the sample.

Fig. 1: Thermo Scientific HAAKE Viscotester iQ

Samples and Preparation

One toothpaste for adults and one for children have been selected for the test described in the following report. At the beginning of the test a small amount of the sample was squeezed out of the tube and placed in the middle of the lower exchangeable plate (Fig. 1). The upper plate was moved down to the measuring gap. The squeezing and gap filling procedure corresponds to a pre-shearing of the sample and a compromise has to be found for the waiting time which the sample needs to relax from the applied stress and other effects which influence the rheological behavior of the sample such as evaporation.

HAAKE RheoWin software and alternative operation

There are different options to operate the HAAKE Viscotester iQ.

On one hand the fully software controlled operation using the Thermo Scientific™ HAAKE™ RheoWin™ software enables the maximum on flexibility regarding the definition of the measuring and evaluation routine. The following

Thermo SCIENTIFIC

test procedure has been used for the investigation: after a waiting time of 5 minutes for temperature control at 20 °C a logarithmical CS ramp in a shear stress range between 20 Pa and 400 Pa was measured within 180 s and 180 data points were measured. A breakup criteria was included in the job to stop the measurement at a deformation higher than 20. The resulting deformation of the sample was plotted as a function of the applied stress in a double logarithmic plot. According to the tangent cross-over method, the yield stress is the point of intersection of two straight lines fitted to the two regimes with different slopes, namely the creep regime and the regime of flow.
Alternatively, the rheometer can be used as a standalone unit with internal measuring routines [Fig. 2] especially recommended for standardized measuring and evaluation routines.

Fig. 2: HAAKE Viscotester iQ used as a standalone unit with internal measuring procedures, HAAKE Viscotester iQ RheoApp for extended measuring capabilities

Measurements, Results and Discussion

The measuring curves of the adult toothpaste and the children's product are shown in Fig. 3. Two curves for each sample are included to demonstrate the high level of reproducibility. The yield stress values are summarized in Tab. 1. As expected the yield stress of the children toothpaste is with a value of approximately 55 Pa much lower than the adult product with an average of 215 Pa.

Toothpaste Sample	Yield Stress [Pa]	Deformation [-]
Children Product Meas. No 1	55.4	0.21
Children Product Meas. No 2	55.6	0.27
Adults Product Meas. No 1	210	8.64
Adults Product Meas. No 1	222	10.61

Table 1: Yield stress values of toothpaste (20 °C, CS mode, manual evaluation)

Two versions of yield stress evaluation are available in the HAAKE RheoWin software: the automatic routine and the manual mode (Fig 4.). Using the HAAKE Viscotester as a standalone unit only the automatic evaluation routine is available.

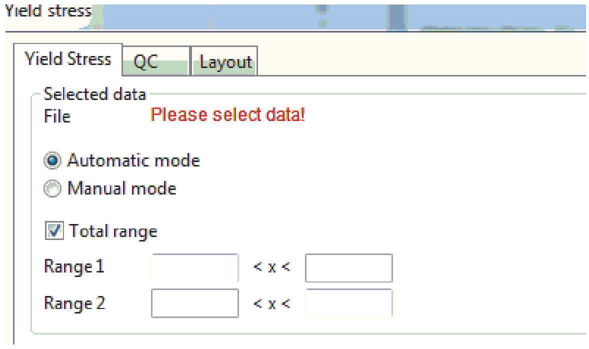

Fig. 4: HAAKE RheoWin software element for the yield stress determination in CS mode: automatic or manual mode

The manual routine gives more flexibility in finding the correct value for the yield stress. The range in which the yield stress is is normally well visually detectable and the measuring ranges for the fit of the linear ranges can be selected. This mode has been used for the calculation of the yield stress values presented in Tab. 1. However, the yield stress strongly depends on sample preparation, the measuring routine itself but also on the evaluation procedure. Fig. 5 includes a comparison between both modes on a children toothpaste.

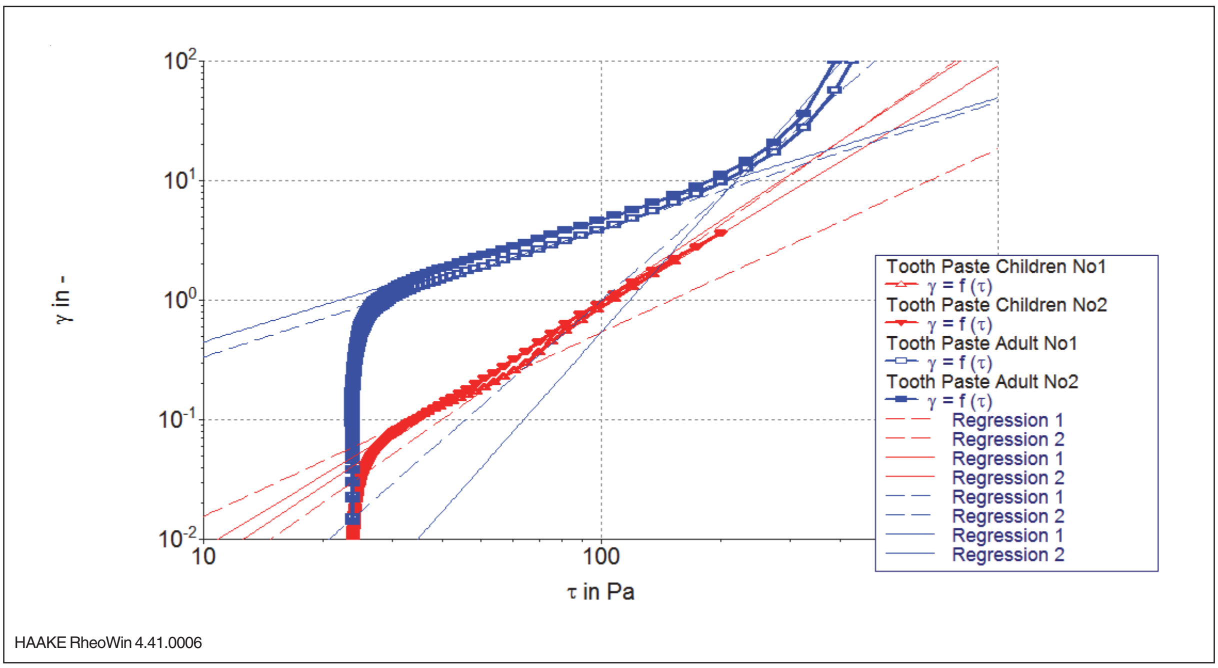

Fig. 3: Deformation as a function of stress for two different toothpastes (20 °C, 35 mm parallel plate measuring geometry with serrated surface, 1 mm measuring gap). The yield stress is calculated as the point of interception of the two tangents fitted on the manually chosen ranges of the elastic deformation and the viscous flow.

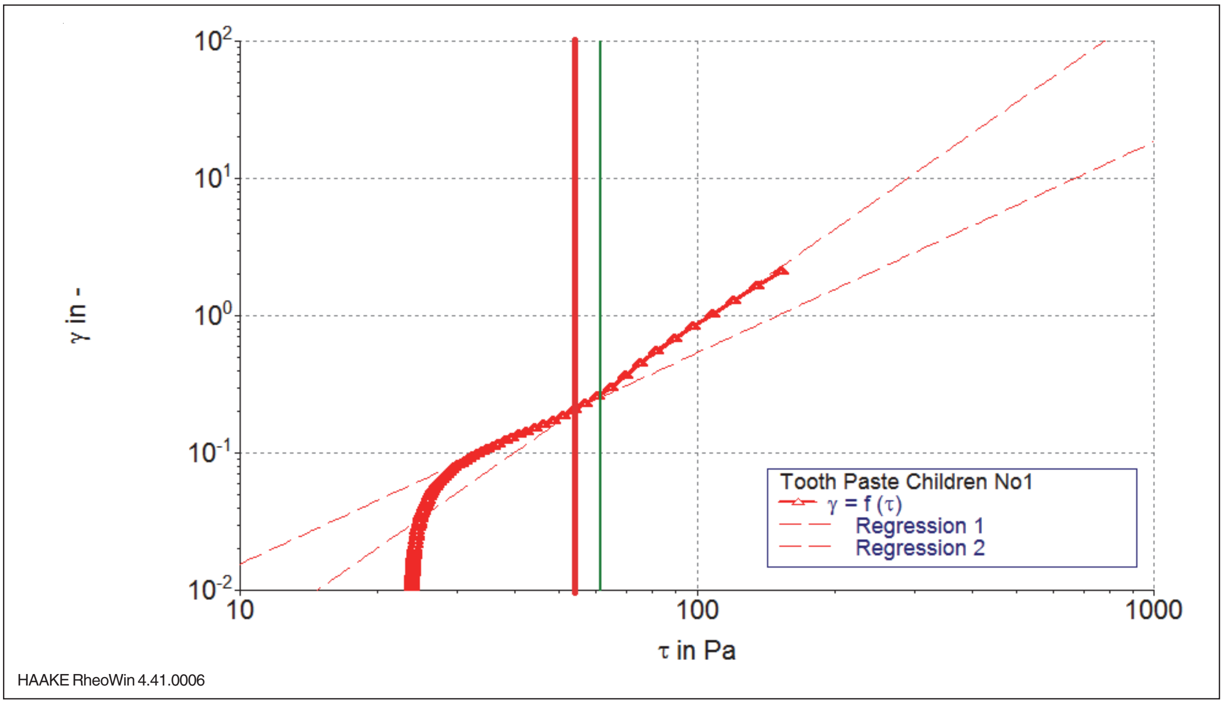

Fig. 5: Yield stress evaluation using the automatic mode (61.68 Pa, green line) and the manual mode (55.39 Pa, red line)

Summary

The determination of the yield stress is an important rheological parameter to understand if a toothpaste meets the expectation of the customer target group. The yield stress strongly depends on the sample history and preparation, the measuring conditions and method as well as the evaluation routine.

The HAAKE Viscotester iQ is a compact rheometer focusing on modern QC demands. The modular design enables an adaptation to individual needs thanks to a broad range of accessories. Quick coupling and recognition of the temperature module as well as the measuring geometry in combination with an intuitive instrument concept with a smart lift function, guarantees smooth handling. The HAAKE Viscotester iQ is the instrument of choice as stand-alone unit for standard measuring and evaluation routines or as fully software controlled version with the HAAKE RheoWin software for highest measuring flexibility.

Literature

[1] H.-M. Petri, E.-M. Kutschmann, "Rheological Charactercation of Toothpaste“ Thermo Scientific Application Note V142

[2] DIN Technical Report No. 143 of the NPF/NAB-AK 21.1 "Rheology“ (Pigments and Extenders)

[3] C. Küchenmeister, J. Nijman, "Exchangeable lower plates for temperature module“ Thermo Scientific Product Information P029

thermoscientific.com/mc

Material Characterization

Benelux
Tel. +31 (0) 76 579 55 55
info.mc.nl@thermofisher.com

China
Tel. +86 (221) 68 65 45 88
info.mc.china@thermofisher.com

France
Tel. +33 (0) 1 60 92 48 00
info.mc.fr@thermofisher.com

India
Tel. +91 (20) 6626 7000
info.mc.in@thermofisher.com

Japan
Tel. +81 (45) 453-9167
info.mc.jp@thermofisher.com

United Kingdom
Tel. +44 (0) 1606 548 100
info.mc.uk@thermofisher.com

USA
Tel. +1 603 436 9444
info.mc.us@thermofisher.com

International/Germany
Dieselstr. 4
76227 Karlsruhe
Tel. +49 (0) 721 4 09 44 44
info.mc.de@thermofisher.com

Thermo SCIENTIFIC
Part of Thermo Fisher Scientific