Performing quality control tests on cosmetic emulsions with the HAAKE Viscotester iQ Rheometer

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Introduction

The formulation of a cosmetic product can be quite complex and will vary depending on the active ingredients. Still, the customer expects a cream from a certain product range always to have the same texture, no matter the particular ingredients like for example aloe vera, lemon grass or milk and honey. Subsequently, one of the challenges when formulating a cosmetic product is to achieve the texture the customer expects or desires. Testing the rheological properties of a cosmetic emulsion therefore is an essential part of such a product's quality control (QC).

Typical rheological parameters tested for cosmetic emulsion are the shear viscosity η, the thixotropic behaviour and the yield stress τ_0. The viscosity is related to various product properties depending on the shear rate range in question. For example, the feel of a cream when rubbing it into the skin is linked to the viscosity at higher shear rates, while the storage stability is related to its viscosity at low shear rates.

The yield stress of an emulsion is important for the storage stability and the look and feel of the product. A cream sold in a pot will have higher yield stress because the customer expects to be able to take a bit of cream out of the pot with the finger. A body lotion is filled in bottles and the customer expects a more liquid-like behaviour. Thus this products needs to have a relatively low yield stress.

To be able to test these different rheological parameters, a QC rheometer needs to offer a variety of test methods and a broad measuring range. To make rheological tests in QC easier and more reliable, the Thermo Scientific™ HAAKE™ Viscotester™ iQ Rheometer has been developed. This unique rheometer comes with various some features especially designed for demanding QC environments.

Figure 1: HAAKE Viscotester iQ Air Rheometer with Peltier temperature control unit and parallel plate geometry..

Materials and methods

A soft cream and a body lotion have been selected for the tests described in this report. To reduce the damage to the emulsion's structure during sample loading, the tests were carried out using a 35 mm parallel plate geometry with a 1 mm measuring gap. Compared to a cylindrical measuring geometry this reduces the sample volume to about 1 ml and helps saving time by reducing the cleaning effort after each test.

Before the tests a small amount of sample was placed onto the lower exchangeable plate placed on the Peltier temperature control unit of the HAAKE Viscotester iQ. The upper plate was lowered carefully by hand down to the measuring gap in order to minimize the preshearing of the samples. Finally a sample cover was put over the closed measuring geometry to improve temperature control and to minimize evaporation.

After lowering the upper plate to the measurement position, the HAAKE Viscotester iQ Rheometer was operated using the Thermo Scientific™ HAAKE™ RheoWin™ Software.

The first part of the test protocol was for sample conditioning. For the emulsions tested here it consisted of a waiting period to give the samples enough time to release the mechanical stress applied during sample loading and to reach a temperature equilibrium. Sample conditioning should always be included in the test method itself to ensure that it is not forgotten and always performed in the same way, to ensure reproducible results.

The sample conditioning step was followed by the rheological test itself and the last part consisted of the data evaluation, the generation of a measurement report and its printout or export if required.

Results and discussion

The viscosity of complex fluids is best tested by recording the steady-state viscosity curve i.e. the steady-state viscosity as a function of shear rate. Compared to transient viscosity data from shear rate ramp experiments, the steady-state viscosity is independent of any time-dependent effects.

Within the almost four orders of magnitude in shear rate covered during the test, both viscosity curves displayed in Figure 2 show a pronounced shear thinning behaviour. As expected, the soft cream has the higher viscosity at low shear rates but it also shows the stronger shear thinning behaviour. Within the range of these measurements, the viscosity of the soft cream drops by a factor of almost 500, while the viscosity of the body lotion only drops by a factor of 120.

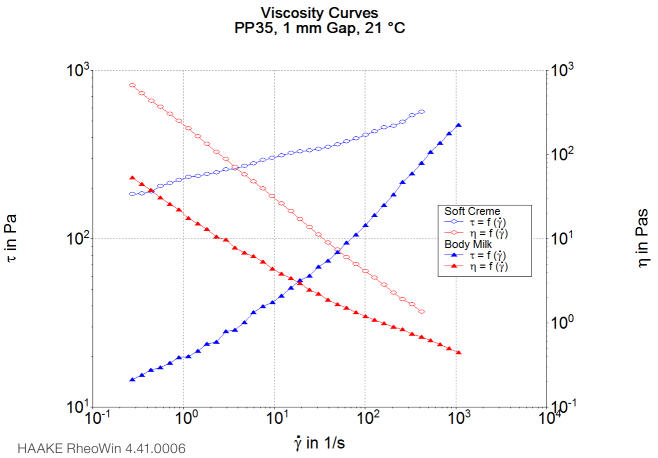

Figure 2: Viscosity curves (red) and flow curves (blue) of a soft cream (open symbols) and a body lotion (filled symbols). The soft cream shows the higher viscosities and the stronger shear thinning behaviour.

Extrapolating both viscosity curves to higher shear rates predicts that the viscosity of both products will be identical at approximately 2000 s^{-1}. This similarity is not accidental because rubbing a cream or lotion into the skin happens in that range of shear rates and the viscosity, which is felt as being pleasant on the skin, is of course the same for both products.

Thixotropy

In QC, the thixotropic behaviour is usually tested with a so-called thixotropy loop experiment. The advantage of this method compared to others commonly used for measuring thixotropic behavior is its short test duration.

The thixotropy loop test consists of three consecutive steps: A shear rate ramp from 0 s^{-1} up to the maximum shear rate (in this case 200 s^{-1}), an element keeping the maximum shear rate constant over some time and a second shear rate ramp going back to 0 s^{-1} (Figure 3). Usually the durations of the upward and downward ramp are identical. The time for the constant shear element should be long enough for the sample to reach a constant viscosity before starting the downward ramp.

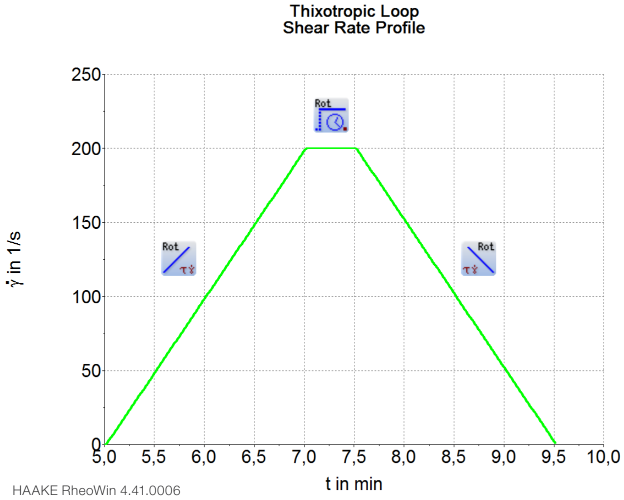

Figure 3: Shear rate profile for a thixotropy loop test consisting of an upward ramp, a steady shear step at the maximum shear rate and a downward ramp.

The results of these tests are evaluated by calculating the area between the flow curves (shear stress vs. shear rate) from the upward and the downward ramp (hysteresis). The bigger the area, the stronger the thixotropic behavior of the sample.

There is one important fact about thixotropy loop experiments to keep in mind: the results of this test strongly depend on the test settings, i.e. the maximum shear rate and the duration of the ramps. Only if the shear rate profile (like e.g. shown in Figure 3) is kept the same, results from different tests can be compared.

The results of the thixotropy loop tests performed on the two cosmetic products revealed significant differences. While the soft cream clearly showed a thixotropic behaviour (Figure 4, open symbols), the body lotion showed no thixotropy at all.

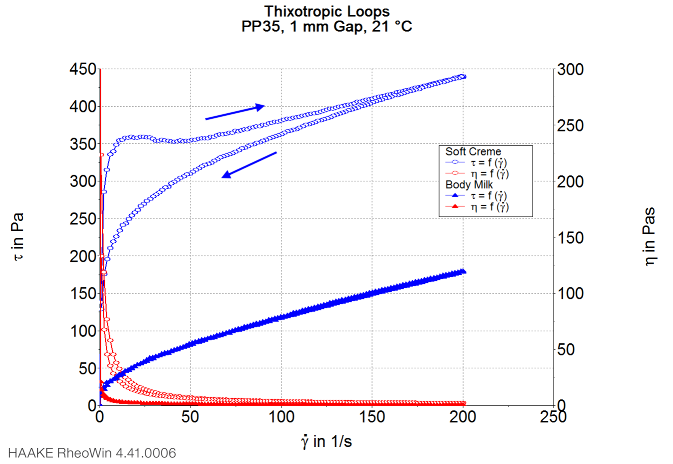

Figure 4: **Results of the thixotropy test for soft cream (open symbols) and body lotion (filled symbols). The soft cream shows a thixotropic behaviour, indicated by the clear difference between the upward and the downward flow curves. For the body lotion both curves are identical, thus the sample is not thixotropic.**

Yield stress

If a material exhibits a yield stress, it behaves like an elastic solid when exposed to a shear stress below the yield stress and like a viscous liquid when the stress applied is higher than the yield stress. Therefore, a recommended method to test, if a sample has a yield stress and at which stress value it appears, is a linear continuous increase of the applied stress (1).

For the evaluation of the test results, the deformation of the sample is plotted as a function of the stress applied. For the determination of the yield stress, the regions before and after the first significant change in slope along the curve are used. Further changes at higher stresses are related to other structural changes in the sample. Straight lines are fitted to the linear parts (in the double logarithmic plot) of the deformation curve below and above the first pronounced curvature. The yield stress is calculated from the point of intersection of these two straight lines (Figure 5).

With this method, data below as well as above the yield stress is recorded. Thus, no extrapolation is needed, which increases the reproducibility and reliability of calculated value for the yield stress considerably.

While the body lotion showed no yield stress within the range of measurement, the soft cream showed a distinct yield stress of 110 Pa (Figure 5).

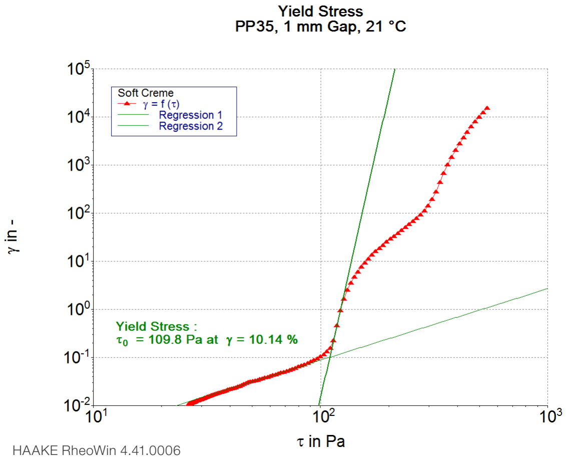

Figure 5: **Deformation as a function of stress for the soft cream sample. The yield stress is calculated as the point of interception of the two green tangents, marking the transition from elastic deformation to viscous flow.**

Conclusion

The HAAKE Viscotester iQ Rheometer is a compact instrument with the right combination of sensitivity and strength to successfully test a wide range of samples with different QC methods in controlled rate (CR) or controlled stress (CS) mode. The sensitivity of the instrument allows covering a wide range of shear rates with only one measuring geometry. For cosmetic emulsions, the parallel plate geometry proved to be particularly useful since its usage reduces the stress acting on the sample during sample loading. In addition, it significantly reduces the time between tests due to a much simpler and therefore faster cleaning procedure.

Reference

1. DIN/TR 91143-1:2022 Rheological test methods – Part 1: Determination of the yield point – Fundamentals and comparative testing methods.