QC Tests on Cosmetic Emulsions with the Thermo Scientific HAAKE Viscotester iQ

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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 his finger. A body lotion is filled in bottles and the customer expects a 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 viscometer 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 (Figure 1) has been developed. This unique viscometer has some new features especially designed for QC applications. For example, due to its improved sensitivity it is possible to use smaller cylindrical measuring geometries and even parallel plate geometries, thus reducing sample volume, time required for temperature equilibration and cleaning effort.

Preparations

A soft cream and a body lotion have been selected for the test 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 gap. Compared to a cylindrical measuring geometry this reduces the sample volume to about 1 ml and helps saving time by allowing much easier cleaning of the geometry after each test.

Fig. 1: The Thermo Scientific HAAKE Viscotester iQ

Before the tests a small amount of sample was placed onto the lower exchangeable plate sitting 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.

The HAAKE RhoWin Job

After lowering the upper plate, the HAAKE Viscotester iQ was operated using the Thermo Scientific™ HAAKE™ RheoWin™ software.

The first part of the HAAKE RheoWin job is the sample conditioning. For the emulsions tested here it simply consists of a waiting period to give the samples enough time to relax the mechanical stress from sample loading and to bring it to the test temperature. Even though it is "just" a waiting period, the sample conditioning should always

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be part of the test method itself to ensure that it is not forgotten and always performed in the same way, which improves the reproducibility of the results.
The next part of the HAAKE RheoWin job is the rheological test itself. The last part usually consists of the data evaluation, the generation of a report and its printout or export if required.

Viscosity

The viscosity 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 ramps, the steady-state viscosity is independent from time-dependent effect and the slope of the shear rate ramp. For comparison of viscosity data the steady-state viscosity is the best choice, because it is independent of the instrument used and can be correlated with the shear rate applied.
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. 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. 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 rate and the viscosity, which is felt as being pleasant on the skin, is of course the same for both products.

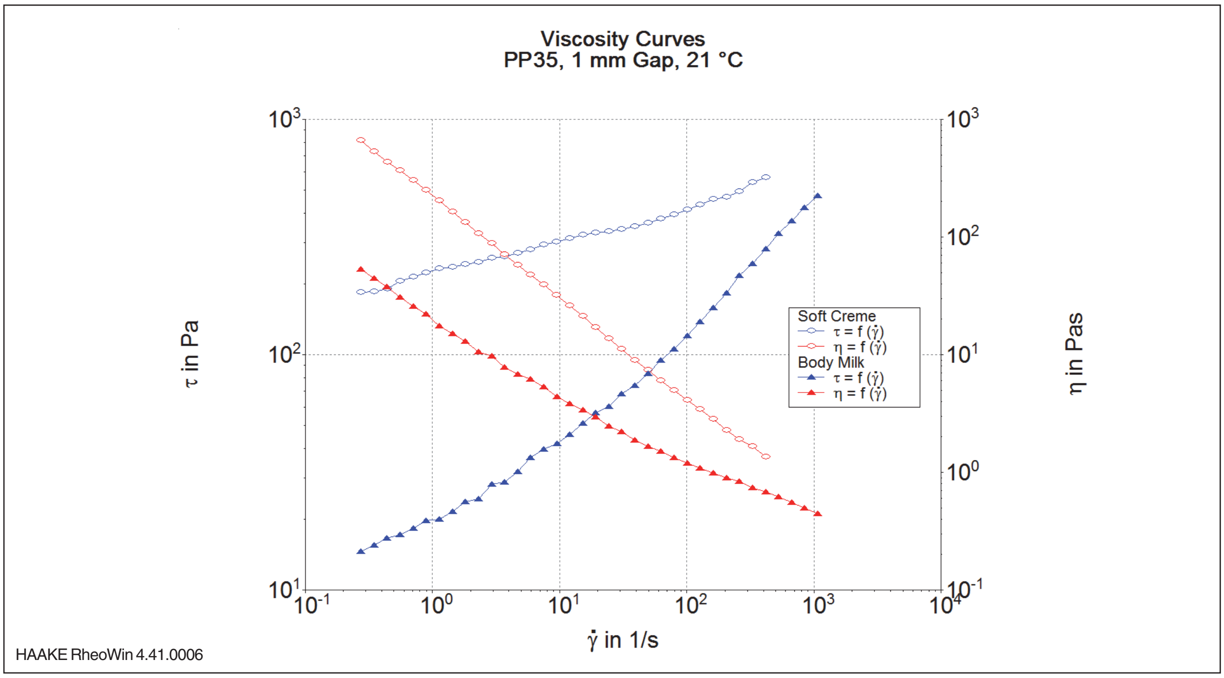

Fig. 2: Viscosity curves (red) and flow curves (blue) of a soft cream (open symbols) and a body lotion (filled symbols). The soft cream shows the higher viscosities and the stronger shear thinning.

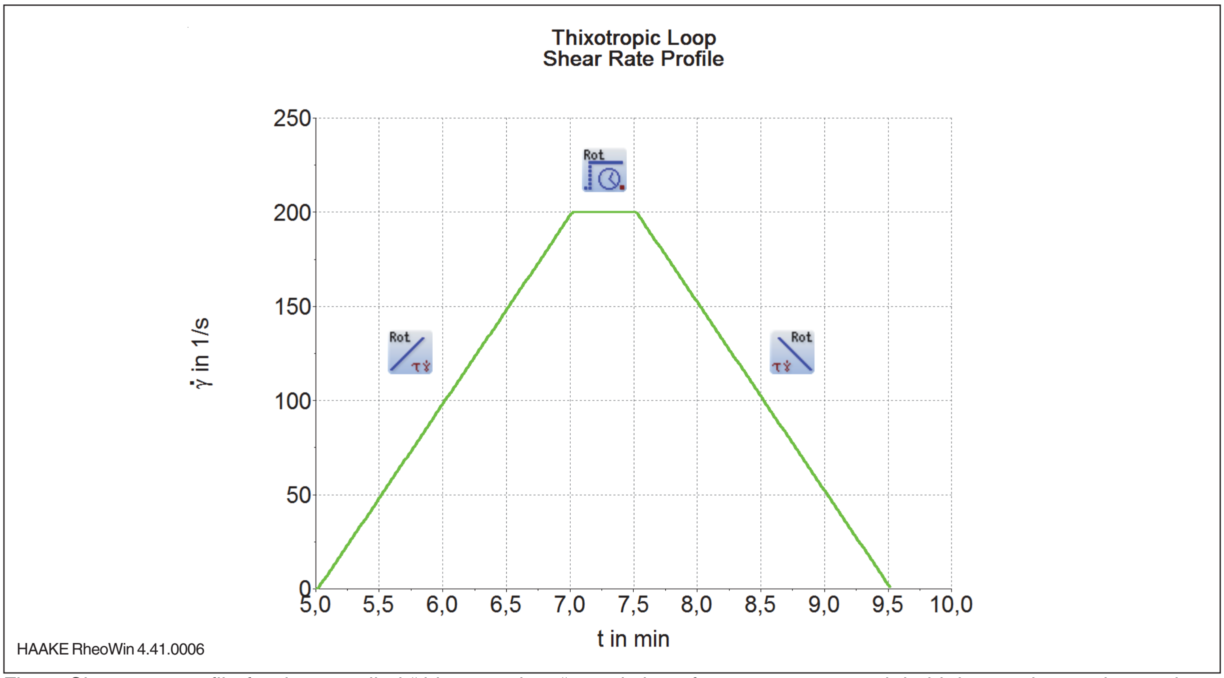

Fig. 3: Shear rate profile for the so-called “thixotropy loop“ consisting of an up ramp, a peak hold time at the maximum shear rate and a down ramp.

Thixotropy

In QC, the thixotropic behaviour is usually tested with the so-called "thixotropy loop". The advantage of this test method is the short time the test takes compared to other commonly used methods.

The test consists of 3 elements: 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 up ramp and down 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 down ramp.

The results of these tests are evaluated by calculating the area between the flow curves from the up ramp and the down ramp. The bigger the area, the stronger the thixotropy of the sample.

One important fact to keep in mind: the result of this test depends on the test parameters, i.e. the maximum shear rate and the slope 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.

Running this test on both samples gave a significant difference in the results. While the soft cream clearly showed a thixotropic behaviour (Figure 4, open symbols), the body lotion showed no thixotropy at all.

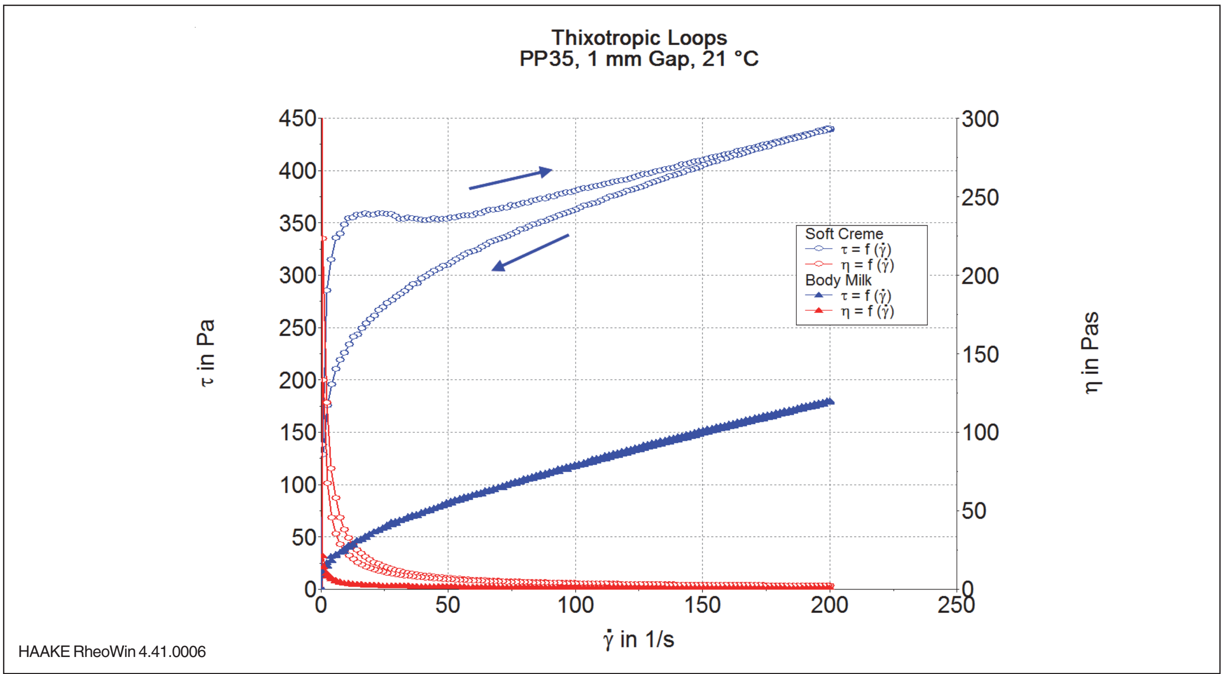

Fig. 4: Results of the thixotropy test for soft cream (open symbols) and body lotion (filled symbols). The soft cream shows a thixotropic behaviour, indicated by the clear difference between the up and down part of the flow curve. The up and down curve for the body lotion are identical, thus the sample is not thixotropic.

Yield Stress

If a material has 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, the recommended method to test, whether 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, only the first discontinuity in the deformation curve can be used. Similar points at higher stresses are related to other changes of structure 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 discontinuity. 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).

Summary

The HAAKE Viscotester iQ 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 use reduces the stress 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.

The very good quality of the results shown in this report is an excellent base for a reliable data analysis with a variety of available methods and models.

Literature

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

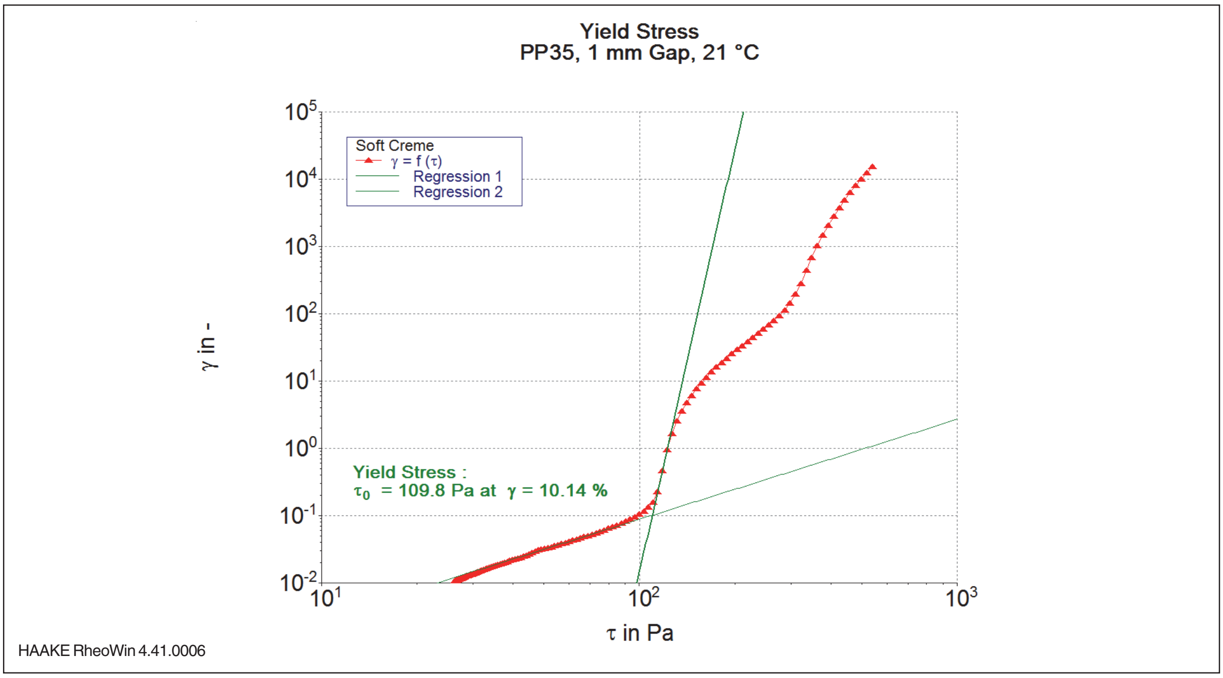

Fig. 5: Deformation as a function of stress for the soft cream sample. The yield stress is calculated as the point of interception of the two green tangents, marking the transition from elastic deformation to viscous flow.

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