Investigation of Cremes and Gel Products with the HAAKE VT550

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In quality control and production monitoring often a lot of samples have to be investigated every day. Usually the time needed for a measurement may not exceed a couple of minutes; therefore, routine measuring procedures are followed with fixed parameters. At computer-operated instruments with application software these measuring and evaluation procedures can be stored. The so-called job streams allow the start of those procedures by a push of a button after having filled the sample in the measuring system.
The used measuring geometries should be easy to fill and to clean after completion of the rheological tests. In case of very expensive products the sample volume is often restricted. For the investigation of cremes, ointments and soft gels the Viscotester HAAKE VT 500 is preferably used with a plate-cone measuring geometry. In order to guarantee a high degree of measuring sensitivity we recommend for cremes and ointments a cone with diameter of 5 cm and a cone angle of 1° (PK 5/1°). For higher viscous products also a cone or a plate with a smaller diameter can be used. Since cremes, emulsions and lotions always show a non-Newtonian flow behavior the measured viscosity alone is not sufficient to characterize the product distinctly. The viscosity does not depend on the temperature alone but also on the shear rate and in many cases also on the shearing time. Are the measurements performed manually pre-tests have to be run to find out if the displayed viscosity value is constant over the time at a selected speed. Also the preparation of the sample before filling it in the measuring system may influence the measuring results.
A good reproducibility of the measuring results can therefore only be guaranteed if the measurement is performed under clearly defined test conditions.
Fig. 1 shows a flow and viscosity curve of a facial care creme at 23°C. Here, the product was carefully filled in the measuring system without stirring it first. Plate and cone were very slowly brought to their measuring position. The flow curve (upward curve) was measured with a shear rate ramp over two minutes from 0 to 250 s^{-1}, then the shear rate remained constant at 250 s^{-1} for one minute. Then the ramp was reversed and the shear rate reduced from 250 to 0 s^{-1} in two minutes.

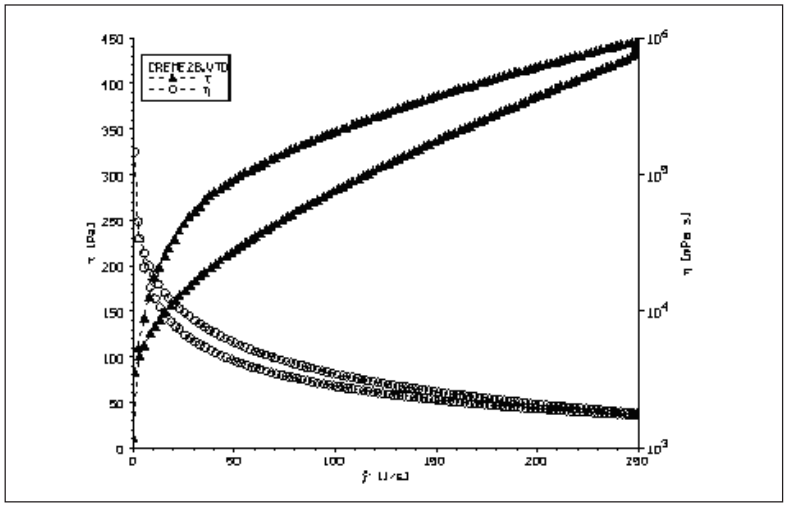

Fig.1: Flow and viscosity curve of a cosmetic creme at 23°C

The flow curve shows that the creme shows a yield point below which it behaves like a soft solid. Beyond the yield point the viscosity of the product decreased with increasing shear rate – a flow behavior which is known as plastic flow.
Furthermore it is clearly visible that the upward and downward curves are not identical. This means it is a thixotropic product whose structure is breaking down depending on the shearing time. The viscosity, measured as function of the time, decreases at a constant speed until a final equilibrium value is reached. This happens only after the complete break down of the structure after shearing. After a relaxation time which can vary from some minutes to several hours depending on the product the structure of the creme will completely recover. Than a new flow curve can be recorded which is identical to the first curve.
With the application software the area between the upward and downward curve can be calculated as measure for the thixotropy of a product.
Fig. 2 shows the flow and viscosity curve of a massage gels. This product is a very soft gel which drips slowly from a spoon. This gel is often used by athletes to cure tensed up muscles. It contains a relative high portion of volatile components (alcohol, ethereal oils).
This product was also measured with the cone PK 5/1° in a shear rate range from 0 to 250 s^{-1}.
Upward and downward curve are identical for this product, the viscosity of the gel depends on the shear rate (pseudoplasticity), a shear time

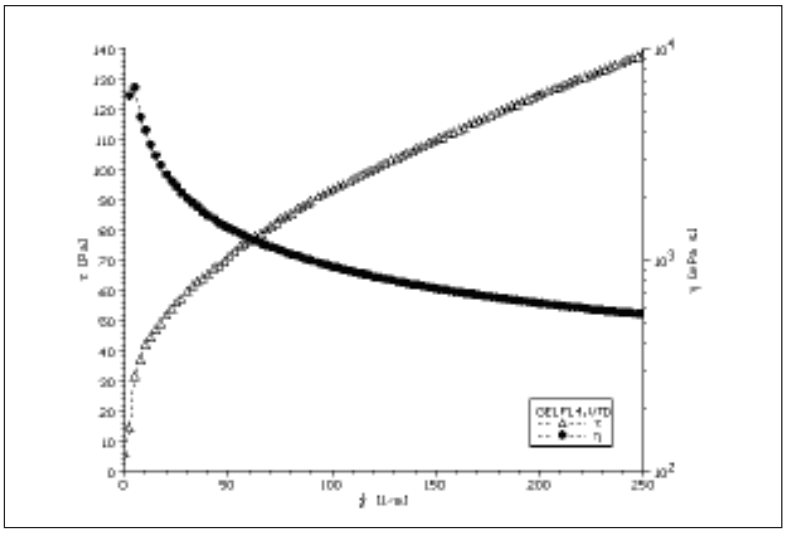

Fig.2: Flow and viscosity curve of a massage gel at 23°C

dependency (thixotropy), however, cannot be determined. This means that for the investigation of this product the recording of reverse curve can be omitted.
The beginning of the curve could imply that this gel also has a yield point. The so-called CD-mode (controlled deformation) of the HAAKE VT 550 allows an exact and reproducible yield point determination of cosmetic and pharmaceutical products (please see to this also application report V 127).
Due to the high portion of volatile components a drying out of the gel during the measurements can be observed. The measured viscosity increases slowly with the time if measured at a constant shear rate.

A second measurement of the same sample (without new filling) shows a flow and viscosity curve shifted to higher values. It is recommended to use a solvent trap in order to avoid a drying out of the sample during the measurement.
The application software of the HAAKE VT550 allows the evaluation of the measuring data with different regression models, comparisons with a reference curve interpolation or determination of characteristic values. The exemplary measuring procedures and evaluation methods can be combined and stored as job stream which is readily available.

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