Rotational Viscosity Testing of Ointments with ViscoQC Series

How can viscosity testing support quality control of pharmaceutical semi-solids? This application report shows how simple viscosity measurements of pharmaceutical formulations with the ViscoQC Series help to retain steady, perfect consistency of medicinal suspensions.

1 Introduction

Ointments, creams and lotions comprise the largest part of semi-solids in pharmaceutical industry. The consistencies of semi-solids during formulation have to be monitored for quality control. In addition, regulations of pharmacopeias must be followed. The viscosity of ointments, for example, has an impact on several stages in the production: It must be possible to pump them, pack them, squeeze them out of the packing easily and apply them on the target body areas. For the final reason, viscosities of ointments have to decrease when a force ("shear") is applied (e.g. rubbing action). Therefore, perfect spreadability can only be obtained with a shear-thinning flow behavior of ointments.

1.1 Keywords

Viscometer pharma, viscometer ointment, viscosity quality control, touch viscometer, rotational viscometer, dynamic viscosity, digital viscometer

2 Experiment

The viscosity of three different types of ointments was determined. Whereas ViscoQC 100 – H was used for a quick single point check at 80 rpm, with ViscoQC 300 – H a multi-point analysis at 9 speeds was performed. With both models, the absolute measuring system CC18 was used (Table 1).

Sample	Ointment A, B and C	
Instrument	ViscoQC 100 - H	ViscoQC 300 - H
Measurement type	Single-point	Multi-point
Spindle	CC18	
Speed	80 rpm	20, 30, 40, 50, 60, 70, 80, 90, 100 rpm
Temperature	ambient conditions	

Table 1: Configuration and measurement conditions during viscosity test of ointments with ViscoQC Series.

2.1 Test Procedure

Single-point viscosity determinations using ViscoQC 100 are perfectly suited for quick quality control checks of pharmaceutical semi-solids.

For multi-point analysis at different speeds to study the flow behavior, ViscoQC 300 is the best choice.

Both upgradeable software packages available for ViscoQC 300, V-Curve and V-Comply, have been activated on the stand alone instrument. V-Curve is needed to see online live data in a graph and for analysis with mathematical regression models. For increased security settings, audit trail, electronic signature functionality, and more, V-Comply is necessary.

The measurement mode "Speed Scan (SpS)" of ViscoQC 300 can be used for performing a speed ramp. The speed is automatically increased step-wise from low to high speeds in a linear scale with a defined measurement point number and duration.

2.2 Test Conditions

- 6.5 mL of the sample were filled into the CC18 using a syringe and the system was mounted on the ViscoQC with the DIN adapter.
- With ViscoQC 100, the viscosity was determined at 80 rpm using the measurement mode "Stop at Time (@t)". The single-point measurement time was set to 30 seconds. With ViscoQC 300, a linear speed ramp from low to high speed with 9 measurement points was performed. The measurement point duration was set to 30 seconds for the whole speed range.

- Measurements were repeated five times and average viscosity values were calculated.
- Using ViscoQC 100, the data has to be printed directly after measurements via V-Collect or Dymo® LabelWriter™ as it does not feature a data memory. While performing measurements with ViscoQC 300, the measurement data are stored in the data memory of the instrument. For that reason, measurement data can be printed/exported after measurement via V-Collect, Dymo® LabelWriter™, pdf export/csv table, page printer (network/USB) and LIMS.

3 Results and Discussion

The determined single-point viscosity value with ViscoQC 100 is shown in Table 2. Ointment A and B show a similar viscosity, whereas ointment C features a viscosity approximately 3 times lower. Ointment A and B are gels, which have to stay long on the affected body site. Ointment C acts as a hydrating lotion and has to be applied fast on a large body area. Thus, it has to be easily spreadable, which its low viscosity makes possible.

	Ointment A	Ointment B	Ointment C
Speed [rpm]	80	80	80
Torque [%]	74	62	25
Viscosity [mPa·s]	2691	2269	839

Table 2: Averaged viscosity value at 80 rpm of ointment A, B and C (n = 5). Measurements have been performed with ViscoQC 100 – H.

By analyzing the viscosity at different speeds with ViscoQC 300, a shear thinning flow behavior of all three samples could be determined. The viscosity decreases as speed increases.

As V-Curve has been activated on ViscoQC 300, mathematical models (e.g Bingham, IPC-Paste, Casson) can be applied to the generated measurement data. As a further consequence the regression model “Best fit” (Figure 2 shows, as an example, ointment C) was applied to each measurement. Using “Best fit”, the mathematical model with the best correlation coefficient is automatically chosen by the device.

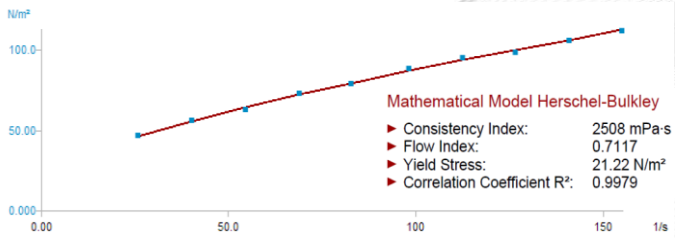

Figure 2: Flow curve of one measurement of ointment C with ViscoQC 300 (V-Curve). The mathematical model “Best fit” was applied and Herschel-Bulkley was selected by the device.

To compare the yield stress of each sample, the mathematical model “Herschel-Bulkley” was applied to them (Table 3). The yield stress is the lowest shear-stress value above which a material will start to flow and behave like a liquid. Ointment A requires the highest force when pressing on the tube to make it flow out of the tube. The yield stress is of vital importance in quality control to satisfy customer’s requirements.

Sample	Yield stress [N/m^2]
Ointment A	82.39
Ointment B	30.94
Ointment C	21.22

Table 3: Calculated yield stress of ointment A, B and C using the “Herschel-Bulkley” model.

4 Summary

The measurements showed that the digital viscometer ViscoQC is perfectly suited to determine the viscosity of ointments, with ViscoQC 300 even at different speeds. The viscosity of the samples gives you valuable information on the quality of pharmaceutical semi-solids. The perfect consistency can be easily checked with the ViscoQC 100 at single-points or with the ViscoQC 300 at multiple points. The flow behavior of suspensions has been tested by performing an automated speed ramp with ViscoQC 300. The torque model H of ViscoQC series is perfectly suited to analyze high-viscosity samples such as ointments.

To simplify and reduce the time during instrument qualification, Anton Paar offers a pharma qualification package smart (PQP-S) and a pharma qualification package (PQP). For the non-storage instrument ViscoQC 100, a PQP-S is available, that fulfills the requirements of GMP, GAMP 5 except 21 CFR Part 11. Upon upgrade of ViscoQC 300 with the optional software package V-Comply, also the documentation PQP is included. Both, software as well as the documentation of ViscoQC 300 with V-Comply fulfill the regulations of 21 CFR Part 11.

If you have further questions regarding this application report, please contact your local Anton Paar representative.

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