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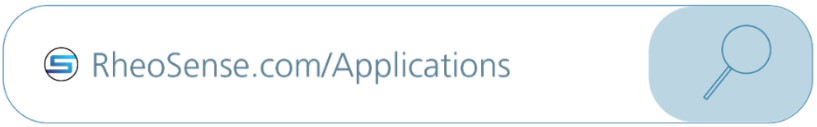
Protocol: Measuring Viscosity of Cosmetics with *micro*VISC™

Key Words: Face lotion, hand lotion, cream, viscosity, shear thinning, cleaning, high shear, non-Newtonian fluid, protocol, thixotropy

Goal: Cosmetic lotions are often non-Newtonian fluids with yield stresses and thixotropic structure. These properties are vital for their function as selfcare products, but also make their rheological properties difficult to characterize. In particular, data collection can be time consuming and instrument cleaning can be challenging. This application note will share an easy-to-implement protocol to precisely measure complex cosmetic formulations using VROC technology.



Introduction

Skin care is an important part of personal healthcare. The epidermis is one of the body's largest organs and is the first line of defense against pathogens, so maintaining and promoting healthy skin is vital for healthy life. Skincare products, specifically lotions, have been used for decades to promote skin health. When developing these products, manufacturers must carefully choose ingredients that must meet both consumer tastes and regulatory standards. These constraints make it vital to characterize lotion formulations, but also these criteria make consistent and fast measurements more challenging. Lotions are comprised of multiple materials including polymers, oils, waxes, gels, and silicones (Sharma et al. 2018). These ingredients can dramatically alter the viscosity of the lotion, but also make measuring viscosity difficult, messy, and variable. The combination of these ingredients can even cause lotions to behave as thixotropic materials.

Thixotropy is the time dependent shear thinning property of non-Newtonian fluids (Mewis 1979). Thixotropic materials will slowly return to their high viscosity state once shearing has ceased. The thixotropic behavior of lotions can result in inconsistent data. The same shear force and/or shear rate can be applied to a material multiple times in quick succession and the measured viscosity will vary after each measurement because the microstructure (one of the driving forces of thixotropy) of the material has not recovered to its original state.

Here we present a ten-step protocol to measure high viscosity, complex lotion samples using Viscometer-Rheometer-on-a-chip (VROC) technology. This protocol will describe the steps to prime the VROC chip with glycerol, run a lotion sample, and clean the VROC chip with glycerol, using minimal material, quickly and efficiently, resulting in repeatable data. The lotion tested in this protocol had an average viscosity of 424.40 mPa-s, a standard deviation of 1.68 mPa-s, and a %RSD (relative standard deviation) of 0.40%.

Materials

*micro*VISC™	B20 VROC Chip	1 mL NORM-JECT Syringe
Temperature Controller	99.5% Glycerol	Compressed Air
*micro*VISC™ Pipette	Lotion Sample	Lint Free Wipe

Protocol

1.1 Insert a B20 VROC Chip into the chip slot, turn on the *micro*VISC™ and temperature controller, set to the desired temperature (i.e. 25 °C) and wait until temperature stabilizes (approximately 30 minutes).
NOTE: The Temperature Controller can be programmed to turn on and stabilize its temperature at a set time so that the *micro*VISC™ is at the appropriate temperature at the beginning of the experiment.

1.2 Use a *micro*VISC™ pipette to draw 400 µL of 99.5% Glycerol solution. Clean the tip of the pipette with a lint free wipe, then use compressed air to remove any excess dust and/or fibers from the tip. Ensure that there are no bubbles in the pipette. If there are bubbles in the pipette remove them by redrawing the solution. Once, the pipette is prepped, place it into the *micro*VISC™. Wait at least 1 minute to allow the solution within the pipette to reach the set temperature.

1.3 Use advanced mode at the following settings: rate value = 300 sec^{-1}, Measurement Volume = AUTO, Priming Volume = AUTO, Pausing Time = 5 sec. Run experiments until the sample volume reaches 0 and save the selected data to a file.
NOTE: If you are concerned about over pressuring the chip with glycerol, run automatic mode first to determine a safe shear rate for cleaning and then run on advanced mode with a safe shear rate.
NOTE: The viscosity results from this step will be used as a reference during cleaning.

1.4 Use a 1mL NORM-JECT syringe to load 400 µL of the lotion sample into the *micro*VISC™ pipette. Use the compressed air to remove any dust and/or fibers from the pipette tip. Place the pipette into the *micro*VISC™ and wait at least 1 minute to allow the solution inside the pipette to reach the set temperature Figure 1.

1.5 Run the *micro*VISC™ on automatic mode. Running the instrument on automatic mode will provide a reference viscosity for the sample. This reference viscosity will be used to determine the shear rate necessary to conduct further measurements. Run the entirety of the 400 µL through the instrument.

1.6 Based on the viscosity measurement acquired in step 1.5, use advanced mode to set a rate value that is appropriate for the sample. For the sample tested in this application note the appropriate shear rate was 150 1/s. The Set the Measurement Volume and Priming Volume to AUTO. The pause time is sample dependent. To determine the appropriate Pause Time for the sample, load another *micro*VISC™ pipette with 400 µL of sample and place it into the pipette slot.
NOTE: This shear rate is appropriate because it is below the pressure safety threshold of 95 % full scale of the VROC Chip. VROC technology uses pressure sensors to measure viscosity which can be damaged if the pressure within the Chip is too high. The *micro*VISC™ monitors the pressure inside the Chip with the % Full Scale which is written as P-Scale, % in the *micro*VISC™ software.
NOTE: Pause Time is the time the instrument waits or "pauses" before performing the next measurement.

1.7 In advanced mode, set the Pause Time to 10 seconds and click run. Once the measurement is complete set the Pause Time to 20 seconds and click run. Repeat these steps for the following suggested pause times: 30 seconds, 60 seconds, 90 seconds, 120 seconds, 180 seconds. The minimum pause time necessary for the sample will be the pause time where the viscosity value stabilizes. Once an appropriate pause time is discovered multiple measurements can be acquired at multiple shear rates to characterize the lotion formulation.

1.8 To clean the VROC Chip use a *micro*VISC™ pipette to draw 400 µL of 99.5% Glycerol solution. Clean the tip of the pipette with a lint free wipe, then use the compressed air to remove any excess dust and/or fibers from the tip. Ensure that there are no bubbles in the pipette. Then place pipette into the *micro*VISC™. Wait 1 minute to allow the solution in the pipette to reach temperature.

1.9 Use advanced mode at the following settings: rate value = 300 sec^{-1}, Measurement Volume = AUTO, Priming Volume = AUTO, Pausing Time = 5 sec. Run experiments until the sample volume reaches 0 and save the selected data to a file. Repeat this process until the measured viscosity stabilizes to the value found in step 1.3.
NOTE: The viscosity typically stabilizes with two (2) 400 µL pipettes of 99.5% Glycerol.

1.10 Once cleaning is complete either store the VROC Chip for future use or repeat steps 1.4-1.9 with a new sample.
CAUTION: Make sure that your samples are miscible with each other before running multiple samples on the VROC Chip.

CAUTION: Do not store the VROC Chip with sample for an extended period of time (> 8 hours). If a sample remains within the channel of the chip for too long, the sample will become impossible to remove by glycerol cleaning. If the sample is stored in the chip for too long the glycerol cannot displace the aged sample. The VROC chip must always be cleaned and stored with glycerol once experiments are finished.

Data and Analysis

A sample of hand lotion was back loaded into the *micro*VISC™ pipette using 1mL NORM-JECT syringe (Figure 1). The pipette was then placed into the *micro*VISC™.

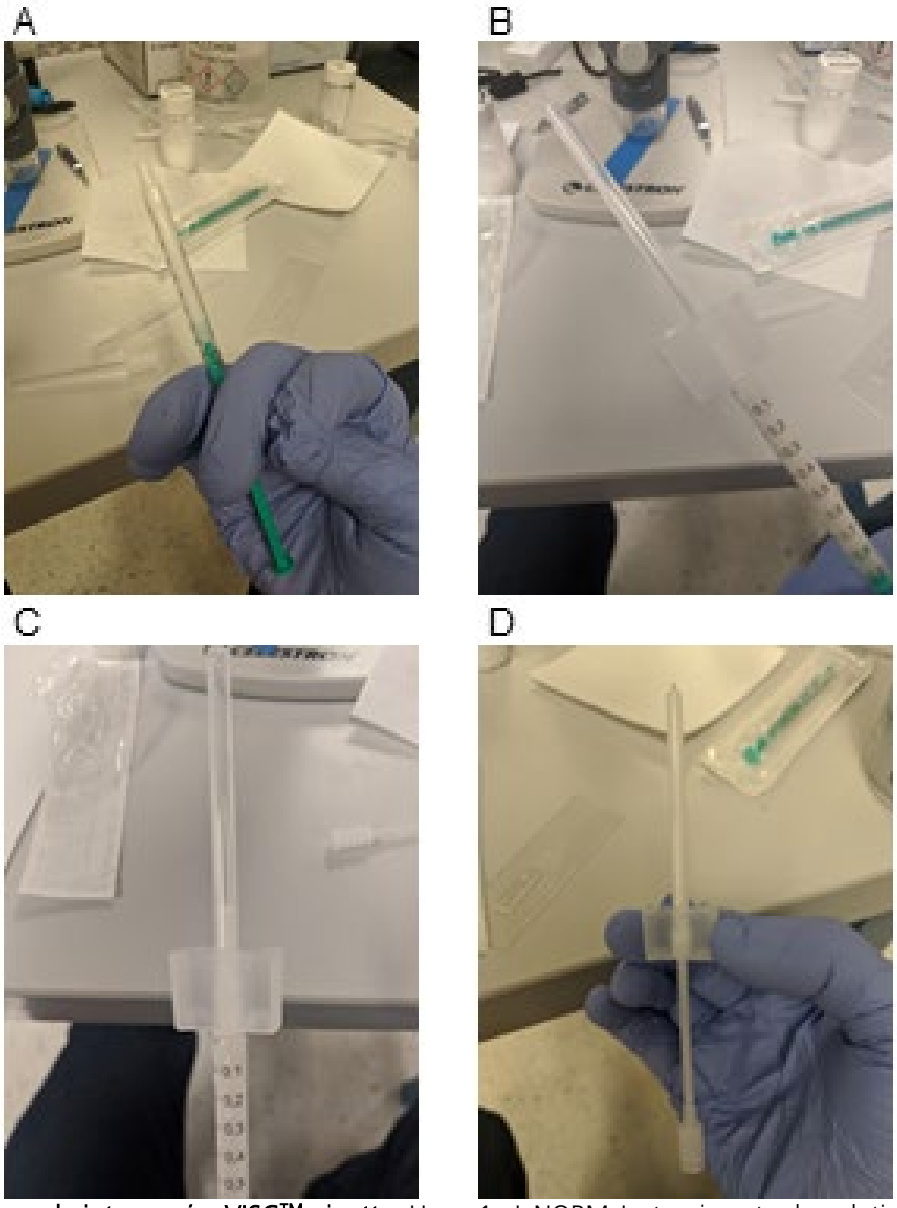


Figure 1. Loading lotion sample into a *micro*VISC™ pipette. Use a 1mL NORM-Ject syringe to draw lotion out of the container (A). Backload the barrel of the *micro*VISC™ pipette with the desired sample (B). Slowly inject the sample into the *micro*VISC™ pipette barrel (C). Insert the *micro*VISC™ pipette plunger into the barrel (D).

The pause time to conduct measurements on this sample was determined by following **step 1.7** of the protocol. For the first pipette used (Run 1), measurements were collected using 10, 20, 30, 60, 90, 120, 180, and 240 second pause times at a shear rate of 150 sec^{-1} **(Figure 2)**. Based on the data collected it was determined that a pause time of 120 seconds was enough to allow the lotion sample to recover its microstructure (relax) and generate consistent measurements.

To demonstrate that a 120 second pause time was appropriate, another pipette of lotion sample was loaded and ran on the *micro*VISC™ at a shear rate of 150 sec^{-1} (Run 2). The average viscosity measured with the 120 second pause time was 424.40 ± 1.68 (standard deviation) mPa-s **(Figure 2)**. The percent difference in viscosity versus pause time with respect to the 120 second pause time measurement was calculated **(Table 1)**. For pause times less than or equal to 60 seconds the percent difference was greater than 1%. For pause times greater

than or equal to 90 seconds this difference was less than 1%. Thus, pause times greater than 90 seconds are sufficient for creating repeatable results. For this application note, 120 seconds was chosen to be cautious.

Pause Time (sec)	Viscosity (mPa-s)	|% Difference|
10	416.70	1.81
20	414.70	2.29
30	417.80	1.56
60	419.00	1.27
90	423.90	0.12
120	427.00	0.61
180	425.60	0.28
240	423.30	0.26

Table 1. Summary of Pause Time vs Viscosity Data Run 1. Viscosity as a function of pause time is described in the above table. The first column is the pause time used to collect the viscosity data in column two. The third column depicts the absolute value of the difference between the average viscosity of sample measured with a pause time of 120 seconds (424.40 ± 1.68 mPa-s) and the measured viscosity presented in column two.

Choosing an appropriate pause time is important for acquiring consistent results because of the rheological properties of the ingredients in lotions. Lotions have ingredients that can introduce yield stress and thixotropic properties such as slow microstructure recovery. Allowing the sample time to recover its microstructure after shearing is essential to collect accurate and repeatable viscosity data.

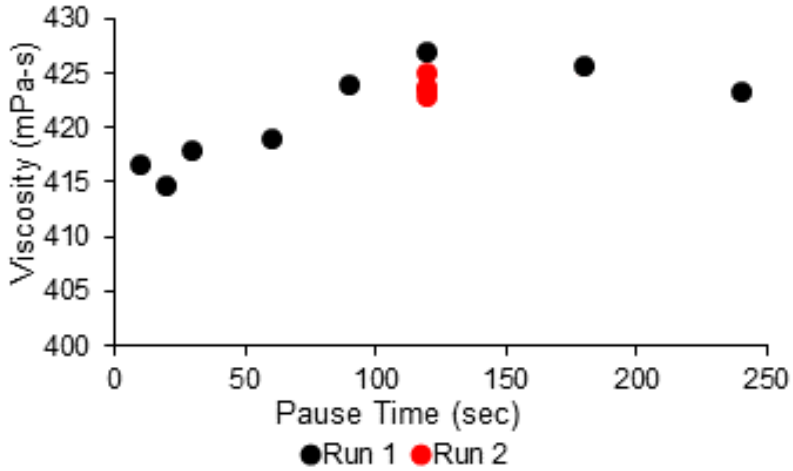


Figure 2. Viscosity of the lotion stabilizes as pause time increases. The viscosity of the sample is affected by the pause time between experiments. As pause time increases the viscosity of the sample increases (Run 1). Once an appropriate pause time is selected the viscosity of the sample stabilizes between multiple measurements (Run 2).

To demonstrate the effectiveness of glycerol cleaning, pipettes containing 99.5% glycerol were ran on the *micro*VISC™ before and after Run 1 and after Run 2 **(Figure 3A)**. Before lotion samples were introduced, one *micro*VISC™ pipette was loaded and ran on the instrument following **steps 1.2 and 1.3** in the above protocol. The viscosity data collected in this step was used as a reference for all future glycerol cleaning steps **(Figure 3B)**. The average viscosity of glycerol 1 was 896.97 ± 1.77 (standard deviation) mPa-s. This glycerol run required only one (1) pipette.

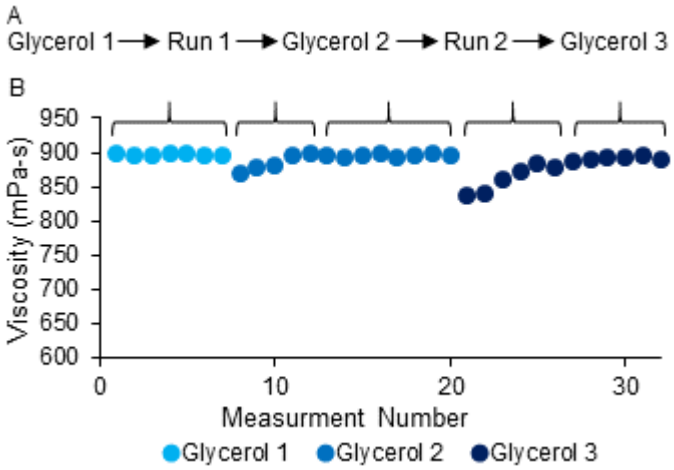


Figure 3. Glycerol cleaning is effective at displacing lotion samples. A diagram of the process described in the above protocol and the order in which glycerol and sample was introduced into the flow channel (A). The viscosity versus measurement number for each glycerol run. The brackets indicate range of points generated by a loaded *micro*VISC™ pipette. Glycerol 1 used one pipette while Glycerol 2 and 3 used two pipettes each.

After lotion sample Run 1 was performed, glycerol was used to clean the channel. The viscosity data from this glycerol run is also plotted in **Figure 3B**. This glycerol run required two (2) pipettes to clean the instrument. The data points collected from their respective pipettes are indicated by brackets in **Figure 3B**. The average viscosity from the second pipette of this glycerol cleaning step was 896.14 ± 2.00 mPa-s. Lastly, after sample Run 2 was measured, glycerol was again used to clean the VROC chip. This glycerol cleaning step required two (2) pipettes to reach a plateau value of 891.48 ± 2.71 mPa-s **(Figure 3B)**.

Concluding Remarks

Characterizing viscosity of cosmetics such as hand lotions is vital for product development. Customers expect a certain "feel" when applying lotions on their skin during their daily routines, while manufacturers need to ensure that the product is viscous enough to maintain its shape while pliable enough to be massaged into the skin. Demonstrating repeatable viscosity data is key to ensuring product quality control and can boost consumer confidence in products.

Reference

Sharma, Gaurav & Gadhiya, Jayesh & Dhanawat, Meenakshi. (2018). *Textbook of Cosmetic Formulations.*
Mewis, J., Thixotropy – a general review *Journal of Non-Newtonian Fluid Mechanics.* 1979, 6, 1.

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