Advanced rheological and tribological methods for cosmetic products

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ADVANCED RHEOLOGICAL AND TRIBOLOGICAL METHODS FOR COSMETIC PRODUCTS

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Standard Rheological Tests: Viscosity curves

Cosmetics

**S1:** hair gel with a solid structure- at- rest

**S2:** shampoo with a liquid structure- at- rest, showing the plateau of the zero- shear viscosity (maybe caused by “worm- like surfactants“)

Two different shampoos:

Same viscosity at larger shear → Same processing behavior

Different zero shear viscosity → Different feel for consumer
Standard Rheological Tests: Strain Sweep

Comparison of two Tooth Pastes, Yield and Flow Points

Strain sweep, plotted against stress

- Strain sweep often allows better testing of yield and flow behavior

Tooth pastes

paste 1

- flow point: \( \tau_f = 125 \text{ Pa} \)

paste 2

- flow point: \( \tau_f = 24.9 \text{ Pa} \)

having the same yield value, but different flow points
Standard Rheological Tests: Amplitude Sweep

Two cosmetic lotions (emulsions): Flow Point

Strain sweep, plotted against stress
Standard Rheological Tests: Amplitude Sweep

No flow point

Flow point
Small peak in $G''$, indicating network structure

Large Amplitude Oscillatory Shear (LAOS)
- Lissajous Plots
- Rheological Fingerprinting
Two cosmetic lotions (emulsions): Physical stability

Stable dispersion: gel-like with $G' > G''$ at low frequencies

Unstable dispersion: liquid-like with $G'' > G'$ at low frequencies

Angular Frequency $\omega$

$\gamma = 0.3\%$
$T = +20^\circ C$
**Temperature Sweep and Temp. Cycle Testing**

![Graph showing temperature sweep and cycle testing](https://www.makingcosmetics.com/Stability-Testing-of-Cosmetics_ep_59.html?locale=de)

**Cycle Testing:** The product should pass three cycles of temperature testing from -10°C (14F) to 25°C (77F). Place the product at -10°C for 24 hours and place it at room temperature (25°C) for 24 hours. This completes one cycle. If the product passes three cycles then you can have a **good degree of confidence in the stability of the product.**

Temperature Cycle Testing with Rheology

Stable Creme
- No change in moduli

Unstable Creme
- Changes in moduli
- $\tan(\delta)$ increasing

Measurements: Prof. Jakob
Technische Hochschule Nürnberg / Germany
Three Categories of Combined Techniques

**Structural Information**
- Additional Information on the Micro-Structure simultaneous to the rheology
  - SALS
  - Microscopy (Polarized, Fluorescence, Confocal)
  - Birefringence / Dichroism
  - Visualization: PTV, PIV
  - Polarized Imaging (SIPLI)
  - IR / NIR Spectroscopy
  - SAXS / SANS
  - Dielectric Spectroscopy

**Additional Parameter**
- Rheological testing under simultaneous application of additional parameters
  - Temperature
  - Pressure
  - Magnetic Field
  - Electric Field
  - UV-Light
  - Water Reduction
  - Humidity

**Non rotational shear rheology**
- Use of the rheometer to perform non-classical shear rheology
  - DMTA in Torsion and Tension
  - Extensional Rheology
  - Tensile Testing
  - Tack
  - Interfacial Rheology
  - Tribology
  - Powder Rheology
Temp. Test: Rheology & Dielectric Spectroscopy

- Cosmetic products are often opaque, i.e. optical techniques are not possible
- Die-electrical spectroscopy is an option to “see” into such samples

The body cream (water in oil emulsion) freezes around -15 °C making the sample stiffer and harder represented by the sudden increase in storage and loss modulus.

Dielectric spectroscopy recognizes the phase transition of water by a sudden decrease in permittivity. This may be related to the decreased mobility of the surrounding molecules once the water is frozen.
Rheo-Microscopy

- Different lenses for different magnification
- Specially designed lens (20x) for highest quality
- Modular light source
- Polarization module
- Fluorescence module
Rheo-Microscopy: Emulsions

Polymer Blend: PIB in PDMS

Droplet size and shape is related to the shear rate and shear history.
Counter Rotation for Microscopy

**Problem:**
With one motor moving structure elements do not stay in the field of view

**Solution:**
Counter Rotation of two motors is producing a stagnation plane
Counter Rotation for Microscopy

Particles are stretched and rotated, but not moving at stagnation plane.

PIB in PDMS
Counter Rotation for Microscopy

Developer cream for hair coloring at small gap (50µm)
0.5s⁻¹ – 50s⁻¹

Strong shear thinning

- Structure consists of small crystallites
- Crossover from a jammed to a flowing system
Counter Oscillation for Microscopy

Release of material from capsules observed by Rheo-Microscopy
Counter Rotation for Microscopy

Capsule for cosmetic applications in a gel: 0.1, 0.3, 1, 3, and 10s-1

- Cup with glass bottom
- Cylinder bob with cone at bottom
- 5x Objective: Image size: 1.33 x 1.7 mm
- Observation point up to 5mm into sample
Counter Oscillation for Microscopy

Capsules for cosmetic applications in a gel
Tribology and Friction Coefficient

(TRIBOS = to rub)

Tribology: The science of interacting surfaces in relative motion

- Study and application of the principles of friction, lubrication, and wear

Friction Coefficient:

\[ \mu = \frac{F_F}{F_L} \]

- \( \mu \) is dimensionless
- Often f is used instead of \( \mu \)

Amontons Laws:

- \( F_F \) is independent on area
- \( F_F \sim F_L \)
Rheology is the science of deformation and flow of materials, with the determination of bulk material properties. Rheology characterizes material properties and inner friction. There is no influence of geometries (in laminar flow), and the gap is set.

Tribology is the science of interacting surfaces in relative motion, including friction, lubrication, and wear, with the determination of tribo-system properties. Tribology characterizes system properties and inter-surface friction. Geometries (or surface of geometries) are part of the sample, and the gap sets itself.
**Experimental Setup: Ball-on-three-plates**

**Rheometry**
- Normal Force $F_N$: 0.01 – 50 N
- Torque $M$: 0.1 µNm - 200 mNm
- Rot. speed $n$: $10^{-6}$ - 3000 rpm
- Min. angle osc.: 0.1µrad
- Deflection angle **and** torque control
- Oscillatory tests

**Tribometry**
- Normal Load $F_L$: 0.3 - 23 N ($F_{N,Tribo}$)
- Friction Force $F_F$: 0.01 - 44.5 N
- Sliding speed $s_v$: $10^{-8}$ - 1.41m/s
- Min. sliding distance osc.: $4.5 \cdot 10^{-10}$m
- Sliding distance **and** friction force control
- Oscillatory or reciprocating motion

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**Geometric Calculations**

P.Heyer, J.Läuger
Lubrication Science (2009)
Tribology on Cosmetic Creams

Sample 1
→ Intermediate break-away point but the resistance to motion keeps increasing.
→ Offers a continuous resistance to being spread, i.e. is less comfortable.

Sample 2
→ Highest force at break-away point, but, least resistance after that i.e. no control.

Sample 3
→ Break-away at higher sliding distance, but after that, slight resistance to continuation of motion.

- S3 is thicker than the other two. It is better in feel than S1 but does not flow as easily as S2
- Agrees well with results from sensory panel
Sample 1
→ offers very high resistance to motion, as indicated by the increase in the friction coefficient with increasing speed.

Sample 2
→ As observed in break-away torque tests, highest break-away torque, but it offers no significant resistance to motion. It flows relatively freely.

Sample 3
→ Has low resistance to initiation of motion and an intermediate resistance to acceleration or spreading.

- Based on sensory perception results, S3 is the most liked among the three
- Agrees well with results from sensory panel
Experimental Setup: Pins-on-disc

- Peltier Temperature control (-30°C - 200°C)
- Adjustable spring in upper geometry
Standard and advanced rheological as well as tribological methods can be of great help for developing cosmetic formulations:

- Flow behavior directly related to use of product
- Amplitude sweeps reveal rest structure and cross-over into yield and flow
- Non-linear testing for “fingerprinting” of different samples
- Frequency sweeps give indication on long time behavior → physical stability
- Temperature cycle test for distinguishing stable for unstable samples
- Optical techniques for gaining insight into the microstructure during flow
- Di-electric spectroscopy to “see” into nontransparent samples
- Tribology as a method to correlate frictional measurements with consumer feel

Conclusions