# RHEOLOGY – TEXTURE ANALYSIS: new keys for access to cosmetic formulation texture.

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## SUMMARY

In cosmetic formulations, texture plays a key role in ingredient choice and formulation optimization.

But texture parameters are often measured by sensorial analysis in the last stages of formulation development.

Rheology or texture analysis, used separately, has the benefit of characterizing the behavior of raw materials (e.g. polymers) and controlling and predicting the stability of formulations.

SEPPIC has developed rheology and texture analysis protocols to obtain a better understanding of the influence of raw materials on the cosmetic texture of formulations.

When used in combination, these two methodologies are complementary and provide useful data regarding the impact of raw material choice on all the development steps: manufacturing procedure, formulation stability, skin feeling.

## INTRODUCTION

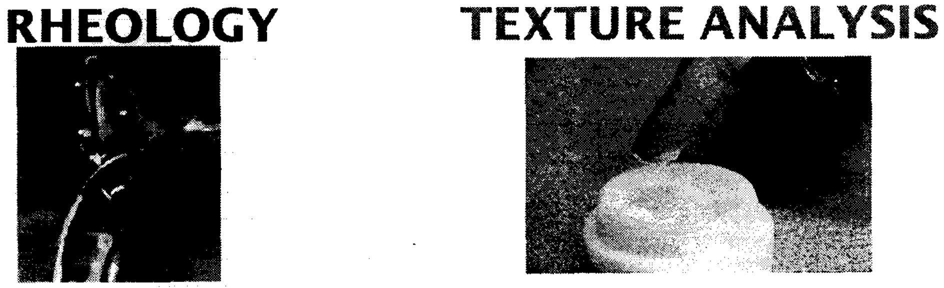
Texture and skin-feeling of cosmetic formulations are becoming fundamental parameters that strongly influence the consumer's choice when selecting a suitable cosmetic product. In addition to the biological activity (anti-age, soothing, etc.), the sensation procured during utilization of cosmetics is becoming a key criteria for consumers and consequently for laboratory formulation teams: parameters including consistency of the final product, ease of pick-up, good spreading properties, and avoidance of any sticky or greasy sensations need to be optimized.
Furthermore, all these texture criteria should be stable over time during the entire life of the cosmetic formulation and also reproducible at the production stage.
Controlling the texture parameters is very challenging. SEPPIC has developed methodologies to help formulators optimize their product development to enhance texture and skin feeling. Rheology and texture analysis, used in combination, are able to predict the texture of cosmetic products at early stages of product development and thus optimize development time.

Rheology and texture analysis study the reaction of the product under shear stress applied by different types of device and movement.
These methodologies can predict the behavior of the cosmetic formulation under actual conditions of use and at all stages of its lifetime (manufacture, packaging, storage, transport, use).

This article gives some examples of practical information obtained using these two techniques.

## MATERIALS AND METHODS

### Definition



**RHEOLOGY**

Science of the flow and the deformation of matter

**TEXTURE ANALYSIS**

Investigation of the mechanical properties of a product: resilience, consistency, adhesion, etc.

Figure 1 Definitions of the two methodologies

The application of these two techniques is of great interest for the consumer. For example, they provide information on how the product will pour out of a bottle or on its behavior when picked-up from a jar: elasticity, stringiness, adhesion, spreading on the skin, etc.

Both these techniques study the behavior of the product when subjected to shear stress; consequently, rheology and texture analysis can be used as models of real situations in which the cosmetic product is used. For example, if the goal is to predict the pick-up properties, the measurement is made directly in the container, the shear stress is applied by a high-speed translation movement with a low depth of penetration into the product, and the measurement is performed at room temperature. Conversely, to predict spreading behavior, it is preferable to work at about 32 °C, which is closer to skin temperature, and a medium-speed rotating movement is selected to apply the stress to a very small amount of product.

Furthermore, both methods have the advantage of generating very reliable and reproducible results. With a trained operator and standardized protocols, the standard deviation of the measured parameters is always below 10%.

**Materials**

- Rheometric measurements are made using a CARRIMED CSL500® controlled stress rheometer from *WATERS-TA INSTRUMENTS* (Figure 2).

As shown in figure 2, the rheometer applies the shear stress by a rotating movement on the product and measures the resulting stress as a function of the shear rate (unit = pascal Pa)

- Texture analysis experiments are performed using a TEC® texture analyzer from *Jean LAMY* (Figure 3). As shown in figure 3, the texture analyzer controls a translation movement of the probe: down into the product and back up, recording the resulting force curve (unit = newton, N) during the displacement.

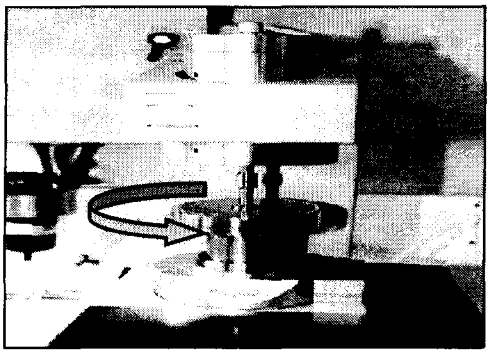


Figure 2 - CARRIMED CSL 500® rheometer

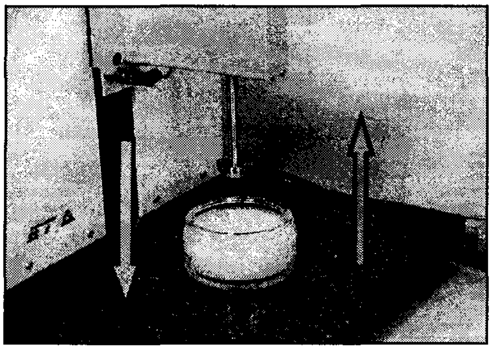


Figure 3 - TEC® texture analyzer

Depending on the movement applied to generate the stress, these two devices are complementary, and representative of the major movements applied to the cosmetic product during manufacturing, transfer, packaging, and use (for example, translation movement for pick-up, rotating movement for spreading).

**Methods**

- ***Rheology***

Two main types of measurement are made using the rheometer: flow experiments and oscillatory experiments. The two experiments give complementary data on the flow properties of the product and on the internal structure of the product at rest.

- During flow experiments, an increasing shear stress is applied to the product for two minutes and then a decreasing shear stress is applied. The resulting shear stress is monitored as a function of the shear rate. One of the most useful ways to express the measurements is to monitor the viscosity as a function of the shear rate, which provides information on product flow characteristics.

  For instance, a product which shows decreasing viscosity with increasing shear rate (i.e shear thinning behavior) facilitates bottle filling and also improves the pouring step. For the consumer, such behavior is also commonly associated with good spreading properties.

- The purpose of oscillatory experiments is to apply very low periodic shear stress for a long time to investigate the internal structure of the product at rest.

  The resulting parameters are the storage modulus G' (expressed in Pa), which represents the elastic character of the product, and the loss modulus G" (expressed in Pa), which represents the viscous character of the product.

  The ratio **G'/G"** represents the tendency of the product to be more elastic (able to recover from deformation).

  A predominant elastic character is typically related to a network structure and consequently helps to stabilize the product.

- ***Texture analysis***

The texture profile follows the behavior of the product on compression and/or on stretching.

- Monitoring the force during compression gives information about the consistency (or firmness) of the product: the higher the force required to achieve the defined movement, the more consistent the product.

- Monitoring the force during stretching gives information about the adhesion properties of the product: how the product stretches (stringy effect or elastic return) and the quantity of product which remains on the probe. All these parameters give indications on the ease of pick-up.

These two methods are fully representative of the stresses of manufacture, packaging, transfer, storage, and use.

## RESULTS AND DISCUSSION

**☆First example: How does the choice of ingredients govern the texture?**
***This first study shows how polymer selection influences the ease of pick-up of gels and cream-gels in relation to their adhesion properties.***

Five polymers were studied comparatively in aqueous gel and in cream-gel at equivalent formulation viscosity: 20,000 mPa.s and 50,000mPa.s (Brookfield LVT, speed 6, spindle 4 at 25 °C)

| | | |
|---|---|---|
| Aqueous gel formula : | Polymer | dose required for viscosity |
| | Water | qs 100% |
| Cream-gel formula : | Polymer | dose required for viscosity |
| | Cetearyl Ethyl Hexanoate | 15% |
| | Water | qs 100% |

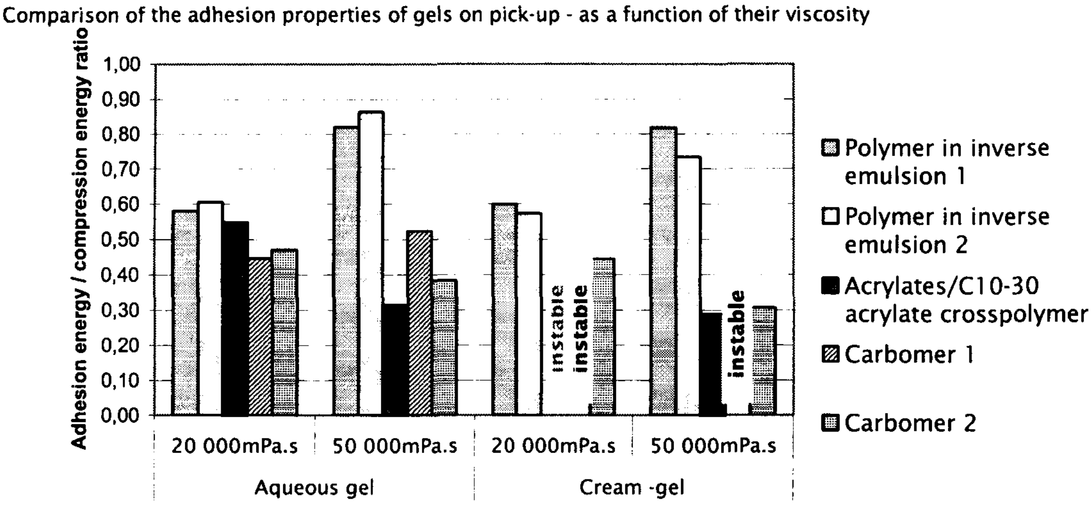
The texture profile of each formulation was determined by a cyclic measurement protocol: compression followed by stretching with a hemispherical probe.
Two parameters were determined:

- compression energy: energy necessary to push the probe into the sample (unit= mJ)
- adhesion energy: energy necessary to pull the probe out of the sample (unit= mJ)

To avoid the effect of firmness differences between the gels, despite viscosity adjustment, the adhesion of the product to the probe was expressed as the adhesion energy/compression energy ratio. The higher this ratio, the more the product stays on the probe.

The results show (figure 4) that texture profiles of cream-gels are similar to texture profiles of aqueous gels, which means that the polymer clearly governs the texture behavior of cream-gels.
An additional study has demonstrated that the quantity of oil incorporated and the nature of the oils have very little influence on the adhesion properties of the cream-gel.

The adhesion of gels and cream-gels on the probe is viscosity-dependent, and different behaviors can be observed depending on polymer tested.
With the two polymers in inverse emulsion, the adhesion increases similarly with the viscosity of the gel or cream-gel. The ease of pick-up of these polymers was confirmed by a trained sensorial panel with the products in jar packaging.
In contrast to polymers in inverse emulsion, other polymers maintain the same adhesion properties as the viscosity increases (Acrylate/C10-30 acrylate copolymer) or, worse, show decreasing adhesion as the viscosity increases (carbomer 1 or 2). For these polymers, the sensorial evaluation demonstrated good pick-up for the 20,000 mPa.s gels but poor adhesion for the 50,000 mPa.s gels.
With these types of formulation, the adhesion property measured by texture analysis is well correlated with the ease of pick-up determined by sensorial analysis.
These results demonstrate how texture analysis can help to select the optimum polymer for formulating firm and viscous gels or cream-gels which still have good pick-up characteristics.



*Polymer in inverse emulsion 1: Sodium acrylate/ Acryloyldimethyl taurate copolymer/ Isohexadecane/ Polysorbate 60 (SIMULGEL EG - SEPPIC)*
*Polymer in inverse emulsion 2: Hydroxyethyl acrylate/ Sodium acryloyldimethyl taurate copolymer / Squalane/ Polysorbate 60 (SIMULGEL NS - SEPPIC)*

Figure 4 – Comparative adhesion of gels

**☆Second example: How can the manufacturing process influence the texture?**
***In this second example, we studied the texture profile of a basic emulsion made with different procedures using a pre-defined mixer.***

Emulsion formula:

| | |
|---|---|
| Cetearyl alcohol/Cetearyl glucoside (*Montanov 68 -SEPPIC*) | 5% |
| Paraffin oil | 20% |

The emulsion is made by emulsification for four minutes with a rotor/stator turbine (SILVERSON®) and then cooling while stirring with an anchor blade.
The emulsifier can be added either to the hot water phase or to the hot oil phase. The texture of the emulsion is then monitored over time with the texture analyzer using a compression protocol. The maximum compression force (unit=N), i.e. the force necessary to push the probe into the sample to a given depth at a fixed speed, is well correlated with the consistency of the emulsion as evaluated by consumers picking up the product from a jar. The higher the maximum compression force, the more consistent the emulsion.

With such firm creams, texture analysis is very helpful for monitoring the real “consistency” of the emulsion, because the compression movement is not sensitive to the sliding effect. With rotational devices such as the Brookfield viscosimeter, there is a strong sliding effect: there is absolutely no correlation between the measured viscosity and the apparent consistency of the emulsion (underestimated).

The compression profiles shown in figure 5 and the maximum compression forces given in the summary table 6 show that the consistency of the emulsion is similar whatever phase the emulsifier is introduced in; moreover, both emulsions have very good stability over time.

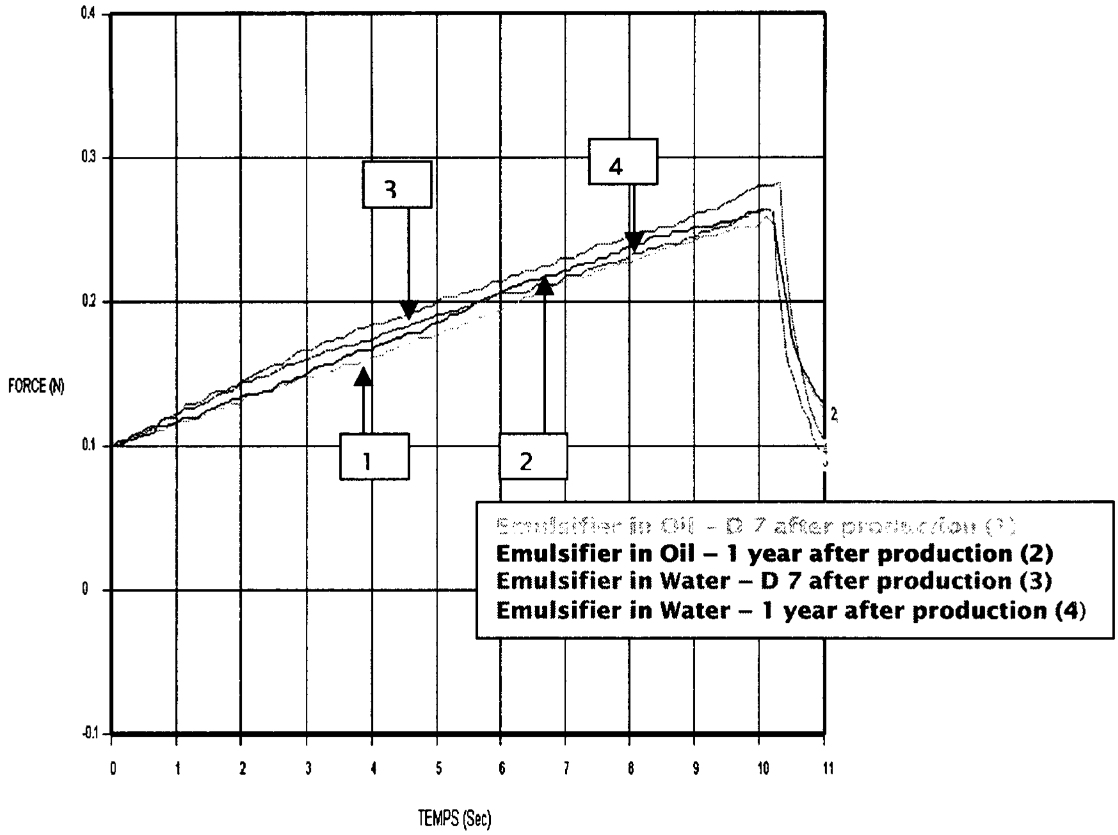


Figure 5 - Compression profile of the emulsion over time

| | Maximum compression force (N) (standard error) | |
|---|---|---|
| Procedure | Emulsifier in oil | Emulsifier in water |
| D7 after production | 0.25 N (± 0.01) | 0.28 N (± 0.015) |
| 1 year after production | 0.26 N (± 0.01) | 0.26 N (± 0.01) |

Table 6

Such glucolipid emulsifiers consequently provide extensive freedom of formulation.

This type of study could also be performed to examine the influence of scale-up on the texture of the emulsion, and provides very useful data for adjusting the procedure when stirring devices are non-homothetic.

**☆Third example: How can the stability of the texture be optimized?**
Cream-gels lead to new textures and have an increasing share of the cosmetics market. However, many questions are often left unanswered:
What is the mechanism of stabilization of a cream-gel? How can the very good stability of this outstanding texture over time be explained?
The viscoelasticity profile of polymers illustrates their ability to stabilize oil phases without any additional surfactant.

The behavior of an aqueous gel and a cream-gel was studied by oscillatory experiments: shear stress sweep (frequency 1 Hz), frequency sweep (from 0.1 to 10 Hz), temperature sweep (from + 5 °C to 80 °C) to monitor the G'/G" ratio.

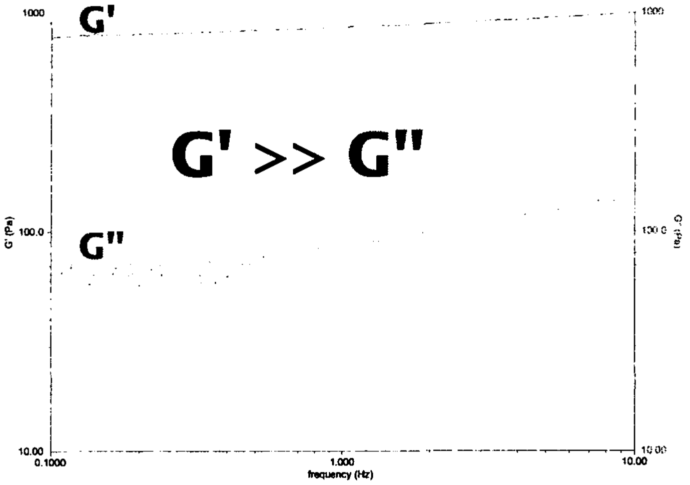
Aqueous gel formula:

| | |
|---|---|
| Polymer<br>*Hydroxyethyl acrylate/Sodium acryloyldimethyl taurate copolymer /Squalane/ Polysorbate 60 (SIMULGEL NS – SEPPIC)* | 3% |
| Water | qs 100% |

Cream-gel formula:

| | |
|---|---|
| Polymer (the same as in the aqueous gel) | 3% |
| Caprylic/capric triglyceride | 10% |
| Water | qs 100% |

As shown in figure 7, the cream-gel had a strong elastic character, stable over the whole frequency range. Further investigation by temperature sweep also confirmed this strong elastic character over the range from 5 °C to 80 °C, which indicates a strong and stable polymeric network in the aqueous phase.



Frequency sweep from 0.1 to 10 Hz

Figure 7 – Frequency sweep of the cream gel

The comparison between the aqueous gel and the cream gel in the same shear stress sweep (Table 8) shows that the elastic character was enhanced in the presence of the oil, which is in perfect correlation with the stability of the cream-gel over time, even after prolonged storage at 50 °C (no exudation of the oil phase).

| Elastic character | G'/G"<br>Mean ratio<br>Linear region |
|---|---|
| Aqueous gel | 6.4 |
| Gel-cream | 9 |

Table 8 – Comparison of elastic character of aqueous gel and cream-gel

The presence of the oil phase reinforces the elasticity of the polymer, which illustrates the stabilization of the oil droplets in the polymeric network.

Further experiments indicated that this phenomena is not dependent on the nature of the oil: polar or nonpolar oils could be easily stabilized in the polymeric network.

## CONCLUSIONS

These three examples illustrate some applications of rheology and texture analysis. The combined use of these two techniques could be very helpful in formulation studies thanks to the complementary movements and types of data obtained.

These two technical tools help to obtain a better understanding of which ingredient has the leading role in the formulation and provide technical data for selecting suitable ingredients for optimizing the manufacturing procedure, the stability of the formulation, and the skin feeling.

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