Application Note
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Rheological characterization of tooth paste

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- Platte / Platte measuring geometry
- Plastic flow behaviour
- Yield point
- Linear viscoelastic range

What is actually in the tooth paste?

Tooth pastes are viscoelastic products well known in every day life. Its main components are abrasives, moisture agents, binding agents, surfactants, sweeteners, preservatives, artificial coloring, flavors and special active ingredients. Hydroxides, carbonates, phosphates or silicates are often used as abrasives to support the mechanical cleaning effect of the tooth brush. Moisturising agents like glycerin, sorbit, lignite or polyethylenglycols prevent a drying out of the tooth paste. At the same time, they increase the low temperature stability and have a texture building effect.

Binding and thickening agents give the tooth paste its desired texture and prevent a phase separation between fluid and solid. Hydrocolloids like Alginate, Carrageenan, Methylcellulose or Xanthan are mainly used as binding agents. A frequently used thickening agent is high-disperse silicium dioxide or Bentonite.

Surfactants decrease the surface tension thus improving the even distribution of the tooth paste in the mouth. In dental care, only tasteless and non-toxic anion surfactants like sodium lauryl sulfate, or coconut aliphatic monoglyceride sulfonate are suitable Sweeteners as well as the flavors serve as taste corrective. Preservatives are necessary as protection from microbial decomposition.

Colors and pigments are finally used for the coloring of striped tooth pastes. Besides this, there may be active ingredients as protection from caries or for the care of the gums or painsensitive teeth.

How is tooth paste produced?

The production of tooth paste is either done batch-wise in a vacuum mixer or in continuous production processes. In the continuous production, 5 different process steps can be differentiated: gel production, metering of liquid components, metering of solids, mixing process as well as homogenization and ventilation.

Assessment of tooth pastes in the product development and application

Like other body care products tooth paste has to remain stable over long periods of time even at changing temperatures. In the product development the products are therefore stored for several months at different temperatures and then different quality criteria are assessed like texture, color, taste, liquid separation. Here, rheological tests can be a valuable help for the quantitative determination of some product properties. In many cases rheology can already be used during the development of new product formulas thus leading to a cost reduction by avoiding cost-intensive storage tests. However, the viscous and elastic properties of tooth paste are not only of interest for the development of a formula but also for the filling process in production. Here stable products are desired which can easily be pressed out of the tube without showing any signs of phase separation or string forming.
The extruded portion of tooth paste on the tooth brush should on one hand have a distinct shape and on the other hand not roll from the tooth brush into the wash basin. A liquid tooth paste should well penetrate properly between the bristles of the tooth brush and moisten them evenly. Finally, the packaging of the product has to be considered. Is the tooth paste offered in the classic tube, in a standing tube or in a dispenser? Last not least the consumer target group has certain expectations regarding the product properties. A test series showed that children for example prefer tooth pastes of a firmer texture (like chewing gum) over soft tooth pastes.

Fig. 1: Modular Advanced Rheometer System HAAKE MARS.

Which rheological tests can be performed on tooth paste?

Tooth pastes are liquids which cannot be poured. They show, as the name implies, a paste-like texture. The flow behavior of those pastes can be described by flow or viscosity curves. For this, usually a cone/plate or a plate/plate measuring geometry is used. For the following measurements an air-beared rheometer is required like a HAAKE Rheostress or the HAAKE MARS (Fig. 1). The use of a plate/plate measuring geometry with a diameter of 35 mm and a measuring gap of 1 mm are recommended.

Tooth pastes show a plastic flow behavior. The beginning of a flow curve is usually recorded with shear stress controlled measurements allowing a very good determination of the product properties at very small shear rates. Also the yield point τ_0 of tooth pastes can be precisely determined and is reproducible with shear stress ramps. Creep and recovery tests allow the characterization of the viscoelastic properties of tooth pastes at very low speeds (i.e. at slow processes). With the determination of the zero viscosity η_0 in those tests, conclusions may be drawn regarding the storage stability as well as the structure recovery after shearing.

Oscillatory tests give information on the viscoelastic properties at fast processes. Here the rheological parameter storage modulus G', loss modulus G", phase shift angle δ as well as the complex viscosity η^* are determined. The firmer the texture of the tooth paste the higher are the values for η^*. The ratio between elastic and viscous portions in the substance is described by the loss angle tan δ. Are the values for tan δ smaller than one, the elastic component dominates. For gel tooth pastes G' is always higher than G". The stronger the gel structure is the higher are the values for G'. Such a tooth paste is harder to press out of the tube than a „softer" tooth paste. The extruded portion of tooth paste retains its shape on the tooth brush and does not penetrate between the bristles.

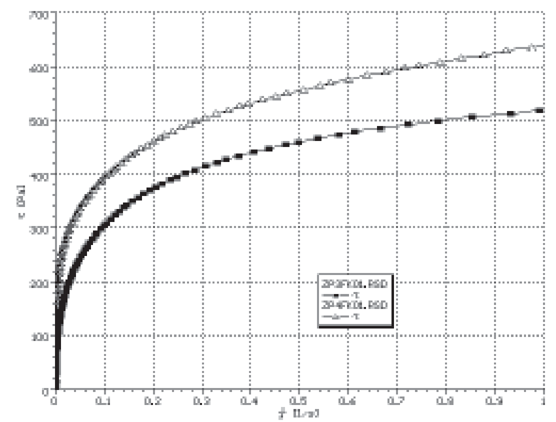

Fig.2: Flow curves of two different tooth pastes at 23 °C

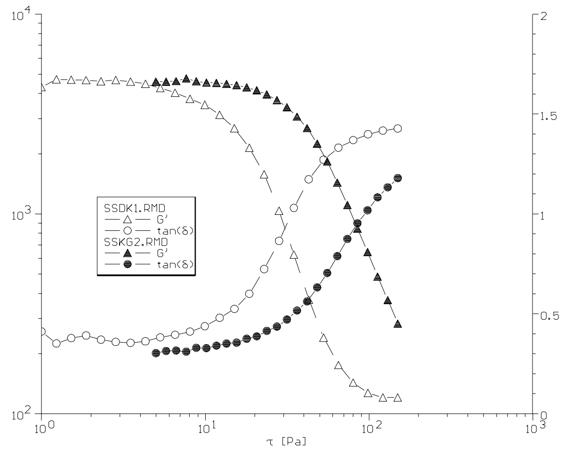

Fig3: Amplitude sweep on two different tooth pastes at 23 °C

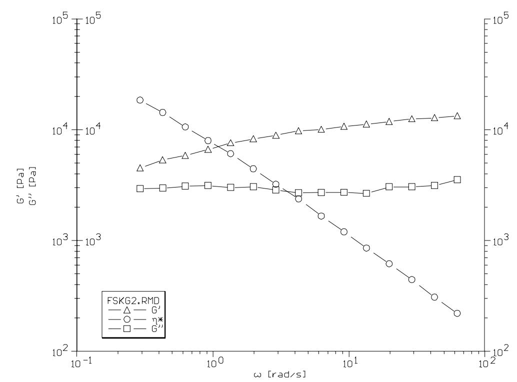

Fig.4: Frequency sweep of a gel tooth paste at 23 °C

Amplitude sweeps, also called stress sweeps can be used as a fast test method to characterize the product stability. A very stable product will show a wide linear viscoelastic range whereas products with a weak, sensitive structure will already be destroyed when subject to small stresses.

Typical measuring resuluts

Fig. 2 shows the beginning of the flow curves for two different tooth pastes, measured with a shear stress ramp. The plastic flow behavior is clearly visible, i.e. the tooth pastes only start to flow beyond a certain shear stress.

In Fig. 3 an oscillation-amplitude-sweep is shown measured at a frequency of 1 Hz. Below a certain critical amplitude, both tooth pastes behave like elastic solids. The substance is then in the so-called linear viscoelastic range in which force and deformation are proportional. At higher oscillation amplitudes the structure of the materials will be destroyed irreversibly. Here an increase of tan δ to values higher than 1 can be observed.

The storage modules of the tested tooth pastes have comparable values of around 4500 Pa. Nevertheless, the tooth paste 1 (open symbols) was found to be softer when tested. For this product, a tooth paste for sensitive teeth, tan δ is higher, i.e. the viscous portion is higher than for product 2 (filled symbols), a tooth gel for children. The gel shows a wider linear viscoelastic range than tooth paste 1. As expected a weaker structure will be destroyed at lower shear stress values.

In Fig. 4 a typical frequency sweep on a gel tooth paste is illustrated. The behavior at high frequencies corresponds to the stress to which the product was subject for only a short time. The response of the product to longer lasting stresses can be derived from the behavior at small frequencies. The frequency dependency of the storage modulus, loss modulus and complex viscosity is in many cases not very distinct; G' and G" often run parallel to the x-axis. At the gel tooth paste G' is higher than G" over the complete frequency range tested, i.e. the product reacts on stress like a viscoelastic solid. At smaller frequencies the modules come closer. At very small frequencies a point may be reached where G' and G" have the same value.

At tooth paste 1 such a crossover point (see Fig. 5) can be observed at a frequency of about 1 rad s^{-1}. At smaller frequencies the viscous portion prevails, i.e. the product behaves like a viscoelastic liquid. The tooth paste flows under the influence of gravity; it is therefore especially suited for standing tubes. Correspondingly, a slow diffluence of the extruded tooth paste portion on the tooth brush and a penetration between the bristles can be observed. This behavior is stronger in case of liquid tooth pastes.

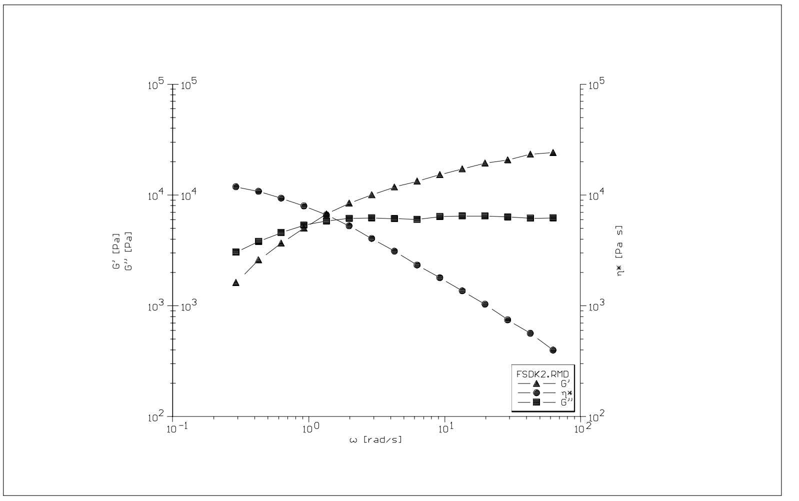

Fig.5: Frequency sweep on a tooth paste for sensitive teeth at 23 °C

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