APPLICATION**NOTE**

Evaluating Product Delivery Characteristics from a Bottle, Tube or Spray Pack

Introduction

Many consumer products are packaged in tubes or bottles where product application involves pumping the product through a nozzle. Such products tend to be shear-thinning products where the viscosity drops during the extrusion process due to the increasing shear rate, and then recovers on exiting the nozzle as the shear rate is reduced. The shear rate encountered during this process is related to the radius, *r* of the orifice and the volumetric flow rate *Q* by the following expression:

$$\dot{\gamma} = \frac{Q}{\pi r^3}\left[3 + \frac{1}{n}\right]$$

The parameter *n* is the power law index, which is one for a Newtonian liquid and between 0-1 for a non-Newtonian fluid. This value can be readily attained from a variable shear rate test by fitting a power law model to the resultant data.

By measuring the volumetric flow rate (volume dispensed in given time) and the internal radius of the orifice, it is possible to estimate the shear rate encountered during the extrusion process. This value can then be used in a shear rate loop test, which ramps the shear rate from a low value (prior to extrusion) to the shear rate of interest and back down again. Such a test allows one to determine the amount of stress required to extrude, spray, or pour the product, and the extent of viscosity recovery following this process, which will ultimately determine the thickness of the product in use. This can be quantified by measuring the enclosed area between the up and down curves.

Experimental

- The extrusion properties of toothpaste and a body lotion were evaluated and compared under process relevant conditions.
- Rotational rheometer measurements were made using a Kinexus rotational rheometer with a Peltier plate cartridge and a roughened parallel plate measuring system[1], and using standard pre-configured sequences in the rSpace software.
- A standard loading sequence was used to ensure that the sample was subject to a consistent and controllable loading protocol.
- All rheology measurements were performed at 25°C.
- The relevant extrusion shear rates were automatically calculated as part of the test sequence using inputted values of extruded volume, extrusion time and aperture radius. The test was programmed to use this calculated value as the maximum shear rate in an ´up then down` shear rate ramp.
- The measured stress at the calculated shear rate and the area between the ‚up and down' curves were both reported. The former conveying the stress requirement for extrusion and the latter the extent of viscosity recovery (thixotropy) following extrusion.

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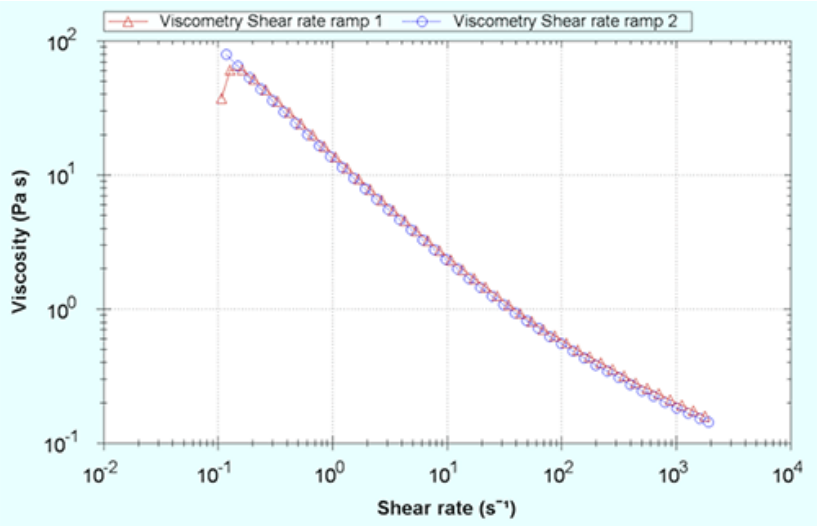
Results and Discussion

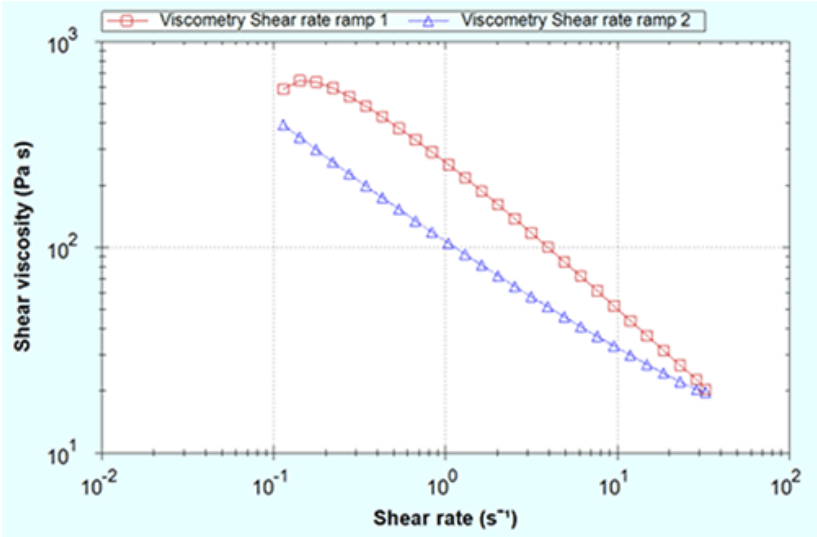
The automatic calculator determined the shear rate to be approximately 2000 s^{-1} for the body lotion and therefore automatically performed a shear rate ramp between 0.1 s^{-1} and 2000 s^{-1} and back up again. The results shown in Figure 1 show that the viscosity at the representative shear rate is 0.143 Pas and would require an applied shear stress (associated with squeezing) of 274 Pa to extrude the product. Furthermore, the area analysis yields a value of just 18 suggesting that the product would recover its viscosity rapidly following extrusion and is thus non-thixotropic.

For the toothpaste shown in Figure 2, the extrusion shear rate was calculated to be approximately 33 s^{-1}. The viscosity at the representative shear rate is 19.73 Pas and would require an applied shear stress of 643 Pa to extrude the product. This material appears to be thixotropic since the area between the upper and lower curves has a much higher value of 775.

The slight peak in viscosity observed at the start of the test for both samples is the result of elastic deformation on start-up and would be expected to be seen for both these products as they both have yield stresses [1].



1 Flow curves for a body lotion sheared up to their extrusion shear rate and back down again



2 Flow curves for toothpaste sheared up to its extrusion shear rate and back down again

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Conclusions

A shear rate value relating to the extrusion of a skin cream from a bottle and toothpaste from a tube were calculated and automatically inputted into a shear rate ramp ´up and down` test. The results showed that a shear stress of 274 Pa would be needed to dispense the skin cream at the desired rate and 643 Pa for the toothpaste. Determination of the area between the up and down curves showed that the body lotion was non-thixotropic while the toothpaste showed thixotropic behavior.

References

[1] White Paper – Understanding Yield Stress Measurements, NETZSCH

[1]Please note

that testing is recommended to undertaken with cone and plate or parallel plate geometry - with the latter being preferred for dispersions and emulsions with large particle sizes. Such material types may also require the use of serrated or roughened geometries to avoid artefacts relating to slippage at the geometry surface.

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