Methodology and Measurements of Extensional Rheology by Contraction and Squeeze Flow

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ABSTRACT

A technique based on contraction flow through a nozzle for extensional viscosity measurements has been evaluated for semi-solid materials. Four well-characterized test fluids were used for the evaluation and the results were compared with extensional viscosities obtained with squeeze flow and filament stretching. The comparison showed that the methods are comparable and well suited for shear-thinning, semi-solid materials such as foods, cosmetics and various pastes.

INTRODUCTION

Extensional flows occur at abrupt changes in process equipment like e.g. nozzles and pipe entrances. It is often more difficult to measure the extensional properties, expressed through the extensional viscosity than the shear viscosity. One major reason to this is that it is difficult to generate homogeneous extensional flows, particularly for low-viscosity fluids.

The deformation of the fluid is often expressed as the Hencky strain:

$$e_H = \ln \frac{H}{H_0} \qquad (1)$$

For fluids, at infinitely low deformation rate, Trouton's law says that the extensional viscosity is three times the shear viscosity. This relation can be used to correlate the extensional viscosity to the shear viscosity[1].

Contraction flow

A technique for measurements of the transient extensional viscosity has been developed by Leif Bohlin (Reologen i Lund, Öved, Sweden). It is based on contraction flow through a nozzle. The nozzle is designed to give a constant strain rate in the sample at a specific volumetric flow rate. The nozzle rests on a load cell that measures the force on the nozzle.

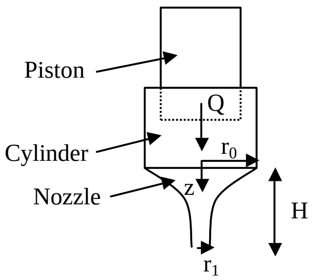

Figure 1. The principle of the contraction flow method.

If a power-law model is assumed the extension rate can be calculated as[2-3]:

$$\dot{e} = -2 \frac{3n+1}{n+1} \frac{Q}{pr^3} \frac{dr}{dz} \qquad (2)$$

The equation above can be solved for the radius r(z) as[2-3]:

$$r(z) = \frac{r_0}{\sqrt{\frac{z}{H}\left(\frac{r_0^2}{r_1^2} - 1\right) + 1}} \qquad (3)$$

Now the constant strain rate in the nozzle can be given as[2-3]:

$$e = \frac{3n+1}{n+1}\frac{Q}{p}\frac{r_1^{-2} - r_0^{-2}}{H} \qquad (4)$$

The total Hencky strain over the nozzle is given by[2-3]:

$$e_H = \frac{3n+1}{n+1}\ln\left(\frac{r_0^2}{r_1^2}\right) \qquad (5)$$

It is not always possible to avoid interaction of shear forces in the nozzle. Over the entire nozzle the shear stress is given by[2-3]:

$$s_{shear} = \frac{4H\left(3+\frac{1}{n}\right)^n\left(\frac{K}{p}\right)^n Q^n\left(\frac{1}{r_0^{3n+1}}\right)\left(\left(\frac{r_0^2}{r_1^2}\right)^{\frac{3n+3}{2}} - 1\right)}{(3n+3)\left(\frac{r_0^2}{r_1^2} - 1\right)} \qquad (6)$$

where n and K are power-law constants. When K and n are known, the extensional stress can be calculated as[2-3]:

$$s_e = s_{zz} - s_{rr} = \frac{F}{p \cdot r_0^2} \qquad (7)$$

The extensional viscosity is then calculated as[4]:

$$h_e = \frac{s_e}{\dot{e}} \qquad (8)$$

<u>Squeeze flow</u>

When a sample is squeezed between two parallel plates a biaxial extensional flow is generated in the sample. The force on one of the plates can be measured and together with the gap between the plates give a value of the biaxial strain rate. To avoid shear contributions at the plates some lubricant is needed[1]. Often the plates are covered with Teflon to minimize shear contribution, however, it has been proven that Teflon does not totally avoid shear contributions[5]. For semi-solid materials, squeeze flow is a simple and useful method[6].

To achieve a constant strain rate in the sample, the upper plate is pressed down with an exponentially decreasing speed.

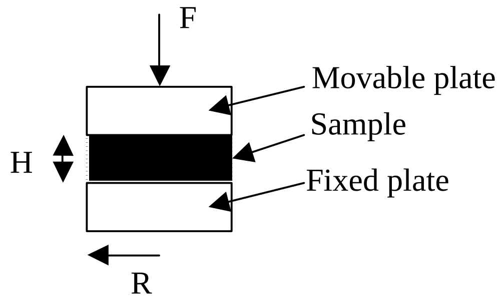

Figure 2. The principle of squeeze flow.

In squeeze flow, the biaxial Hencky strain is given by[1]:

$$e_{Hb} = -\ln\left(\frac{H}{H_0}\right) \qquad (9)$$

The strain rate is calculated as[1]:

$$e = -\frac{1}{2H}\frac{dH}{dt} \qquad (10)$$

The flow is driven by a normal stress difference[1]:

$$s_{11} - s_{33} = \frac{F}{p \cdot R^2} \qquad (11)$$

The biaxial extensional viscosity can now be expressed as[1]:

$$h_b = \frac{s_{11} - s_{33}}{e} \qquad (12)$$

<u>Strain hardening</u>

The phenomenon strain hardening occurs when the material has reached a limit for how much it can be extended. The viscosity then begins to increase with a faster strain rate than before. If a power-law model is assumed, the stress dependence of the Hencky strain can be modelled by[7]:

$$s = K \cdot e_H^n \qquad (13)$$

Strain hardening means that n makes an abrupt upturn at a specific extensional strain.

MATERIALS AND METHODS

Four different test fluids were used for the measurements, PDMS, syrup, SUA-1 and SUP-5. PDMS, polydimetylsiloxane (Infra scientific, Stroud, Great Britain) is a shear thinning, high viscosity fluid. Syrup (Danisco sugar, Arlöv, Sweden) is a Newtonian fluid and SUA-1 and SUP-5 (University of Strathclyde, Glasgow, Great Britain) are two shear-thinning polybutulene solutions.

First the shear viscosity was measured to make it possible to correlate the shear viscosity to the extensional viscosity with Trouton’s number on the Newtonian plateau. Then the contraction flow method was used to measure the extensional viscosity at different extension rates. The biaxial extensional viscosity was measured in squeeze flow, a method that also made it easy to see if the material showed strain hardening or not. A sample of SUP-5 was sent to DTU in Lyngby, Denmark where measurements were conducted with a filament stretching method.

RESULTS AND DISCUSSION

<u>Contraction flow</u>

Trouton's number was calculated for the measurements made on the Newtonian plateau. For PDMS, the Trouton number became 14 instead of the expected three. This means that the results from the contraction flow method not are exactly comparable to the theoretical values. The reproducibility of the method is good and the method is still a useful technique for determining the extensional viscosity.

The results showed that for the Newtonian fluid syrup, the shear stress contributions were much larger than the total extensional stress measured. This also shows that the contraction flow method is not suitable for Newtonian fluids since the contribution of shear forces and friction is larger than for fluids showing a shear-thinning behaviour due to the high shear contribution dominating the response.

<u>Squeeze flow</u>

The reproducibility was good for all the conducted squeeze flow measurements. The results clearly showed the strain hardening behaviour of the fluids.

<u>Comparison between different techniques</u>

The comparison of the contraction flow method with filament stretching and squeeze flow showed that these methods are comparable, although they do not match each other completely. This is shown for SUP-5 in Fig. 3. The biaxial extensional viscosity obtained from squeeze flow is twice the uniaxial extensional viscosity given by contraction flow and filament stretching. PDMS showed similar trends when comparing contraction and squeeze flow. The measured extensional viscosities are all transient due to the relatively low Hencky strain.

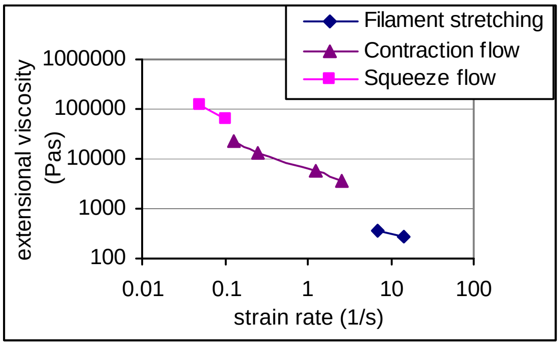

Figure 3. Comparison between the three different methods for SUP-5 at a Hencky strain of 3.07.

CONCLUSIONS

The extensional viscosity given by the contraction flow method did not completely comply with theory when related to the shear viscosity by means of the Trouton number. Since the reproducibility in most cases was good, the contraction flow method is still a reliable technique for extensional viscosity measurements on shear-thinning semi-solid fluids.

The study showed that the method is unsuitable for Newtonian fluids since the shear stress contributions are larger than the total extensional stress measured.

The squeeze flow technique is simple to use and gives a good reproducibility. This method also shows when a fluid shows strain hardening. Some disadvantages with the method are that it is difficult to avoid shear contribution by friction at the plates.

The comparison of the contraction flow method to squeeze flow and filament stretching showed a difference in absolute numbers for the extensional viscosity, though similar behaviour suggesting that the methods are comparable.

ACKNOWLEDGEMENTS

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