Contraction flow measurements of extensional properties

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ABSTRACT

The extensional flow properties of semi-solid materials such as viscoelastic foods can be measured in contraction flow. The sample is fed through a nozzle, specially designed to give a constant extension rate. The transient stress is measured and the extensional viscosity calculated. The contribution of shear to the measured stress was shown to be negligible for shear-thinning materials, whereas the measured stress of a Newtonian material was significantly influenced by shear. Since most semi-solid foods are shear-thinning, the method is well suited for measurements of extensional flow properties of such materials.

INTRODUCTION

Processing of viscoelastis fluids involves a significant amount of extensional flow, which has an impact on both the processing technology and on the final properties of the product. Many food systems are non-Newtonian elastic liquids and any change in geometry during the process generates a flow with an extensional component. In particular, a flow through a sudden contraction, or out of an orifice leads to a flow that cannot be described solely by the shear viscosity, η. The extensional viscosity, η_E, may be significantly higher than η and can behave in a completely different manner. A shear-thinning solution of a synthetic polymer e.g. often exhibits a tension-thickening extensional viscosity[1].

Despite the documented influence of η_E in processing, it is seldom measured due to experimental difficulties. There is commercial equipment available to measure the extensional properties of dilute solutions and there are several publications on the extensional properties of polymer melts[2-4]. Neither of these methods are suitable for semi-solid foods, which have a viscosity significantly higher than dilute solutions, but are not solid enough for the Meissner-rheometer[5]. Bohlin Reologi AB has developed a new test method suitable for semi-solid foods where the sample is subjected to extensional flow in a contraction flow geometry[6]. The transient stress is measured under constant extension rate through a specially designed contraction nozzle. This is followed by stress-relaxation measurements after cessation of flow. The method has been used for measurements of the extensional rheology of dough[6, 7] and is now used for studies of other semi-solid foods such as dairy products.

The present paper describes the contraction flow method and demonstrates its applicability to different types of materials.

THEORY

Measurements of uniaxial extensional flow were performed in contraction flow using the experimental set-up shown in Figure 1. The downward thrust of the piston was obtained using a linear guide unit driven by a stepper motor. The actual contraction flow device comprised a feeding piston, a cylindrical sample cell, and a contraction nozzle. The nozzle rested on a load cell with a central annulus, while the sample holder rested on an outer frame. This design allowed the forces on the nozzle alone to be measured by the load cell, and not the spurious friction forces between the piston and the wall of the sample tube.

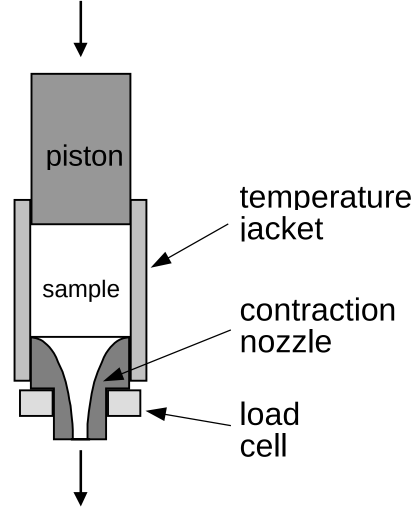

Figure 1. The principle of contraction flow measurements.

The contraction nozzle was designed to give an extensional strain rate, ε, which was constant throughout the length of the nozzle at a constant displacement speed. Figure 2 is an illustration of the contraction nozzle. The radius at the nozzle inlet is r_0, the radius at the nozzle outlet is r_1, the radius at z is $r_{(z)}$, Q is the volumetric flow rate and H is the length of the nozzle. The extensional strain rate, ε, at the centre of an axi-symmetric flow can be expressed as[8]:

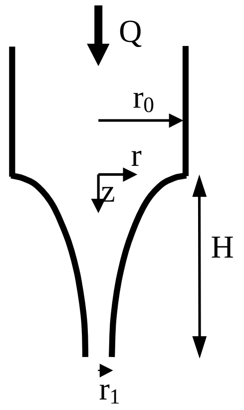

Figure 2. The contraction nozzle.

$$\varepsilon = -2\frac{3n+1}{n+1}\frac{Q}{\pi r^3}\frac{dr}{dz} \qquad (1)$$

where n is the power-law index for shear flow. Equation 1 is a first order differential equation with the solution for the radius r(z) calculable as[6]:

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

The constant strain rate in the nozzle is

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

and the total Hencky strain ε_H over the nozzle is

$$\varepsilon_H = \frac{3n+1}{n+1}\ln\left(\frac{r_0^2}{r_1^2}\right) \qquad (4)$$

The extensional viscosity is defined by

$$\eta_E = \frac{\sigma_{zz}-\sigma_{rr}}{\varepsilon} \qquad (5)$$

where σ_{zz} -σ_{rr} is the normal stress difference, which is calculated from

$$\sigma_{meas} = \sigma_{zz} - \sigma_{rr} = \frac{F_t}{\pi r_0^2} \tag{6}$$

F_t is the total force measured by the load cell (see figure 1).

Influence of shear

The relative importance of shear flow in the nozzle can be calculated from the pressure drop over the nozzle for the case of shear flow only. If we assume a power-law liquid

$$\sigma = K\dot{\gamma}^n \tag{7}$$

we obtain the volumetric flow rate Q for tube flow as[9]:

$$Q = \frac{\pi}{K} R^{3+\frac{1}{n}} p^{\frac{1}{n}} \frac{2^{\frac{1}{n}}}{3+\frac{1}{n}} \tag{8}$$

where p is the pressure drop per unit length of a tube segment with radius R. By integrating equation 8 over the height H of the nozzle, using the expression for the radius in equation 2 we obtain the total pressure drop, σ_{shear}, over the nozzle

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

(9)

MATERIALS AND METHODS

Materials

The samples tested were a PDMS material from Infra Scientific (Albertslund, Denmark) and different fermented milk products. The PDMS putty is viscoelastic with a G'=G"=22.7 kPa at 0.7 Hz at 25°C.

The fermented milk products were traditional Swedish products: “filmjölk” and “långfil” containing 3% fat. The third product was a stirred yoghurt. The “långfil” has a very long and thready texture with visible elongational strength.

Methods

The contraction flow instrument was a prototype from Bohlin Reologi AB (Öved, Sweden) as described above.

RESULTS AND DISCUSSION

Shear contribution

The influence of shear on the measurement of the extensional viscosity was evaluated using the PDMS putty at its lower Newtonian plateau. The plateau value of the viscosity is $\eta_0 = 25$ kPas and the plateau extends up to $\dot{\gamma} \approx 0.1$ s^{-1}.

The PDMS material was measured in contraction flow at low extension rates. The measured stress was $\sigma_{meas} = 28.0$ kPa at $\varepsilon = 0.02$ s^{-1}. The measured stress in extension includes contributions from both extensional flow and shear flow. The contribution from shear flow decreases the more shear-thinning the material is, as can be seen from equation 9. Equation 9 gives for the PDMS material at the lower Newtonian plateau, with a given nozzle geometry of r_0=20mm, r_1=4.8, H=15mm, Q=3x10^{-8} m³/s, n=1 and K=28000 Pas

$\sigma_{shear} = 25.9$ kPa at $\varepsilon = 0.02$ s^{-1}

When the shear stress is subtracted from the measured total stress, the extensional stress becomes

$$\sigma_E = \sigma_{meas} - \sigma_{shear} = 28.0 - 25.9 = 2.1 \text{ kPa}$$

at $\dot{\varepsilon} = 0.02 \text{ s}^{-1}$

Shear is the major contribution to the measured stress, and the compensated stress corresponds to an extensional viscosity of

$$\eta_E = 105 \text{ kPas}.$$

In the limit of low strain rates [10]

$$\eta_E(\dot{\varepsilon})\Big|_{\dot{\varepsilon}\to 0} = 3\eta(\dot{\gamma})\Big|_{\dot{\gamma}\to 0}$$

In our measurements $\eta_E \approx 4.05\eta$, which is close to the theoretical prediction and well within the experimental error.

It is clear that the method is not suitable for Newtonian fluids since the major contribution to the measured stress comes from shear. However, for shear thinning fluids the shear contribution to the measured stress is small as illustrated in Figure 3. The measured stress in the contraction flow measurement of the PDMS was $\sigma_{meas} = 28.0$ kPa which could be taken as a typical value of the measured stress. This could be compared to the calculated shear contribution from equation 9 for various values of n and K as shown in Figure 3. The figure shows that the shear contribution is low for low n and low K.

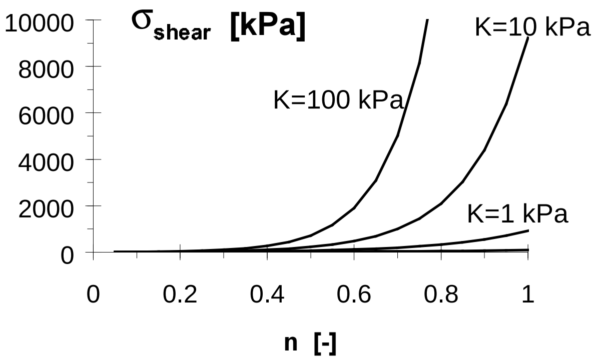

Fig 3. Calculated shear contribution to the measured stress for a nozzle with r_0=10mm, r_1=5mm, H=15mm and Q=3mm³/s.

Flow behaviour of fermented milk products

The perceived texture of the three different fermented milk products in the study is very different. The “långfil” has a long texture and is very different from “filmjölk”. The measured flow curves in shear are despite the perceived differences quite similar as shown in Figure 4. Both apparent viscosity and shear thinning behaviour are similar for all three samples.

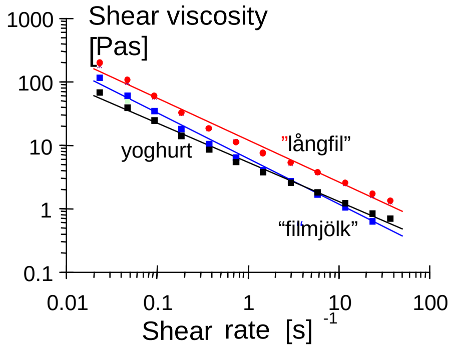

Fig. 4. Flow curves in shear for fermented milk products.

The flow behaviour of the same products was measured in elongation by contraction flow measurements. Both apparent viscosity and tension thinning was different for the three samples, as shown in figure 5. The calculated shear contribution was negligible for these samples. The example shows clearly that the extensional flow behaviour sometimes correlates better with the perceived texture than the shear behaviour.

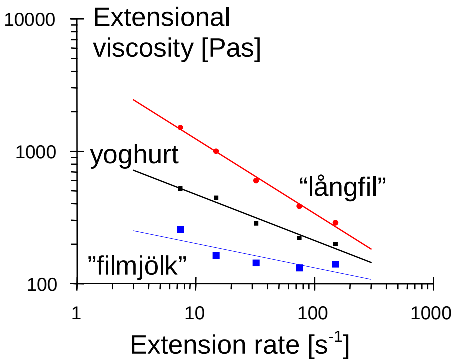

Fig. 5. Flow curves in extension for fermented milk products.

Contraction flow measurements can therefore be used to characterize foods for which extensional flow is expected to influence the perceived texture. Such foods are semi-solids such as dairy products, spreads and other emulsions, which we squeeze between the tongue and the palate causing an extensional flow. Spreadability is also expected to depend largely on extensional flow behaviour thus favouring contraction flow measurements.

CONCLUSIONS

- Contraction flow is a useful technique for determining extensional flow properties of semi-solid foods.
- The technique covers a wide viscosity range.
- The extensional flow behaviour may show perceived differences better than the shear flow behaviour.
- Contraction flow measurements works well for shear-thinning materials, but Newtonian and shear-thickening materials should be avoided.
- The method is not suitable for low-viscous fluids.

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