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A Low Cost Extensional Viscometer for Small Relaxation Times

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Abstract

An extensional rheometer based on capillary breakup is described which has been used to measured short relaxation times. The design using parts found in the university department incorporates a fast acting solenoid, a high speed video camera, long distance microscopic lens and the Mathematica® software to obtain the capillary breakup data from a series of images. The rheometer was verified with a Newtonian 500 cSt silicon oil where the shear viscosity was within 1% of that obtained from a cone and plate measurement with a Kinexus Pro rheometer. A 200k molecular weight (MW) polyethylene oxide (PEO) solution was used as the test fluid and for concentration below 1 wt% a power law exponent of 0.78 was found for the relaxation time variation with concentration. The relaxation times measured ranged from 0.5 to 15 ms.

Introduction

The extensional viscosity, η_E, and the relaxation time, τ_R, of a fluid is an important parameter in many engineering applications, as in the extrusion of plastics, coating and spraying flows, food manufacture and blow moulding [1]. However the standard rheometer generally measures shear viscosity, η_0, as a function of shear rate or shear stress and viscoelasticity parameters from an oscillatory flow. The relaxation time relates to the amount of energy being stored in the fluid similar to the relaxation of a spring after being stretched. The measurement of the extensional properties of a fluid is important, both for adequate characterisation of fluids with complex behaviour as well as providing physical data for modelling the fluid behaviour.

For a Newtonian fluid, the ratio η_E/η_0 is known as the Trouton ratio and has a value of 3 but is usually much larger for complex fluids. Macro and micro instruments can be found for measuring extensional viscosity with differing accuracy and complexity. The macro or large devices have been developed from the early work of Trouton [1] on the stretching of a cylinder of pitch wherein most devices attempt to place the fluid in an extensional flow while minimising shearing effects. The first commercial device, the opposed jet rheometer developed by Fuller *et al.* [2], could measure the extensional properties of fluids with viscosities down to a few mPas. A review of extensional rheometers for low viscosity fluids has been given by Galindo-Rosales *et al.* [3]. Drop based devices include the Rayleigh Ohnesorge Jetting Extensional Rheometer (ROJER) [4, 5] capable of measuring relaxation times as low as 60 μs and the optically-detected elastocapillary self-thinning dripping-onto-substrate (ODES-DOS) extensional rheometer [6] claiming resolution down to 0.35 ms.

Capillary Breakup Extensional Rheometers (CaBER)

Commercial equipment for the measurement of elongational viscosities include the Rheotens® for highly viscous polymers, Sentmanat extensional rheometer for stretching polymer films, FiSER® which elongates a filament at exponential rates, CaBER® and VADER®. The concept of utilising the breakup of a thin filament of fluid to determine the elongational viscosity and relaxation times in the CaBER rheometer was first conceived by Bazilevskii *et al*. [7, 8] and is currently marketed by Thermo Scientific. In the CaBER, the filament is rapidly stretched in opposing directions, and the diameter at the midpoint of the filament length is monitored as a function of time until breakage occurs. The range of relaxation times of dilute polymer solutions that can be studied range from 10ms to 1 s [5]. Ensuring that the opposing movement of filament ends occurs at a similar rate requires precise mechanical setup and a simpler approach with only one end of the filament being stretched was used in the Seymour extensional rheometer [9] and its successor [10]. A low cost attempt using a smartphone and a clothes peg gave reasonable values for the relaxation times of a high MW PEO solution [11].

In this work, a filament breaking type extensional viscometer was designed and tested. The setup is similar to the Seymour extensional viscometer [9] with some variation in the design allows relaxation times down to 1 ms to be measured.

Equations governing the capillary breakup

For a Newtonian fluid with a constant shear viscosity (η_0) and surface tension (σ), the contraction of the filament dimensionless diameter (D/D_0) is given by

$$\frac{D}{D_0} = 1 - \frac{(2X-1)\sigma t}{3\eta_0 D_0}, \qquad (1)$$

where a correction factor (X) to account for the non-cylindrical nature of the filament as it is attached to plates at its ends. Numerically, for the case of a viscous filament with no inertial effect and a smoothly necked profile, X=0.7127 [12, 13].

For a visco-elastic fluid, the rate of filament thinning is related to the constitutive equation that best describes its behaviour. For an upper convected Maxwell model [14], the filament thinning for the largest relaxation time is given by

$$\frac{D}{D_0} = e^{-t/(3\lambda)}. \qquad (2)$$

The dimensionless filament diameter change with time (t) allows the relaxation time, λ, to be determined.

Equipment

Extensional viscometer

The extensional viscometer consists of an electromagnetic solenoid for its upper part with a 1.18±0.01 mm diameter circular flat plate cut into the solenoid plunger. The lower part consists of a bolt with an identical plate fixed to its tip which can be raised or lowered by turning the bolt; this allowed the initial gap between the two plates to be set. The setup is shown in figure 1.

The fluid to be studied is placed between the two plates. A switch is used to control the voltage pulse into the solenoid, which induces a magnetic field causing the solenoid plunger to rise vertically away from the lower plate. The fluid between the plates is stretched and a filament forms between the two plates. The filament thins and contracts due to capillary forces acting on the surface and after a finite time, it thins sufficiently to break up. The vertical motion of the solenoid

was restricted by a pin inserted horizontally though the solenoid plunger. The solenoid motion was arrested when the pin struck an O-ring. The position of the O-ring could be adjusted and fixed with a circular clip and this allowed the final separation of the plates to be varied. The circular clip ensured that the O-ring will not slip after repeated impacts by the pin.

The solenoid and plate set up was mounted on a stand with a rod that allowed the vertical position of the extensional viscometer setup to be moved along a rod. This provided the flexibility to accommodate the positions of the light source and camera.

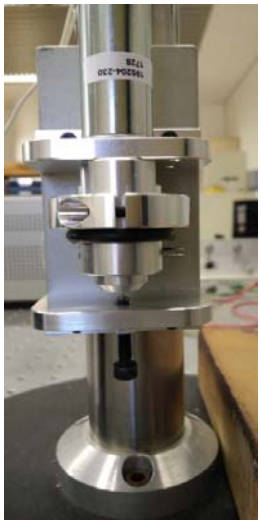

Figure 1. The extensional viscometer.

The lighting used was a variable 0-50W LED array but 5/15 W inputs were sufficient for 15000/30000 fps respectively. A Photron AX-100 high speed colour video camera was available and was used to test the limits of the extensional viscometer. The solenoid was a Ledex® STA 195204-230 pull tubular solenoid (sourced from Element14) with a 30 awg coil and can handle 12-38 VDC. It was chosen to have a fast acting response. Most fast acting response solenoids require a high voltage and this has a recommended voltage of 24V at 25% maximum duty cycle but will still operate at voltages below or above it. The plunger is 7.82 mm in diameter, has a central vertical slot of 2.36 mm width and 9.52 mm length, and a 2.34 mm diameter hole located halfway up the slot.

To obtain high resolution images of the filament, a user defined frame size was used. For a framing rate of 15000 fps, 384×568 pixels were used to frame a 2 mm final gap. The lens setup included a Nikon 65-300 mm lens, a +10 close-up attachment and 153 mm length of F-mount extension tubes, mounted on a 2D traverse with support. The image sharpness as determined by the depth of field, decreased as the focal length was increased. A resolution of 2.5 μm per pixel could be achieved but the small depth of field made focussing difficult. A compromise with a focal length of 150 mm was used which gave a 5 μm per pixel resolution but provided a much larger depth of field. A 160 μm diameter wire was placed at the centreline to assist in focussing.

Plunger speed

The plunger speed determines how rapidly the filament can be formed and responses for different applied voltages from a power supply are shown in figure 2 for a final gap of 2.9 mm from an initial gap of 0.42 mm. The plunger initially moves further than the endpoint and bounces back after striking and compressing an O-ring (natural Buna rubber of 60 Shore A hardness) which is used to limit its motion. The bounce back is approximately 15% of the stroke but the bounce back is almost damped out thereafter. The bounce back affects the time when measurements can begin.

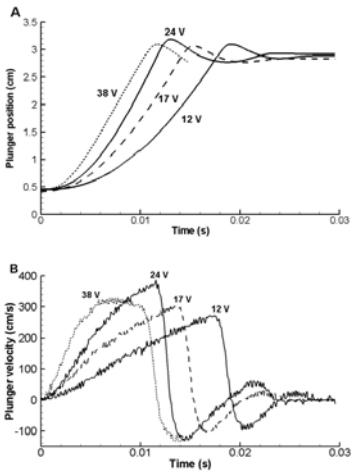

Figure 2. A. Solenoid plunger position with time for different excitation voltage from a power supply. B. The velocity of the plunger for different excitation voltages.

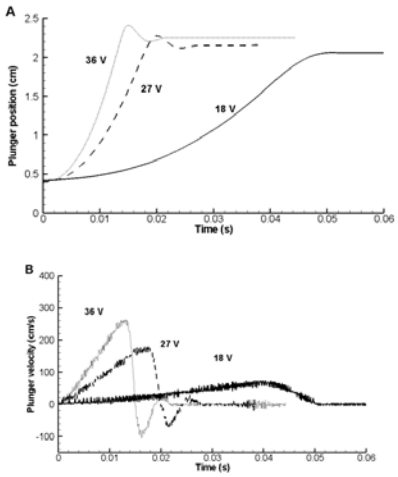

Figure 3. Solenoid plunger position (A) and plunger velocity (B) for different battery excitation voltages.

As a comparison with the CaBER and Seymour extensional viscometers, the rise times for the CaBER and Seymour extensional viscometers are approximately 9 and 7 ms respectively is required for a final 2 mm gap [9]. The rise times for this setup are 6.3, 8.0, 9.5 and 12.4 ms for applied voltages of 38, 24, 17 and 12 V respectively. However similar to the the CaBER and Seymour there is a bounce after the initial rise which needs to be damped out before measurements can be taken; a minimum of 24 ms for an applied voltage of 24 V. As the bounce increases with applied voltage this negates the effect of a faster rise time. Fluids that break up with a 10 ms time frame after being stretched will not have reached an equilibrium enabling measurements to commence. The acceleration during the rise of the plunger is fairly uniform but increases with applied voltage of 12 to 38V ranging from 1.5*g* to 5.1*g*. The deceleration of the plunger on contacting the O-ring is fairly similar for all the applied voltages and varied only between −22.4*g* to −27.6*g*.

A second test of the applied voltage using 9V batteries in series to test portability, for a 2.2 mm final gap spacing, is shown in figure 3. Adjusting the O-ring and using batteries instead gave a slower rise times for 49.5, 19.3, and 14.7 ms for 18, 27 and 36 V battery voltages. The 18 V was slow enough

that there was no rebound of the plunger. The plunger rebounded for the 27 and 36 V input with a total settling time of 27.4 and 21 ms which allows an earlier start when measurements can be taken. The use of batteries reduced the pull-up acceleration down to 1.7*g* for the 36V input and only 0.3*g* for the 18V input. The plunger rebound was reduced further with some adjustments to the O-ring.

Experimental method

A drop of test fluid is carefully placed between the plates while the upper plate is raised. A 25G needle is used to place the drop and excess fluid is absorbed on a thin wedge of adsorbent paper. The video camera is started followed by an excitation voltage applied to the solenoid. The video camera is then stopped and the excitation voltage removed. The frames of the filament dynamics are saved as a series of tiff images.

Results and Analysis

Image Processing

The resolution of the image is approximately 5 μm per pixel while the thread width varies from 80 pixels to 19 pixels at the start and zero when the thread breaks. Thresholding and binarizing the image will give a staircase plot of the thread width with time if only the change in the number of pixels of the width is tracked. The edge of the thread results in a change in the light intensity over a few pixels and edge detection is used to track the shape. To provide better resolution,

i. the image is initially magnified to double its size to increase resolution with a bi-cubic sampling to fill in the interleaved pixels,
ii. sharpened with a pixel radius of 2 where a high frequency version of the image is combined with the original to add detail and highlight the edges,
iii. converted to an 8-bit gray scale,
iv. increased contrast with gamma correction to enhance gray scale to ensure that the lighter and reduced gray scale variation is made more pronounced as the thread continues to thin,
v. edge detection using the Canny detection algorithm with a specified pixel range varying from 2 to 5. A larger value of the pixel range is required for the magnified image to ensure that the edge is detected.
vi. The positions of the edges and the gray scale intensity along the centre of the thread are written out. A Fortran program was written to carry out sub-pixel interpolation from the position data of the edge to provide a smoother variation of the thread width with time.

Measurement of a Newtonian fluid and its extensional viscosity

A 500 cSt silicon oil (25°C, Sigma-Aldrich) was used as the test fluid. The slope of the dimensionless diameter with time provides the extensional viscosity of the fluid which is three times the shear viscosity for a Newtonian fluid. The silicon oil shear viscosity (figure 4) was also measured at 21.5 and 25°C on a Kinexus Pro rheometer with a 1°-60 mm cone.

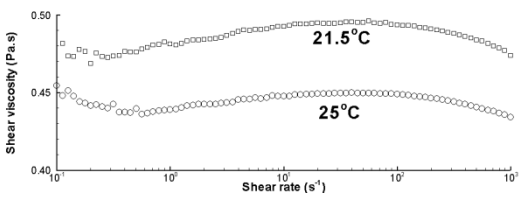

Figure 4. Shear viscosity of a 500 cSt silicon oil measured with 1°-60 mm cone and plate fixture for 21.5 (room temperature) and 25°C.

The average shear viscosity is 0.487±0.007 Pa·s for shear rates between 0.1 to 1000 s^{-1}. The surface tension of the silicon oil measured with a Wilhelmy plate was 0.021±0.005 N/m. The dimensionless diameter with time of the filament for a 1.46 mm gap height (within the capillary length of 1.48 mm) is shown in figure 5. The slope gives a shear viscosity of 0.474±0.006 Pa·s for three runs.

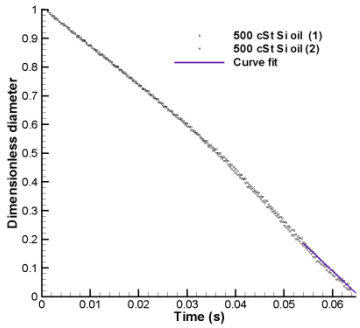

Figure 5. Dimensionless diameter against time for a 500 cSt silicon oil. Two sets of almost identical results are presented with only a quarter of the data plotted for clarity. The straight line of fit for estimating the shear viscosity is over the 0 to 0.2 dimensionless diameter section (the limiting region indicated in [14]).

Relaxation times of a 200k MW PEO solution

A 5 wt% 200k MW PEO solution was prepared and diluted to provide a series of lower concentrations (2.5, 1.0, 0.5, 0.25 wt% solutions). The critical overlap concentration, c^*, where polymer coils start to overlap, is estimated to be 0.5 wt% [6]. An initial set of runs were made with an initial gap of 0.3 mm and a final gap of 2.2 mm. The final gap was too wide for the lower concentrations so a second set of runs were made with a final gap of 1.3 mm. The relaxation times were obtained from the elasto-capillary region of the thinning dynamics. Figure 6 shows representative data of the thinning of the PEO filament for the different concentrations. The 0.25 wt% solution had a shear viscosity close to that of water. The stretching and thinning processes are captured in figure 6 since a large portion of the process occurs while the plunger is moving to its final position.

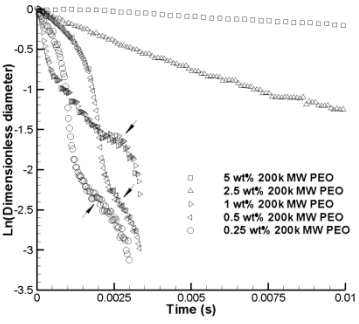

Figure 6. Dimensionless diameter against time for different concentrations of a 200k MW PEO solution. The starting times vary with 1 wt% and lower, beginning when the plates are still separating and the arrows show where the relaxation times were obtained after the plates have stopped moving.

Figure7 shows the relaxation time obtained from the exponential decay portion of the dimensionless filament radius using equation (2) in the elasto-capillary region. The data for the 1, 0.5 and 0.25 wt% PEO gives a power law exponent of 0.74 with the concentration which is in line with previous investigators [6]. However for the higher concentrations of 2.5 and 5.0 wt% PEO, a much higher power law exponent of 1.66 was obtained and this could be due to the fact that they are not dilute solutions.

The apparent viscosity as a function of the Hencky strain is shown in figure 8 and the 2.5 and 5 wt% PEO solutions show strain hardening as the filament progressively narrows.

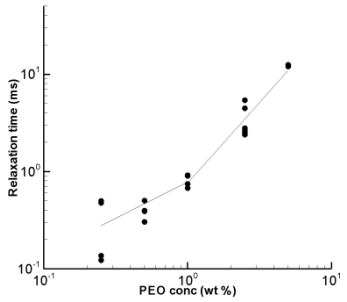

Figure 7. Relaxation times for the different 200k MW PEO concentration. Two separate power law exponents were determined, 0.74 below 1 wt% and 1.66 above 1 wt%.

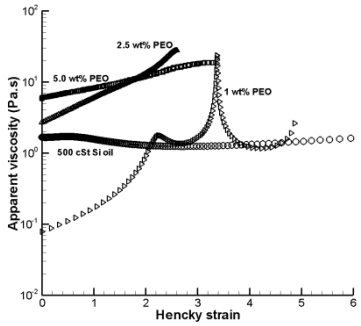

Figure 8. The apparent viscosity $\left[\eta_E = -\sigma/(\frac{2dR}{dt})\right]$ against the Hencky strain $[\varepsilon = 2\ln(R_0/R(t))]$ showing the Newtonian behaviour of the silicon oil and the strain hardening of the 2.5 and 5 wt% PEO solutions. The 1wt% PEO includes the filament behaviour during the stretching phase, with stretching stopping at the sharp peak.

Costing of the extensional rheometer

The extensional rheometer has been constructed largely from items salvaged from older equipment while more expensive items were borrowed. Table 1 gives a breakdown of costs of parts used in this project as well as a cheaper version that could be used which will give a lower resolution on the relaxation time.

Item	Current	Cheapest
Solenoid	$25.00*	$25.00
Lighting	$500.00	$15.00 (LED torch)
Camera		
- Photron AX200	$48,000	$300 (used iphone 6)
- Lens	$800	–
- Extension tubes	$50*	–
- Macro lens	$30*	$20+ (online)
- 2-D traverse	$28*	
Laptop	$800+	Personal laptop
Software	University site licence	Free (*imageJ, Fiji*)
Stand	Recycled part	$35 (stand and clamp)
Workshop costs	Not included	Machining skills

Table 1. Listing of the cost of items. *Parts purchased specifically for this project. The rest were obtained around the department. The cheapest option is based on parts that can be bought on-line but does not include shipping.

A number of standard parts can often be sourced either from equipment that is used for other purposes or older items that are being recycled or disposed. Access to workshop facilities is assumed to be available and that cost must be factored in based on the local charges. Camera accessories can be obtained cheaply through online stores but the quality is difficult to determine *a priori*. Small parts sourced from workshop include an O-ring, gauge sheet metal, screws, steel, and other consumables.

The cheapest option that can be constructed will require some capability in machining and is also limited by the framing rate and resolution (720p at 240 fps for an iphone 6) but is suitable for viscous and fluids with larger relaxation times.

Conclusions

A simple construction for an extensional viscometer has been presented which can be easily constructed from parts that can be found in a university department. The results show that it is possible to extract relaxation times down to 1 ms. If specialised parts, such as a high speed camera, are not available, longer relaxation times can be measured with mobile phones and cheaper parts from the internet, assuming that most researchers will already have access to a mobile phone and laptop.

Acknowledgments

Butler K. for machining of the solenoid cage.

References

[1] Galindo-Rosales, F.J., Alves, M.A. & Olveira M.S.N. Microdevices for Extensional Rheometry of Low Viscosity Elastic Liquids: A Review, *Microfluid Nanofluid,* **14**, 2013, 1–19.

[2] Trouton, F.T. On the Coefficient of Viscous Traction and its Relation to that of Viscosity, *Proc. Roy. Soc.*, **A77**, 1906, 426–440.

[3] Fuller, G.G., Cathey, C.A., Hubbard, B. & Zebrowski, B.E. Extensional Viscosity Measurements for Low-Viscosity Fluids, *J Rheol.* **31**, 1987, 235–249.

[4] Keshavarz *et al.* Studying the Effects of Elongational Properties on Atomization of Weakly Viscoelastic Solutions using Rayleigh Ohnesorge Jetting Extensional Rheometry (ROJER), *J. Non-Newtonian Fluid,* 222, 2015, 171–189.

[5] Greiciunas, E. *et al.* Design and Operation of a Rayleigh Ohnesorge Jetting Extensional Rheometer (ROJER) to Study Extensional Properties of Low Viscosity Polymer Solutions, *J Rheol.*, **61(3)**, 2017, 467–476.

[6] Dinic, J., Zhang, Y., Jimenez, L.N. & Sharma, V. Extensional Relaxation Times of Dilute, Aqueous Polymer Solutions, *ACS Macro Lett.*, **4**, 2015, 804–808.

[7] Bazilevskii, A.V., Entov, V.M. & Rozhkov, A.N. In *Proceedings of the 3rd European Rheology Conference* (Ed. Oliver, D. R.), Elsevier, 1990, 41–43.

[8] Bazilevskii A.V. *et al.* Failure of Polymer Solution Filaments. *Polym. Sci.*, **A39**, 1997, 316–324.

[9] Collett *et al.* A Portable Extensional Rheometer for Measuring the Viscoelasticity of Pitcher Plant and Other Sticky Liquids in the Field. *Plant Methods*, **11**, 2015, 16.DOI 10.1186/s13007-015-0059-5.

[10] Hallmark *et al.* A Portable and Affordable Extensional Rheometer for Field Testing. *Measurement Science and Technology*, **27**, 2016, 125302.

[11] Swift, A. Development of a Smartphone App to Easily Measure Extensional Viscosity, *BSc (ChE) thesis*, 2018, Oregon State University, USA.

[12] Papageorgiou, D.T. On the Breakup of Viscous Liquid Threads, *Phys. Fluids*, **7**, 1995, 1529–1544.

[13] Entov, V.M. & Hinch, E.J. Effect of a Spectrum of Relaxation Times on the Capillary Thinning of a Filament of Elastic Liquid. *J. Non--Newtonian Fluid Mech*., **72**, 1997, 31-53.

[14] McKinley, G H. & Tripathi, A. How to Extract the Newtonian Viscosity from Capillary Breakup Measurements in a Filament Rheometer. *J. Rheol*., **44(3),** 2000, 653-670.