Instrumentation and start up of a new elongational rheometer with a preshearing history

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This article describes the design, start up, and some preliminary results of a rheometer that preshears the test fluid and measures its elongational viscosities. Preshearing takes place within a concentric cylinders arrangement. Fluid goes out from the lower part of the cylinders arrangement where an elongational flow is produced, by means of a vacuum chamber, which draws out the fluid filament. The influence of the preshearing history upon the elongational properties is analyzed. Data of elongational stress, uniaxial stretch rate, elongational normal stress difference, and apparent elongational viscosity are obtained. The rheometer design is based on a previous work by Sridhar and Gupta [Rheological Act **24**, 207 (1985)]. Glycerin was used as the test fluid. Stretch rates up to 1250 s^{-1} were found. Trouton's ratios ranging from 2.6 to 3.6 were obtained. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1494856]

I. INTRODUCTION

The elongational properties of most materials are of paramount importance for different applications, therefore, there is a need for apparatuses capable of providing trustable measures of the elongational viscosity under quasisteady elongational flow, since a pure extensional flow is difficult to obtain.

While great advancements have been made in the development of extensional rheometers for concentrated solutions, the progress for dilute solutions has been relatively slow. Recently, some designs have been proposed and these may rectify the problem.[1–3] In any case, imposing a constant stretch rate on these fluids is important to obtain a well-defined extensional deformation, but this has not been attained in current devices. Consequently, there are few or no data on the steady extensional viscosity. The impetus for the study of the extensional deformation of solution comes from its importance in a variety of applications.[4–8]

II. DESCRIPTION OF APPARATUS

Figure 1 shows the general scheme of the overall system. It consists of a fluid supply system, a preshearing section, an elongational section, a vacuum generation system, and the electronic instrumentation system. The various parts of the system are described in the following sections.

A. Fluid supply section

The fluid supply system was designed to supply the test fluid at a constant flow rate. The system (Fig. 1) consists of two closed interconnected cylindrical containers mounted on a specially designed metallic structure. In the first container (R_1), a water column is set to a specific height, whereas the empty space left is pressurized with nitrogen. The second container (R_2), is filled with the testing fluid. It also has a neoprene balloon fitted under its top lid. The balloon is filled with water at a constant flow rate and it works as a pump pushing the testing fluid outside the container towards the rheometer. Both flow rates, water and test fluid, are bound to be the same according to the principle of mass conservation.

To quantify the water flow rate, there are two flow meters on the feed line, one analog and one digital. The flow rate is controlled by means of a control system, which consists of a flow meter, a solenoid valve, and the neoprene balloon. The amount of testing material going inside the rheometer is controlled with the opening percentage of the valve.

B. Preshearing section

The preshearing section of the rheometer (R_3) consists of two high-precision concentric cylinders joined together by two metal lids (Fig. 1), the 10 mm gap between the cylinders being filled with a viscous testing fluid and the inner cylinder made of aluminum with a diameter of 280 mm can rotate at a steady angular velocity. The outer cylinder made of acrylic stands still as it cannot rotate. The height of the cylinder is 600 mm. The dimensions of the cylinders were established to ensure a long residence time of the fluid inside the preshearing section, i.e., to diminish the helical flow as much as possible. The rate of shear in the gap between cylinders is constant since this gap is smaller compared with the inner cylinder's radius. To measure the stress inside the cylinders, three 200-mbars Druck™ pressure transducers are placed on the main body of the rheometer (Fig. 1). This section was designed to support an inner pressure of up to 2.5×10^5 Pa. A 15-mm perforation drainage is located, in the opposite side of the pinhole.

C. Elongational section

The elongational section is found where the fluid filament is formed (Fig. 1). This is in the gap between the lower

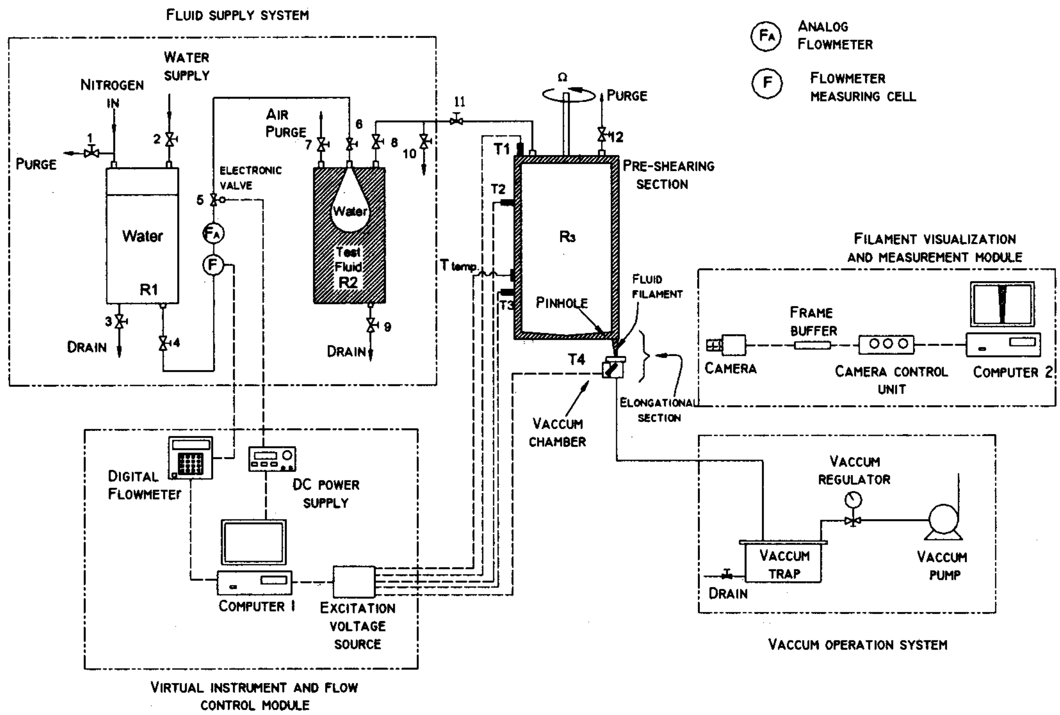

FIG. 1. Scheme of overall system.

lid of the cylinders and the vacuum chamber. The fluid filament exits through a 1-mm flat pinhole, with a thickness of 0.013 m, placed on top of a 2.7-mm perforation located on the cylinders lower lid. The pinhole is centered between the two cylinders' gap. The main function of the pinhole is to control the filament diameter. The fluid filament is aligned with the center of the suction chamber.

D. Vacuum generation system

The vacuum generation system consists of a vacuum pump, a vacuum trap, and a suction chamber. The chamber is an acrylic device specially designed (Fig. 2) to hold two pressure transducers and to suck the testing fluid filament once the vacuum pressure is established. A one-bar vacuum pump is connected to the chamber for this purpose. The fluid filament is sucked through an existing 3-mm hole on top of the chamber. The suction chamber is movable to adjust the filament extension distance. To measure the vacuum generated, inside the vacuum chamber, two one-bar Druck™ transducers were positioned.

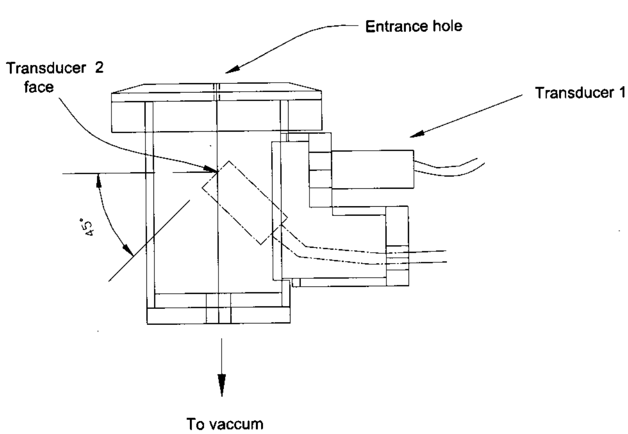

FIG. 2. Diagram of vacuum chamber.

E. Electronic instrumentation system

This system includes a filament visualization and measurement module together with a virtual instrument that make possible the data acquisition of the operating parameters, the fluid flow control, and the data processing.

The filament visualization and measurement module consists of a solid-state Kodak™ Megaplus change-coupled device (CCD) high-resolution digital camera IBM™ compatible computer, a SVGA Display card 1 Mb memory, a solid-state Kodak™ Megaplus CCD high-resolution digital camera and camera control unit, a 55-mm micro Nikkor™ lens, with extension tube, a VGA computer monitor, Imagraph™ frame grabber board, and a adapter box, Model Hidef II, Image application program software Omegatek™

The camera photographs and digitalizes the fluid filament image with a 1317 horizontal $\times$ 1035 vertical format. The exposure time is set to 10 ms to ensure an undisturbed image. The camera is mounted on a moving platform capable of moving upward and downward as well as forward and away from the filament, in order to take full or partial fluid filament photographs. Once the image is digitalized, it is transferred from the camera unit frame buffer to the computer random access memory. Then, the image-processing software locates the edges of the filament and measures the distance between them. Up to 99 samples along the filament can be taken. Through a previous calibration procedure, the software converts the distance between edges from pixels to centimeters. The data are automatically stored in a hard disk

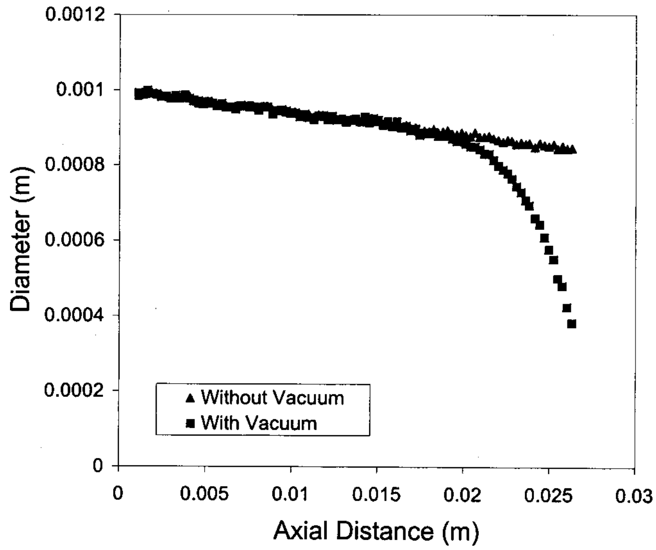

FIG. 3. Comparison of diameter vs axial distance for filament with and without vacuum, flow rate 30 ml/min.

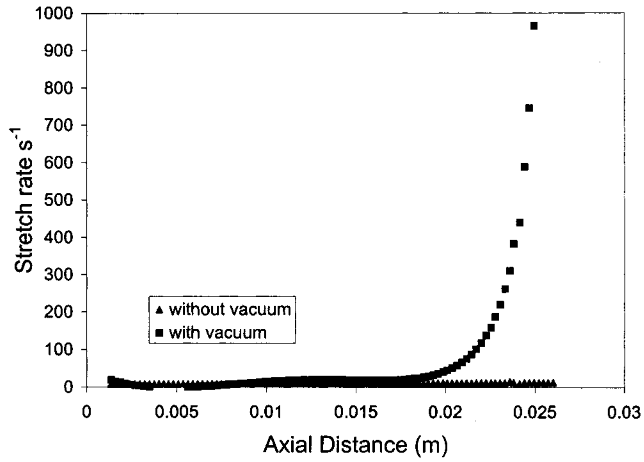

FIG. 4. Comparison of stretch rate vs axial distance for filament with and without vacuum, flow rate 30 ml/min.

file for further treatment. The filament image is visualized in the computer monitor; even though its definition is lower than the image used for the measurement.

The first part of the virtual instrument is the data acquisition module that consists of a DAQ board, a personal computer, and a specially design program. The DAQ is a multifunction I/O data acquisition board model 6023E from National Instruments (Austin, Texas). It is used to acquire the transducer signals. The board has eight differential inputs with a resolution of 12 bits and a sampling rate of 200 kS/s. The board is a multichannel one. One of the channels was programmed to drive the pressure transducers with the maximum entrance gain (100) since their signal was in the range of ± 50 mV. Another channel was used to drive the temperature transducer with an unitary gain as the range of this signal is 0–5 V.

The data acquisition computer software is an instrumentation graphic code program developed in LabVIEW, which is a National Instruments package (Austin, Texas). Many of its data acquisition functions are already in the libraries of the package, which makes its utilization easy. The developed software allows the conditioning, display, and data storage of the voltage signal generated by the transducers.[9] The personal computer is used as the front panel of the data acquisition module. This panel displays the real-time graphics and the instant numerical values of the operating parameters. The virtual panel has controls to switch on and off the system functions. It also has others that modify the parameters of the functions.

III. AUTOMATIC CONTROL MODULE

The data acquisition software simultaneously executes a proportional integral derivative algorithm that provides the automatic flow control.

The measured flow comes from the digital flow meter through a serial communication via a RS-232 bus. The desired flow is fed into the computer through the front panel. With both values, the measured and desired flows, the algorithm works out the control signal (voltage). This is transformed in a string format and sent to the dc power supply by a serial communication via a RS-232 bus. The power supply provides a control signal of the actuator: a voltage-proportional-electronic valve.

IV. DATA PROCESSING

The data processing is carried out with a LabVIEW program specially designed for the purpose. The data utilized are: elongational stress (at $x=0$), flow, material density, and diameter profile. The calculated data: fitted diameter, axial velocity, stress rate, elongational stress, and apparent elongational viscosity are plotted versus axial distance. Elongational stress versus stretch rate is presented as an additional plot.

V. EXPERIMENT

To evaluate the rheometer behavior, glycerin (99% purity) was used during the experiments. The glycerin shear viscosity (1.01 Pa s) was measured by carrying out a test in an AR1000-N rheometer from TA Instruments. The tests were always performed at room temperature (around 20 C). In all tests the flow rate used was 30 ml/min.

For this preliminary experiment, a 25-mm distance between the vacuum chamber and the pinhole was set. The test fluid is pumped from the container R_2 (Fig. 1) into the main body of the rheometer (R_3). Once the pressures inside R_3 are constant, the steady state is achieved, and then the vacuum is applied and the filament photograph is taken. Pressures, with and without vacuum, are recorded. This procedure is repeated three times in order to prove repetitively. Rotating the internal cylinder with a constant angular velocity preshearing is applied to the test fluid. The procedure is repeated for every chosen angular velocity.

VI. RESULTS AND DISCUSSION

Figure 3 shows the plot for the filament with and without vacuum. Between both diameter profiles there is a considerable difference in the last filament section, i.e., the section nearer the vacuum chamber. This difference indicates that the

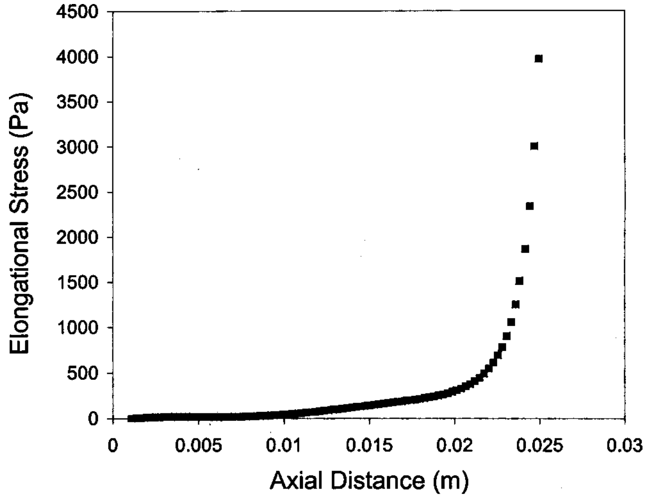

FIG. 5. Elongational stress vs distance, flow rate 30 ml/min.

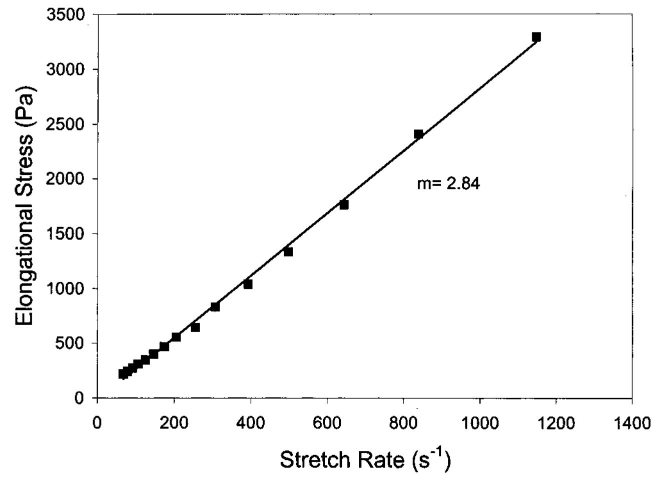

FIG. 7. Elongational stress vs stretch rate.

vacuum does not affect the whole filament. This result was expected since glycerin is a Newtonian fluid. However, it has been reported that for non-Newtonian fluids, the opposite results are obtained with a similar rheometer, the stretch rate varies in a smoother way.[3] Newtonian liquids have a Trouton's ratio (ratio of extensional viscosity to shear viscosity) of three.

VII. CALCULATIONS

Knowing the flow rate (Q) and the fluid filament diameter (W), the velocity (v) values are calculated using a mass balance equation (1)

$$v_i = \frac{4Q}{\pi W_i^2}. \tag{1}$$

The stretch rate $(\dot{\varepsilon})$ is worked out at several points (x) along the fluid filament with Eq. (2)

$$\dot{\varepsilon}_i = \frac{v_{i+1} - v_{i-1}}{x_{i+1} - x_{i-1}}. \tag{2}$$

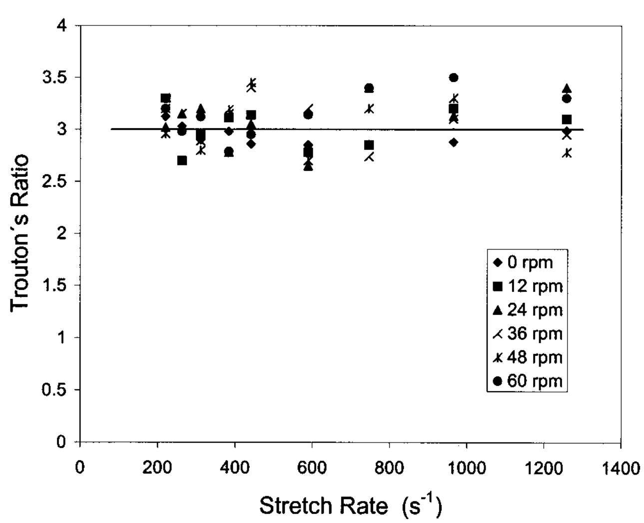

FIG. 6. Trouton's ratio vs stress rate for different preshearing angular speeds, flow rate 30 ml/min.

The calculated stretch rates values range between 250 and 1200 s^{-1} (Fig. 4).

The elongational stress along the filament is worked out with the following balance Eq. (3). The first value is the pressure difference inside R_3 with and without vacuum, where the terms of gravity and surface tension are neglected. Figure 5 shows the variation of stress along the spinline.

$$(\tau_{11} - \tau_{22})_{i+1} = V_{i+1}\left[\frac{(\tau_{11} - \tau_{22})_i}{V_i}\right] + \frac{\sigma\pi}{2Q}(W_{i+1} - W_i) - \rho(V_{i+1} - V_i) + \frac{\rho g}{2}\left(\frac{1}{V_i} + \frac{1}{V_{i+1}}\right) \times (x_{i+1} - x_i). \tag{3}$$

The apparent elongational viscosity in each position is calculated from Eq. (4)

$$(\eta_e)_n = \frac{(\tau_{11} - \tau_{22})_n}{\dot{\varepsilon}_n}. \tag{4}$$

Figure 6 shows Trouton's ratios between 2.6 and 3.6 for stretch ratios between 250 and 1250 S^{-1}, it also shows preshearing effects. It can be seen clearly that pre-shearing does not modify the elongational viscosity. Another plot was prepared to check the viscosity value. Figure 7 shows the stress–strain relation. It can be appreciated that this relation is constant only in the final part of the filament. The gradient of this graph is taken as the elongational viscosity. Both values are in good agreement.

ACKNOWLEDGMENTS

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