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Jetting of dilute polymer solutions

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Abstract Light emitting devices containing conjugated polymers are conveniently fabricated using ink-jet printing. A common problem in the processing of these materials is that the Newtonian viscosity of the polymer solution is not sufficient to describe the jetting performance because the molecular weights and concentrations employed are such that the resulting solutions are elastic. These differences in fluid elasticity levels cannot be measured using traditional techniques like dynamic mechanical experiments or the first normal stress difference in shear, but strongly impact the jetting behavior of the liquid. In this study, a variety of polystyrene solutions in DECALIN having a shear viscosity of ~ 5 mPa s but different elasticity levels were examined for their jetting behavior. The jetting behavior of these solutions was studied visually using drop-on-demand jetting equipment and their rheology was characterized using a custom extensional rheometer designed for measuring the elasticity of such low viscosity liquids. If elasticity effects are absent as in Newtonian liquids (corresponding to a Trouton ratio of 3) satellite drops are formed resulting in loss of liquid and poor positioning. On the other hand, if elasticity effects are very large (Trouton ratios $\gg 4$) separation problems occur at the nozzle with undesirable "tailing." The optimum range for stable, efficient jetting occurs at Trouton ratios in a narrow band between 3 and ~ 5. A very slight degree of elasticity corresponding to a Trouton Ratio around four thus seems to be optimum for the jetting process. This appears to be the first time that such a design criterion has been outlined for this process. Such an approach complements thermal techniques for elucidating the role of molecular and flow properties on the processing behavior of polymeric systems.

Keywords Dilute polymer solutions · Light emitting polymers · Light emitting diodes (LEDs) · Extensional viscosity · Jetting · Ink-jet printing · Satellite drops · Design criterion

Introduction

Light emitting devices (LEDs) are typically used in low information content segmented displays like score logos in stadiums, and in full color video and graphics displays like flat screen liquid crystal displays. A typical polymer LED (Light emitting diode), shown schematically in Fig. 1, is made by sandwiching a layer of light emitting LEP polymer between a pair of electrodes across a pixel having characteristic dimensions of $\sim 50\ \mu$ and typical polymer depths of 100–150 nm [1]. Each pixel is addressed individually using a thin film transistor. When electrons and holes are injected into the polymer, electron exchange reactions occur as they meet together during movement in the electric field. The result is an emissive excited state generated when the electrons and holes recombine. One or both electrodes need to be transparent to let the light generated to pass through it for display and the voltage bias is between 2 and 20 V. The transparent anode is typically indium-tin oxide (ITO) and the usual cathode materials are Ca, Mg, and Al, or Al.Li alloys. Commonly used LEPs are conjugated polymers like poly (phenylene vinylene), polythiophene, and poly (*p*-phenylene). Hole-transport layers like PEDOT-PSS (poly(3,4-ethylenedioxythiophene) doped with polystyrene sulphonic acid) are frequently used to enhance performance as shown in Fig. 1 [1].

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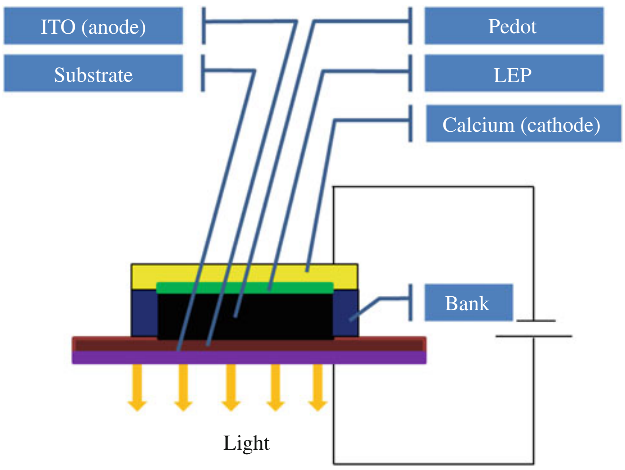

Fig. 1 Schematic of LED device

The polymer film can be deposited by spray coating, dip coating, spin coating or ink-jet printing (IJP). IJP, which is often the most efficient technology for these systems, employs an ink-jet printer with “ink” of conjugated polymer solution to print polymer light-emitting diodes with high resolution. The state of the art regarding this technology has been described by other workers [2, 3]. With this method there is no wastage of expensive polymer solution and large areas can be coated effectively [2]. Note that commercial liquids typically have a low and constant shear viscosity of 3–5 mPa s and low storage modulus or elasticity. Techniques like DMTA (Differential mechanical thermal analysis) based on standard mechanical oscillation rheometers are therefore unable to capture the material properties that are relevant to this flow. Although these elasticity levels are very difficult to measure, especially at these low shear viscosities, our previous work [4] has unambiguously demonstrated that drop formation can show surprising differences which can be related to differences in fluid elasticity; it is reasonable that the best technique for the determination of fluid elasticity is extensional viscosity in view of stretching flow field involved during jetting. Very recently, a specialized piezoelectric vibrating (PAV) viscometer had been developed to characterize viscoelastic properties of low viscosity jetting fluids at high frequency (5 kHz) but it does not yield direct information about the extensional behavior [5]. We expect that this approach outlined in this study will help fill a much needed niche in the range of thermal–mechanical properties available to the practicing engineer.

A spherical drop with no satellites is ideal behavior for the jetting process while a non-spherical drop, tail-formation, and satellite drops are all undesirable. There is a severe need for an understanding of the effect of solution properties on the rheology and consequently the jetting behavior. In this study we examined the jetting behavior of a series of polystyrene solutions in DECALIN having a constant shear viscosity but varying elasticity to simulate commercial liquids and establish guidelines for efficient jetting.

Background

Drop break-up

The break-up of Newtonian jets has been studied by illustrious workers like Rayleigh [6] who pioneered the theoretical study of surface tension driven break up of an inviscid jet in an inviscid medium using linear stability analysis. Weber [7] extended the linear stability analysis to include the jet viscosity. Although, linear stability analysis predicts the size of the main drops it is unable to predict non-linear phenomena such as the formation of satellite drops [8]. Yuen [9] was fist able to predict the formation of satellite drops using non-linear stability analysis. Various studies have focused on the dynamics near the point of break up of viscous jets (e.g., [10], [11]) although the effect of viscoelasticity has not been successfully incorporated into any of these mathematical treatments [12].

Regardless of the form of the constitutive equation, linear stability analysis predicts that an axisymmetric disturbance will grow faster on a viscoelastic jet than on a Newtonian jet [13]. Kroesser and Middleman [15] observed shorter break up length in jets with the addition of polymers while Goldin et al. [14] observed longer break up length. Bousfield et al. [16] applied non-linear stability analysis to the surface tension driven break up of viscoelastic filaments using the Oldroyd-B constitutive equation. They concluded that viscoelasticity could either increase or decrease jet stability depending on the its relative impact on the delay of the onset of disturbance growth, the enhanced disturbance growth, and retardation of the final break up. In the more recent work of Christanti and Walker [17] a second generation of drops was shown to occur on the filaments connecting the main drops. These so called iterated string instabilities were predicted by Chang [18]. The results of Mun et al. [19] on solutions with different polymer molecular weight and concentration indicate that fluids with high apparent extensional viscosity have a longer break up length and form beads-on-a-string structures while fluids with smaller values have a shorter break-up length compared to Newtonian liquids. This has been attributed to strain hardening which is the increase of extensional viscosity with extension rate [2]. The occurrence of beads-on-a-string structure has been shown both experimentally and numerically in the more recent work by Monica et al. [20].

Extensional viscosity

Since jetting flow is primarily extensional in nature the extensional viscosity is the key viscoelastic property. The viscosity of polymer solutions under extensional flow can be several orders of magnitude higher than the corresponding value in simple shearing flow. Flexible polymer solutions display strain hardening (an increase in viscosity with stretch rate) which can impact jetting behavior. In addition to jet stability extensional flows impact many processes such as jet flow through porous media and turbulent drag reduction.

When a material is subjected to homogeneous simple extension, i.e., to a flow which is spatially uniform, with constant rate of strain, $\dot{\varepsilon}$, in the x_1 direction and $-\frac{1}{2}\dot{\varepsilon}$ in every direction perpendicular to the x_1 axis, the ratio of "net tensile stress", $\sigma_E \equiv \sigma_{11} - \sigma_{22}$, to rate of strain is monitored as a function of time. The "tensile viscosity," "elongational viscosity", or "uniaxial extensional viscosity" as per the standard nomenclature introduced by the Society of Rheology, is defined as follows [21].

$$\eta_E(\dot{\varepsilon}) = \lim_{t\to\infty}\left[\frac{\sigma_E\left(t,\dot{\varepsilon}\right)}{\dot{\varepsilon}}\right] = \frac{\sigma_{11}-\sigma_{22}}{\dot{\varepsilon}} \quad (1)$$

There are three different forms of extension: uniaxial (e.g., fiber pulling), biaxial (e.g., balloon inflation), and planar (e.g., cylinder inflation). The latter two forms are less demanding on any polymeric microstructure [22]. Extensional viscosities are extremely difficult to measure because of severe problems in maintaining a homogeneous flow field. High viscosity materials such as polymer melts can be handled relatively easily with testing equipment that is similar in many ways to tensile instruments used for solids. Low viscosity polymer solutions are very difficult to study in extension and the field is still under development for this class of materials. Gupta and Sridhar [23] has reviewed a number of extensional viscosity devices for polymer melts and solutions. Methods like filament stretching [24], fiber spinning [25], capillary break-up [26], and converging flow [27] techniques, which can be used to make measurement on polymer melts, cannot easily deal with polymer solutions of low viscosity. An opposed nozzle device [28] has been used for "apparent extensional viscosity" measurements of polymer solutions but has inherent problems related to non-homogeneity of the flow and data analysis [23].

Experimental details

Materials

Newtonian mineral oils and solutions of polystyrene in DECALIN (cis and trans decahydronaphthalene) were used to study the rheological and jetting behavior. Polymer solutions of different molecular weight (45,000, 100,000, and 140,000), molecular weight distribution and concentration were made by dissolving the polymer in DECALIN; this enabled preparation of polymer solutions with a constant shear viscosity of 5 mPa s and different elasticity. A Newtonian mineral oil of the same viscosity was used as a standard for comparison purposes. Shear viscosity measurements were made on a Carri-Med CSL 100 stress-controlled rotational viscometer at 25 °C using parallel plate fixtures with a 200 μm gap. No obvious difference in shear viscosity was observed before and after stretching indicating no reduction in molecular weight as a result of extension.

The surface tension was measured using a Kruss Drop Shape Analyzer (Model: DSA-10) using a 0.56 mm needle. All the tested liquids had surface tension values lying within a narrow range from 27 to 31 mN m^{-1}.

Extensional viscometer

The extensional viscosities of these fluids were determined using a novel extensional viscometer developed by Agarwal and Gupta [29, 30] that was modified to handle small quantities of fluids in view of the expensive nature of these materials. A schematic of the device is shown in Fig. 2. The viscometer is made of glass and consists of two connected glass bulbs forming a liquid reservoir. Two lines were scored at the top and bottom of the lower bulb and a water bath around the viscometer helped maintain the temperature of liquid inside the reservoir at 25 ± 0.1 °C. The liquid inside the reservoir was drained through a thin wall stainless capillary which was attached to the bottom of the viscometer. The drainage time was determined for the fluid to drain between the upper and lower marks under the pull of gravity. After refilling fluid in the reservoir the

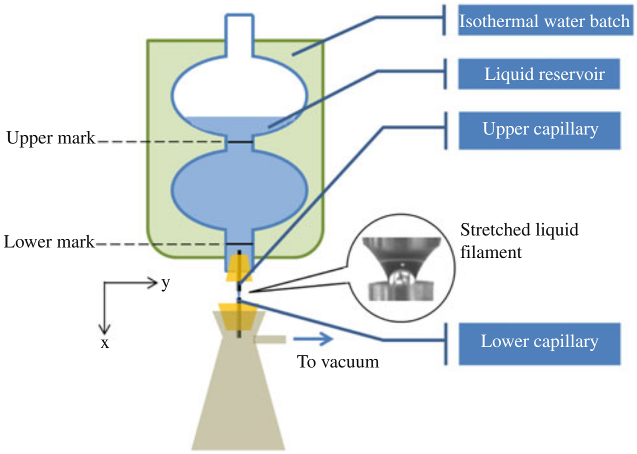

Fig. 2 Schematic diagram of extensional viscometer

drainage time was again determined in a similar manner except that the liquid stream was stretched by sucking it into a lower capillary that was connected to a vacuum flask. The drainage time was recorded again and it was shorter than the earlier value as a result of the imposed tensile stress. The extensional viscosity was determined from the drainage times with and without application of suction as indicated in the Results section. Additional details related to the materials, instruments, and procedures are provided in Ref. [29].

Jetting equipment and drop visualization

To observe "ink-jetting" behavior of these polymer solutions, custom-made jetting equipment which could supply a liquid drop-on-demand was used. All the experiments were conducted at room temperature. A schematic of the experimental set-up is shown in Fig. 3. This setup is composed mainly of two parts: parts: a jetting system and an optical system.

The jetting system consists of a MJ-AB jetting device, a JetDrive III controller and a small strobe light all of which were acquired from Microfab Inc. The orifice size for this jetting device was 60 µm. A computer was connected to the JetDrive III in order to control the process. Jetting parameters were set and a command signal was sent to the jetting device; illumination was provided by a strobe light which was also computer controlled.

The optical system included a Stemi 2000-C stereo microscope (made by Carl Zeiss) and a CCD camera which was attached to a microscope and connected to a computer to record the observed image. A magnification of 100× was used to acquire pictures of jetting drops. To avoid safety issues related to inhalation of DECALIN, experiments were carried out inside a fume hood. During experimentation a transparent plastic cover was used to

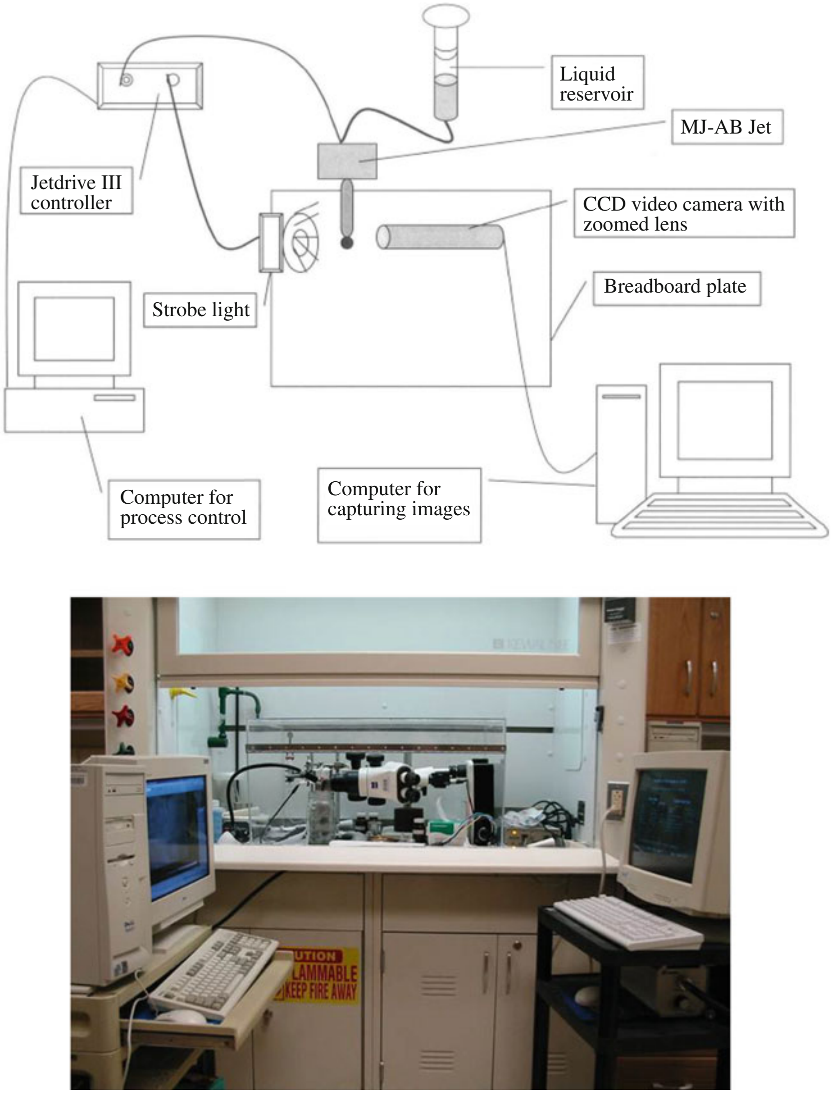

Fig. 3 Schematic diagram (*top*) and photo (*bottom*) of jetting set-up

cover the jetting set-up to avoid air flow disturbance inside the hood which could adversely affect the jetting process. To begin an experiment, the reservoir inside the jetting device was filled with the test fluid and to insure that there was proper back-pressure. After the relevant parameters were set by the software (a jetting frequency of 1000 Hz was employed), continuous signals were sent to the jetting device and the strobe light through the controller. Continuous drops were ejected from the jetting device, and the strobe light fired at the set delay time (from 50 to 450 μs) after each drop was jetted. Pictures of multiple drops, which appear at the same moment after jetting, were taken by the CCD camera. We assume that consecutive drops exhibit the same behavior at the same moment after jetting; this is confirmed by the absence of blurring in the images. The picture taken at any instant thus actually contains many drops although it apparently seems like the motion of a single drop. In this way, multiple drops give the CCD camera sufficient light to capture a clear drop image. Drop images at different moments (from 50 to 450 μs at an interval of 50 μs) and different applied voltages (50, 80, and 100 V) were attained by adjusting the strobe delay. In this manner, a series of pictures was obtained for jetting drops.

Results and discussion

The fluid extensional viscosity, and consequently, Trouton Ratio can be acquired by applying the Hagen–Poiseuille equation to the situation with and without vacuum suction. The following expression for the extensional stress can be obtained for a Newtonian liquid,

$$P_{xx} - P_{yy} = \sigma_{\text{ext}} = \frac{8\eta l V}{\pi r^4}\left[\frac{1}{t_2} - \frac{1}{t_1}\right] \tag{2}$$

where P_{xx}–P_{yy} or σ_{ext} is the extensional stress at the exit of the capillary, η is the shear viscosity of solution, l the capillary length (fixed at 5 cm), V the reservoir capacity between the upper and lower mark, r the inner radius of the upper capillary, t_1 the drainage time without stretching and t_2 the drainage time with stretching. The stretch rate $\dot{\varepsilon}$ is determined by analyzing the filament profile at the exit of upper capillary and is obtained by differentiating the axial liquid velocity v with respect to the axial distance x,

$$\dot{\varepsilon} = \frac{\mathrm{d}v}{\mathrm{d}x} \tag{3}$$

For a constant volumetric flow rate with no radial variation in the velocity the local velocity v and stretch rate $\dot{\varepsilon}$ are given by

$$v = \frac{V}{t_2}\frac{1}{\pi r^2} \tag{4}$$

and

$$\frac{\mathrm{d}v}{\mathrm{d}x} = (-2)\frac{V}{t_2}\frac{1}{\pi r^3}\frac{\mathrm{d}r}{\mathrm{d}x} \tag{5}$$

where dr/dx is the variation of filament radius with axial distance and is obtained by analyzing the liquid filament profile. The apparent extensional viscosity at the exit of upper capillary is then given by:

$$\eta_{\mathrm{e}} = \frac{P_{xx} - P_{yy}}{\dot{\varepsilon}} \tag{6}$$

and the Trouton Ratio (TR) by η_{e}/η.

For illustrative purposes a typical picture taken during the experiment with and without stretching applied to the upper capillary is shown in Fig. 4; the stretch rate is obtained by differentiating this spin line and using Eq. 5.

A series of pictures at an interval of 50 μs was taken for each polymer solution at the moment of jetting. Ideal jetting behavior involves no satellite drop formation, no tailing, and clean detachment of spherical drops from the jet port. Figure 5 shows representative ideal behavior and undesirable behavior as illustrated for the 2.5% polymeric solution with a TR of five and the Newtonian DECALIN which has a TR of three.

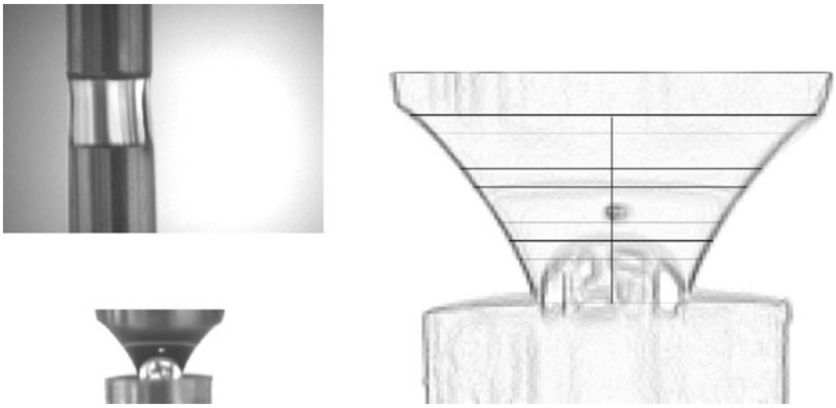

Fig. 4 Typical image for data analysis: *Upper left* image for obstruction under the upper capillary for non-stretching situation; *lower left* image taken during the stretching; right, ×5 magnification of stretched image

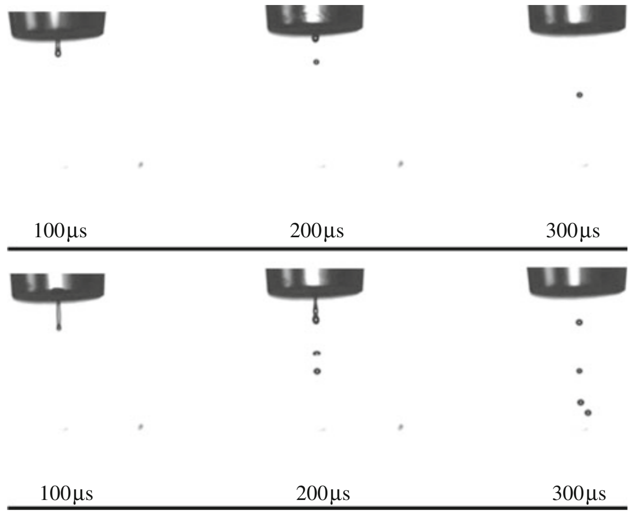

Fig. 5 Typical jetting behavior of liquids (trajectory views of liquid drops at different time intervals after jetting with a chamber driving voltage of 50 V); *Top* ideal behavior for 2.5% polystyrene (MW = 45000) in DECALIN with a TR of five; *Bottom* undesirable behavior for DECALIN with a TR of three

Extensional viscosity and Trouton Ratio data for the polystyrene solutions studied in this study are displayed in Table 1 as well as for DECALIN and the mineral oil. Note that the viscosities and Trouton ratios shown for the Newtonian liquids are the theoretical values.

Similar jetting behavior was observed for the liquids with a TR of 7–9 (two jetting images shown at the right hand side of Fig. 6). Both jetting images show similar features highlighted by a combination of a long tail and separate bead(s). In other words, two liquids, with similar elasticity or close Trouton Ratios, show similar jetting behavior but distinctly different features from the other liquids with significant higher and lower Trouton Ratios. This observation indicates that a good repeatability of both jetting observation and rheological (especially the liquid extensional behavior) characterization was achieved.

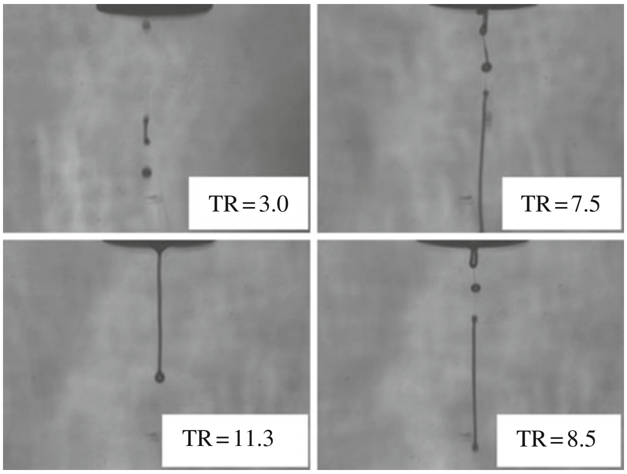

Fig. 6 Jetting images of four liquids with a shear viscosity of 5 mPa s s taken at 200 μs after jetting with a chamber driving voltage of 100 V (Trouton Ratio, or TR, of the liquids increase clockwise in shown images); *Top left* Newtonian mineral oil with a TR of three; *Top right* 5% polystyrene (MW = 45000) in DECALIN with a TR of 7.45; *Bottom right* 3% polystyrene (MW* = 140000) in DECALIN with a TR of 8.47; *Bottom left* 2% polystyrene (MW = 140000) in DECALIN with a TR of 11.3. *Note that molecular distributions are different

As shown in Fig. 6 the solution with higher elasticity (TR = 11.3) tends to form a long tail that is harder to break into drops as compared to the solution with lower elasticity (TR = 8.47). On comparing the Newtonian liquid (TR = 3) with a low molecular weight polymer solution (TR = 7.45), it is reasonable to infer from Fig. 6 that while the Newtonian liquid is easier to detach from the jet port it tends to form satellite drops. This indicates that ideal jetting behavior is related to a proper elasticity level which is high enough to prevent the appearance of satellites and low enough to permit clean separation from the jet port. The data indicate that ideal jetting is restricted to a very narrow range of Trouton Ratios between 3 and 5. Stated differently, slight elasticity corresponding to a Trouton Ratio around four is optimum for the jetting process.

The occurrence of an optimum range of extensional viscosities for jetting might be anticipated from general

Table 1 Extensional viscosity data

Solution	MW	Shear viscosity/mPa s	Extensional viscosity/mPa s	Trouton ratio (TR)
DECALIN	–	2.5	7.5	3.0
2.5% polystyrene in DECALIN	45,000	3.3	16.5	5.0
Mineral oil	–	5.0	15.0	3.0
5% polystyrene in DECALIN	45,000	5.0	37.2	7.5
3% polystyrene in DECALIN	140,000[a]	5.0	42.4	8.5
2% polystyrene in DECALIN	140,000	5.0	56.5	11.3

[a] Note that the molecular weight distributions are different

trends reported in the literature, both theoretical and experimental, that have been summarized in the Background section. First, at low extensional viscosity or elasticity, droplet formation is increased because of increased propagation of any wave disturbance. Second, at high extensional viscosity or elasticity, drop-in-filament structures, and primary drops interspersed with secondary drops are formed as a result of strain hardening. Consequently, having a optimum jetting range is consistent with the physics of the problem. The findings of this study are in qualitative agreement with the results of De Gans et al. [31] who studied the effect of concentration and molecular weight on ink jetting of polyacrylamide solutions. In the first regime a long tail is formed that breaks up along its tail to form satellite drops; at higher extensional viscosity the number of satellites decreases; at even higher values a single drop without a tail is observed and finally the drop does not detach and retracts into the nozzle. These results complement this study and serve to add a preliminary quantitative design criterion for ink jetting of polymer solutions.

Conclusions

The jetting behavior of low viscosity dilute polymer solutions was studied using a custom extensional viscometer and a specially designed drop-on-demand jetting device with visualization capability. The results indicate that varying the Trouton ratio or extensional viscosity (by adjusting variables like the molecular weight and polydispersity) strongly influences the jetting behavior. A polymer solution having a Trouton ratio of three can be easily detached from the jet port but tends to form satellite drops. In contrast, a large Trouton ratio ($\gg 4$) results in a long tail that is harder to separate from the jet port. A Trouton ratio within a narrow range ($3 <$ Trouton ratio $< \sim 5$) results in the formation of uniformly sized spherical drops that are connected to the jet port by a short tail that retracts into the nozzle upon separation. Thus, slight elasticity corresponding to a Trouton Ratio around four is optimum for the jetting process. These findings are consistent with studies on breakup of viscoelastic jets reported in the literature. This appears to be the first quantitative report which provides such simple design guidelines.

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