Appendix A

AUTOMATING ANALYSIS OF DRIPPING-ONTO-SUBSTRATE EXTENSIONAL RHEOMETRY

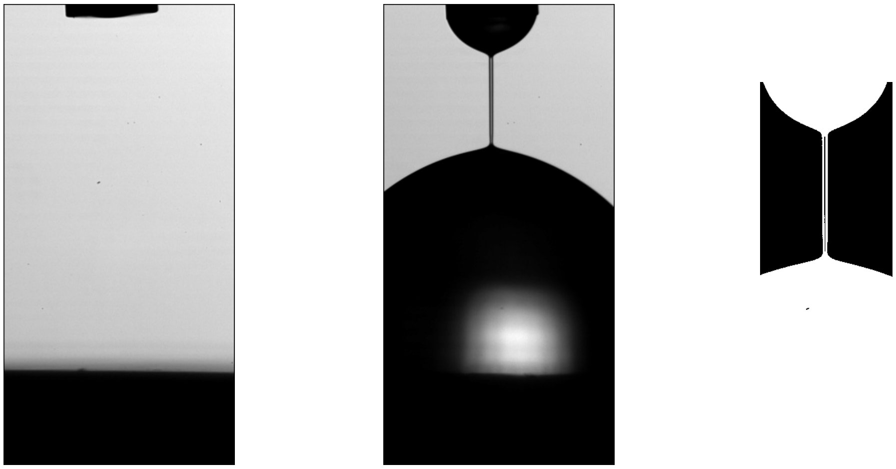
The automation techniques dicussed in this chapter were pioneered by Robert Learsch, and then formalized in the python package *dosertools*, which was co-developed by Robert Learsch and Red Lhota. The *dosertools* package is hosted at https://github.com/rlearsch/dosertools, along with documentation, and is installable via *pip install dosertools* for use with Python 3.8+.

A.1 Premise

Dripping-onto-substrate extensional rheometry (DoSER), as introduced in Chapter 1 is a form of capilliary break-up rheometry where a fluid is dripped from a nozzle onto a substrate and the resulting liquid bridge is observed via a high-speed camera. The diameter of the liquid bridge as a function of time is extracted from these videos and analyzed to determine key quantitative information about the fluid. Prior literature completed several steps in this analysis via user inspection; through the *dosertools* package, we sought to automate each step of the processing to reduce user-to-user variation and improve our reproducibility.

A.2 Background Subtraction and Binarization

Because light sources and cameras are not always uniform in their ability to produce and observe (respectively) light, we use background subtraction to reduce the impact of noise and other features of a non-uniform background (such as particulates on a lens or a dead pixel) on our processed images and thus



(a) Background (b) Experimental (c) Binary

Figure A.1: Example (a) background, (b) experimental, and (c) binarized images for a 6M PEO, 0.0066 wt% in diionized water solution.

our diameter data. Our recommended best practice is to capture a 100 frame background video of the nozzle in its experimental position above a substrate with both a clean nozzle and substrate (Figure A.1(a)). In our experiments, it was sufficient to capture a single background video for a group of 5 experimental runs; however, if the light source and/or camera varies on shorter time scales, we recommend a background paired with each experimental video (Figure A.1(b)).

To perform the background subtraction, the *dosertools* package uses the median of the background frames, crops both the image and the background, subtracts the background median from the image, then rescales the background subtracted image based on the maximum pixel value.

After background subtraction, the images are binarized using the Otsu threshold method. Compared to the literature method, where an arbitrary cutoff value for binarization is chosen by the user,[1–5] the Otsu threshold does not require a user determined cutoff as it uses an algorithm to decide the cutoff that creates the two most distinct "bins" for the image.

After binarization, pixels where either the fluid, the nozzle, or the substrate are visible will be white, while the surrounding background will be black (Figure A.1(c)).

A.3 Liquid Bridge Diameter

To process the binary images into normalized diameter data, the *dosertools* package determines the diameter of the liquid bridge at all heights, then extracts the minimum diameter. In addition, the nozzle diameter is determined from either the background or experimental video, depending on which video has an image of a clean nozzle.

First, *dosertools* determines if the liquid bridge has already pinched off by looking for rows where no white pixels are present. If the liquid bridge is still intact, the minimum diameter is then computed as the average diameter of all diameters within 2 pixels of the absolute minimum. This averaging alleviates observed problems with stair stepping in the diameter with time due to the finite size of a pixel. By taking the average with similar rows, we obtain a better estimate of the minimum diameter. This minimum diameter (D) is divided by the nozzle diameter (D_0) determined earlier to obtain the normalized diameter (D/D_0) (Figure 1.8).

The frames-per-second reported for the video is used in conjunction with the frame number to determine the time (t) for each frame.

A.4 Determining the Critical Time

Core to the principle of DoSER is quantifying the slope of the elastocapillary (EC) regime to find the extensional relaxation time. To do so, and to fairly compare videos which may have different relative start and end times depending on user choices of when to cut videos, the critical time of transi-

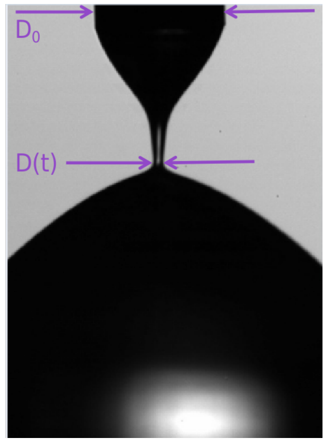


Figure 1.8: Example DoSER image showing the needle diameter D_0 and the minimum diameter of the liquid bridge $D(t)$. (repeated from page 16)

tion from either the inertio-capillary or the visco-capillary regime to the EC regime is used to align the normalized diameter data. Prior literature determined the critical time by inspection.[1–5] Robert Learsch developed a method for detecting the critical time through finding the moment of maximum strain rate within the window of normalized diameter in which transition occurs, which was implemented in the *dosertools* package. By finding the critical time systematically rather than by inspection, we significantly reduced user-to-user variation in analysis.

The *dosertools* determines the instantaneous measured strain rate via Equation A.1. An example strain rate curve is shown in Figure A.2. Given a window of normalized diameter values, it then finds the maximum strain rate in that window and specifies the time at the maximum strain rate as the critical time (t_c). Limiting the lower bound for the window of possible critical time addresses issues of noise in the data as the liquid bridge shrinks in diameter and becomes more difficult to accurately measure.

$$\dot{\epsilon} = \frac{-2(\frac{D/D_0}{dt})}{D/D_0} \tag{A.1}$$

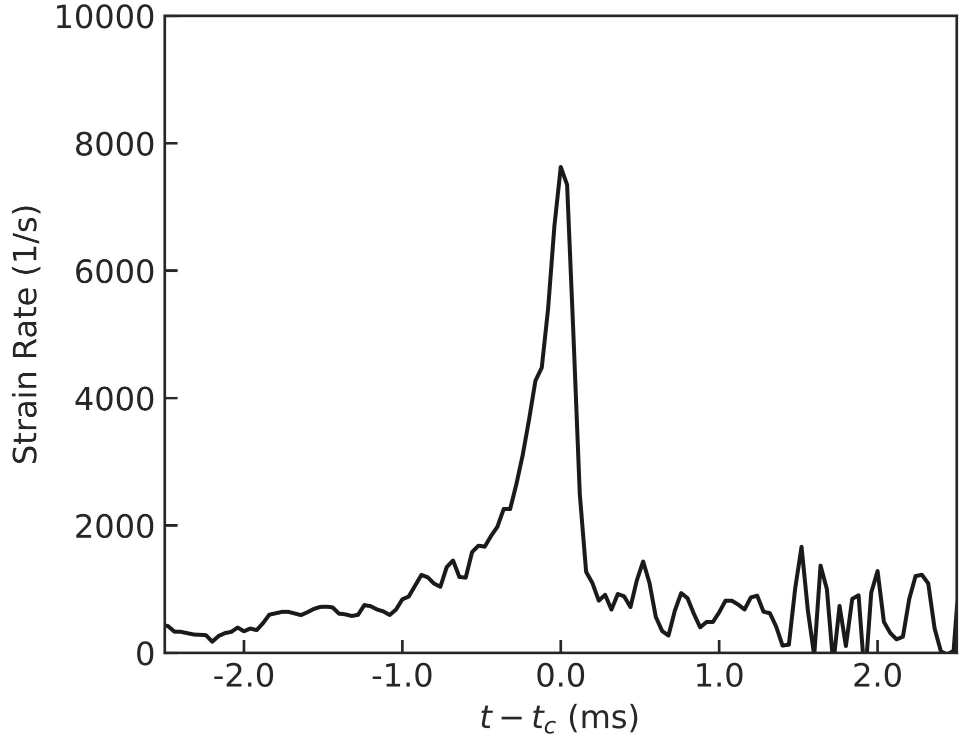


Figure A.2: Example of strain rate curve from a DoSER run.

A.5 Extensional Properties

Once the critical time has been determined, the EC regime (Equation 1.6) will consist of the data after the critical time and before the finite-extensibility regime (Figure 1.6). To determine the extensional relaxation time (λ_E), a linear regression is performed on data recast as $ln(D/D_0)$ versus $t - t_c$ (Equation A.2). The slope (m) obtained is thus used to calculate λ_E ($\lambda_E = -1/(3m)$).

$$\ln\left(\frac{D(t)}{D_0}\right) = A - \frac{1}{3\lambda_E}(t - t_c) \tag{A.2}$$

The process of determining the extensional viscosity of a solution from the liquid bridge diameter as a function of time will be discussed as part of Robert Learsch's thesis.

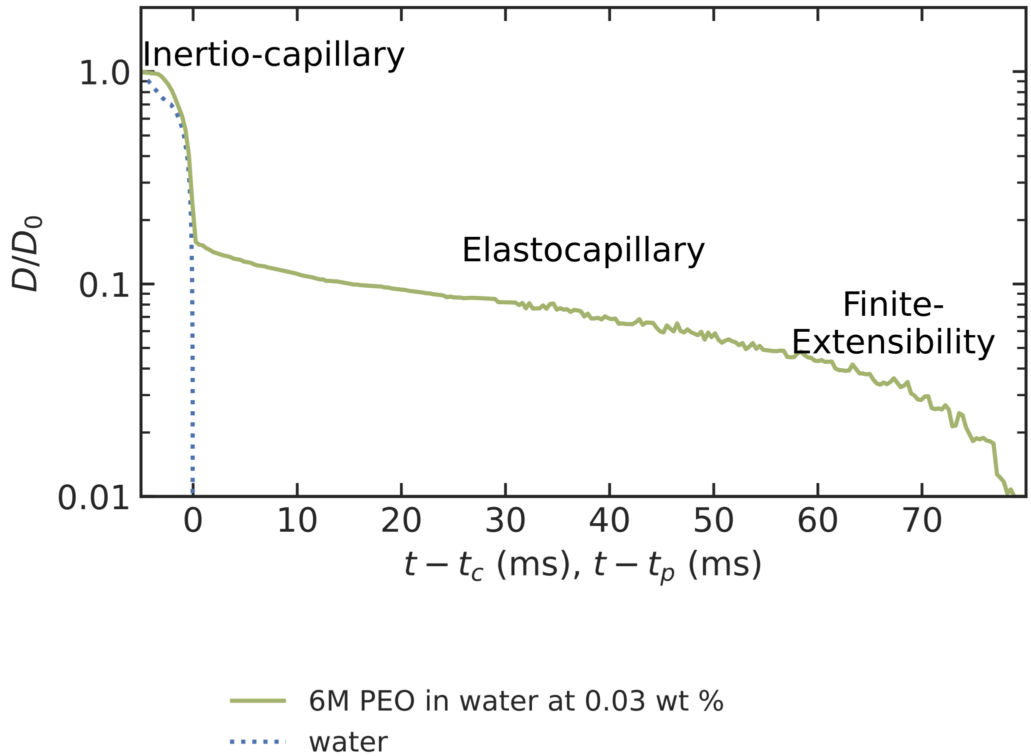


Figure 1.6: Example of measured capillary thinning for a polymer solution compared to water, demonstrating transitions for the polymer solution from the inertio-capillary regime (corresponding to water alone) to the elastocapillary regime, followed by finite-extensibility. (repeated from page 13)

A.6 Next Steps

The *dosertools* package uses arbitrary cutoffs for the fitting of the elastocapillary (EC) regime–work is ongoing to detect the beginning of the EC regime and the beginning of the finite-extensibility regime; however, finite resolution of the camera may mean that the finite-extensibility may not occur within the observable time of the thread, and the initial transition to EC does not immediately reach a exponential decay, inhibiting automation of this step. Additionally, the majority of data tested with *dosertools* has been from a single instrument–testing under other conditions and with other equipment will determine how robust the automation process is to alternate conditions.

References

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