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Errors in parallel-plate and cone-plate rheometer measurements due to sample underfill

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Abstract
The effect of sample underfill on parallel-plate and cone-plate rheometers is examined. Sample underfill can be caused by incomplete filling of a sample or loss of fluid during a test by, for example, evaporation. It is shown that even a small degree of sample underfill can lead to significant errors in measuring viscosity. A method is proposed to reduce these errors by directly monitoring the sample radius over the full course of the test. It is shown that the accuracy of the rheometer even while testing simple fluids like water is greatly improved.

Keywords: rheometer, sample underfill, evaporation

1. Introduction

Errors in parallel disk rheometry can arise from many sources, such as uncertainties in the gap size [1], lack of parallelism in the plates [2], viscous heating effects [3], and wall-slip errors [4]. However, the two main failures in rotational rheometers are considered to be edge failure [5, 6] and radial migration, both associated with the dynamics of the interface at the rim [7]. Here, we address an additional interfacial error: sample underfill, a condition where the sample boundary radius is less than that of the disk and the volume between the disks is not entirely filled.

One of the causes of sample underfill is evaporation. It is possible to avoid the effects of evaporation by creating an enclosure with a saturated environment [8], but this technique can cause condensation if cold-spots are present [9]. Schweizer [10] addressed this problem by manufacturing a partitioned measuring head consisting of a central stem surrounded by an annulus, where only the torque on the stem is measured and the surrounding annulus limits any effects of under-filling. As a consequence, measurements may be achieved without knowing the exact radius of the sample, as long as the radius is much larger than that of the stem. This method is unsuitable for low viscosity systems and polymer solutions where the small gap between the stem and annulus is penetrated by fluid, causing additional friction. The proposed design is also limited to rheometers with a non-displacing force measuring cell, since the sensing area of the stem must not move relative to the fixed annulus [10].

Here, we address the errors due to under-filling in measuring the viscosity of homogeneous liquids. We show that these effects can be significant for both parallel plate and cone plate geometries, and propose a method to account for such errors. The proposed method is suitable for any parallel plate or cone plate rheometer where there is visual access to the sample.

2. Analysis

A general derivation of the relationship between the torque T and the shear stress is given by Macosko [6]. Since the apparent shear stress is affected by the degree of sample underfill, we extend this derivation to relate the torque to the apparent viscosity. The torque measured by a rheometer is given by:

$$T = 2\pi \int_0^R \tau r^2 \mathrm{d}r \qquad (1)$$

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where τ is the local, axisymmetric, shear stress, r is the radial distance from the center of rotation, and R is the sample radius. For a fluid with a linear velocity profile between the upper and lower plates, the shear stress can be described as:

$$\tau = \mu \frac{\partial U}{\partial y} = \mu\omega \frac{r}{h(r)} \tag{2}$$

where μ is the apparent viscosity, U is the azimuthal velocity, y is a rotational axial coordinate and $\partial U/\partial y$ is the local velocity gradient across the gap, ω is the angular velocity, and $h(r)$ is the local gap height. For the parallel-plate and cone-plate geometries,

$$h_p = \alpha_p \tag{3a}$$

$$h_c = \alpha_c + \beta_c r, \tag{3b}$$

where α and β are constants, and the subscripts p and c indicate parallel-plate and cone-plate geometry, respectively. The total torque is then given by

$$T_p = 2\pi\mu\omega \int_0^R \frac{r^3}{\alpha_p} \mathrm{d}r = \frac{1}{2}\frac{\pi\mu\omega}{\alpha_p} R^4 \tag{4a}$$

$$T_c = 2\pi\mu\omega \int_0^R \frac{r^3}{\alpha_c + \beta_c r} \mathrm{d}r = 2\pi\mu\omega \left[\frac{1}{3\beta_c} R^3 - \frac{\alpha_c}{2\beta_c^2} R^2 + \frac{\alpha_c^2}{\beta_c^3} R - \frac{\alpha_c^3}{\beta_c^4} \ln\left(1 + \frac{\beta_c}{\alpha_c} R\right) \right] \tag{4b}$$

(see also Lee *et al* [11]).

For a typical cone-plate rheometer, $R \gg \alpha_c/\beta_c$, and only the leading order term need be retained in equation (4*b*). The viscosity is then found according to

$$\mu_p = \frac{2}{\pi} \frac{T_p \alpha_p}{\omega R^4} \tag{5a}$$

$$\mu_c = \frac{3}{2\pi} \frac{T_c \beta_c}{\omega R^3} \tag{5b}$$

where R is the liquid boundary radius, being R_0 in the fully-filled case and $R < R_0$ when underfilled. The resulting relative error, η, in the apparent viscosity can be written as

$$\eta_p \equiv \frac{\mu_p}{\mu} - 1 = \frac{T_p}{T_0} \left(\frac{R_0}{R} \right)^4 - 1 \tag{6a}$$

$$\eta_c \equiv \frac{\mu_c}{\mu} - 1 = \frac{T_c}{T_0} \left(\frac{R_0}{R} \right)^3 - 1, \tag{6b}$$

where μ_p and μ_c are the measured apparent viscosities, and subscript 0 indicates a value for a properly filled setup. We investigate how under-filling affects the viscosity measured by an operator who assumes erroneously that $R = R_0$. In this case, the relative viscosity error can be written as

$$\tilde{\eta}_p \equiv \frac{\tilde{\mu}_p}{\mu} - 1 = \frac{T_p}{T_0} - 1 = (\eta_p + 1) \left(\frac{R}{R_0} \right)^4 - 1 \approx -4\delta \tag{7a}$$

$$\tilde{\eta}_c \equiv \frac{\tilde{\mu}_c}{\mu} - 1 = \frac{T_c}{T_0} - 1 = (\eta_c + 1) \left(\frac{R}{R_0} \right)^3 - 1 \approx -3\delta, \tag{7b}$$

where the final approximation is found using the binomial theorem for small η and changes in radius, $\delta = (1 - R/R_0)$. By measuring the radius R simultaneously with the torque, equations (5) can be used to estimate the true viscosity.

3. Experimental procedure

The effects due to under-filling were measured using an Anton-Paar GmbH. Physica MCR 301 rheometer with a torque sensor accuracy of 0.2 μN m or 0.5%, whichever is more conservative (as given by the manufacturer). The rheometer has two discs separated by a fluid-filled gap where the lower disc is stationary and temperature controlled to be 20 ± 0.2 °C. The upper disc rotates at a prescribed angular velocity and uses an air bearing to minimize friction. The experiment was conducted using two different configurations. First, a 49.966 mm diameter parallel plate configuration (PP50) was used with a 1 mm gap height. Second, a cone plate configuration (CP50-1/TG) was used with a 49.962 mm diameter and a 0.979° cone angle. The cone tip is truncated by 49 μm to allow the virtual tip of the cone to coincide with the surface of opposing plate without added friction, giving $\alpha_c = 0$ in equation (3*b*).

The tests were conducted for three pure fluids: water, ethanol, and isopropyl alcohol. The volume between the plates was first completely filled by the sample fluid. The experiment was then commenced, and the sample radius was altered over the course of the experiment by allowing the fluid to evaporate. Evaporation was the sole method by which the sample volume was altered, and the fluids were chosen such that their properties remained insensitive to evaporation. The radius of the sample R was recorded using a Nikon D7100 SLR camera with an AF-S Micro Nikkor 85 mm lens, which has a resolution of 9.95 μm/pixel. The experiments involving water were carried out for 45 min while an image was taken every minute. For the more volatile fluids, ethanol and isopropyl alcohol, the experiments were performed over a period of 10 min with an image taken every 10 s. Figure 1 shows the experimental setup for a parallel plate with water as the sample fluid. Figure 1(*a*) is the initial condition and figure 1(*b*) shows the sample after 45 min where the radius has decreased by approximately 3% . The sample radius was determined by measuring the distance between the waists of the two opposing menisci to the center of the rheometer setup, resulting in a left and right radius (R_L, R_R). The radial migration was monitored by the change in the ratio R_L/R_R between two consecutive images. Once this difference exceeded 0.3%, all remaining data in the time series were disregarded.

4. Results and discussion

Figure 2 shows the percentage deviation in the apparent viscosities (η and $\tilde{\eta}$) as a function of the change in the sample radius, δ. The three data sets for water, ethanol, and isopropyl alcohol

Figure 1. Rheometer setup for a parallel plate head, (*a*) properly filled sample, (*b*) under-filled sample.

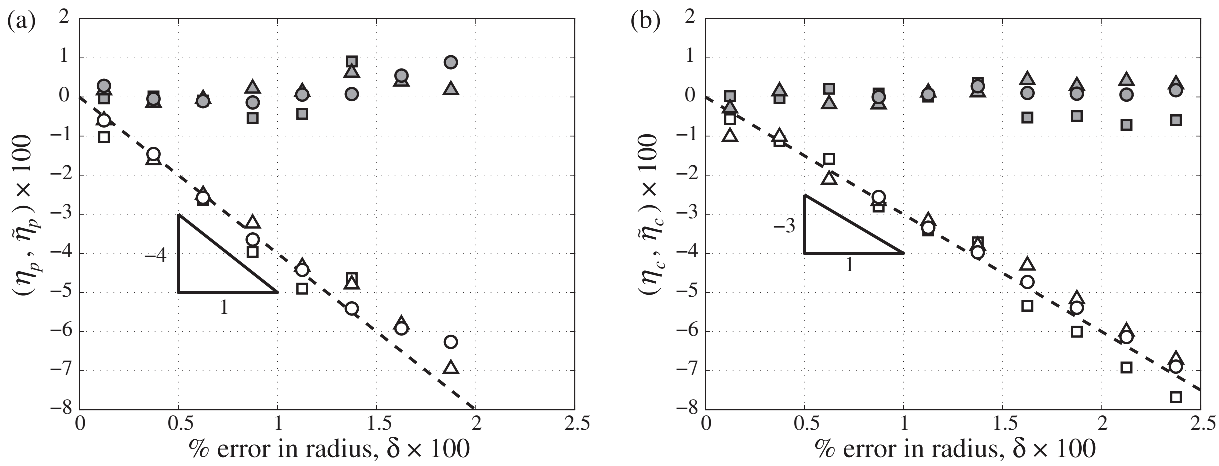

Figure 2. Percent error in measured viscosity for increasing error in radius, $\delta = (1 - R/R_0)$. (*a*) Parallel plate (η_p and $\tilde{\eta}_p$, see equations (6*a*) and (7*a*); (*b*) cone plate (η_c and $\tilde{\eta}_c$, see equations (6*b*) and (7*b*). Here, □, Δ and ○ represents water, ethanol and isopropyl alcohol, respectively. Open symbols represent $\tilde{\eta}$, assuming constant radius R_0. Filled symbols, η, use instead the wetting radius R, as recorded by the camera. Dashed lines show the expected behavior for small errors.

were post-processed using the two different methods shown in equations (6) and (7). We note first that the results for the three different fluids agree well, showing a fluid-independent behavior. Second, the errors due to under-filling follow the expected behavior for small deviations. Most importantly, the differences between η and $\tilde{\eta}$ represent the errors introduced in the measurement of viscosity by neglecting the effects of under-filling. Specifically, we see that in our experiments on water, ethanol and isopropyl alcohol, up to 7% bias errors were incurred due to the change in radius of the sample during the tests. We also see that by accounting for this under-filling by using the measured sample radius eliminates the bias error, leaving a random error in the viscosity of less than ± 1%. The significance of under-filling can also be demonstrated by noting that a 100 μm change in the radius of the sample (corresponding to a 0.2% variation) will cause a 1.6% error in the apparent viscosity for a parallel plate setup, and 1.2% for a cone plate setup.

Note that the method presented here is limited to cases of under-filling with centered sample placing. For uncentered sample placing, or radial migration, parts of the sample is displaced towards areas with higher shear stress, resulting in a higher measured torque.

5. Conclusions

Small changes in the radius of a rheometer sample can cause significant errors in the measured apparent viscosity. These errors can be accounted for by adopting the following procedure, which can also be used to verify the initial sample radius and to estimate the effects due to under-filling. First, image the sample and estimate the true radius from the image. Second, if there is under-filling, either caused by evaporation or other means, verify that the sample remains centered by comparing the meniscus recession at both sides of the image. Third, use equation (5) to calculate the correct viscosity using the measured sample radius. Note that most rheometers keep the angular velocity constant, not the shear rate.

Although we have assumed a Newtonian stress, our analysis could be simply extended to examine, for example, the viscoelastic properties of polymers using small amplitude oscillatory shear measurements.

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