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Measuring rheological properties using a slotted plate device

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Abstract

The slotted plate technique has previously been shown to be a successful method for directly measuring the static yield stress of suspensions. In this study, we further establish the usefulness of the slotted plate device as a rheometer especially at low shear rates, taking advantage of the extremely low speeds of the slotted plate technique. Newtonian fluids, a shear thinning fluid, and yield stress fluids were tested using the slotted plate device and the results were compared with those from a commercial rheometer using different standard flow geometries. The relationship between the stress on the plate and the viscosity for the slotted plate device obtained by dimensional analysis (drag) predicts a linear relationship between the force at the plate and the plate speed, consistent with the experimental data. The slotted plate device can measure viscosities at very low shear rates. The apparent viscosity – shear-rate data obtained from the slotted plate device are complementary to those obtained using a commercial rheometer. That is: the slotted plate can measure viscosity in the shear rate range $10^{-7} < \dot{\gamma} < 10^{-3}$ s^{-1}, while the commercial rheometer measures viscosity at shear rates higher than 10^{-3} s^{-1}.

Keywords : slotted plate device, rheological properties

1. Introduction

The slotted plate technique has been successfully demonstrated to be a sensitive method to determine a static yield stress of suspensions (Zhu *et al.*, 2001). The slotted plate device was constructed with a balance and a linear-motion platform to directly measure the yield stress by moving the plate in the suspension in a similar mode as with the well-known Wilhelmy-plate technique for surface tension measurements. Wall effects associated with the original plate yield-stress instrument (De Kee *et al.*, 1980; De Kee *et al.*, 1986; De Kee and Chhabra, 1994) were minimized by opening a series of slots on the plates (Zhu *et al.*, 2001). The slotted-plate technique does not rely on an assumed yield surface, as is the case with the vane technique, a popular method employed in the direct yield stress measurement (Nguyen and Boger, 1983; Nguyen and Boger, 1985), and is more reliable for evaluating small yield stress values. The plate method is not associated with secondary flow problems and the stress distribution on the plate's surface is uniform. It can be used for suspensions at both high and low concentrations.

Conventional rheological instruments employed for viscosity measurements include plate-plate, cone-and-plane, and Couette-type rheometers. When dealing with many industrial products exhibiting complex behavior, wall-sample interactions can result in slip effects or in "stress reduction", especially at very low shear rates. The probability of wall slip increases when dealing with smooth walls, small flow dimensions, low flow rates, concentrated solutions of high molecular weight polymers, suspensions of large or flocculated particles, and emulsions of large droplets (Meeker *et al.*, 2004; Barnes, 1995). The low viscosity of the liquid near the wall leads to higher measured shear rates at a fixed shear stress, and therefore the viscosity and yield stress are under-predicted (Nguyen and Boger, 1992). A common technique to avoid such complications is to roughen the surface in order to increase the friction between the suspension and the wall (Vinogradov *et al.*, 1975; Kao *et al.*, 1975; Magnin and Piau, 1990). The wall can also be covered or chemically treated to promote adhesion between the particles and the wall (Princen, 1985). With the slotted plate device, the openings created on the plate surface effectively allow the material to shear within itself at the slot-sample interface, thus preventing wall slip to occur. (Zhu *et al.*, 2001).

Since the slotted plate technique is not associated with

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secondary flow problem, it has a potential use as a rheometer. In this study, we established that the slotted plate technique can also be employed as a rheometer for flow property measurements especially at very low shear rates, taking advantage of the extremely low speed motion of the slotted plate device. Newtonian fluids, a shear thinning fluid, and yield stress fluids were tested using the slotted plate device and the results obtained were compared with those from a commercial rheometer.

2. Experiments

2.1. Materials

Silicone oil and glycerol were used as Newtonian fluids. Silicone oil (Aldrich, MW = 136.22, $\eta = 0.1$Pa.s and $\rho = 967$ kg/m^3 at 20°C) and glycerol (Aldrich, MW = 92.09, $\rho = 1262$ kg/m^3 at 20°C) were used as received.

A 1.45 wt.% polyacrylamide solution was used as a shear thinning fluid. Polyacrylamide (Acros, MW = 5–6×10^6, $\rho = 1302$ kg/m^3) was dispersed in a 40/60 mixture by weight of water and glycerol by stirring at low speed with a magnetic bar for a week.

Carbopol 980 aqueous dispersions were used as yield stress fluids. Aqueous Carbopol dispersions (0.08 and 0.09 wt.%) were prepared according to Noveon TDS-103 (Noveon, 1993) and neutralized to pH 7 according to Noveon TDS-237 (Noveon, 1998).

2.2. Measurements with the slotted plate device

Details of the slotted plate device, including a linear motion platform driven by a step motor and a balance connected to a computer for data analysis, were described in Zhu *et al.* (2001). The plate used was made of stainless steel and had dimensions of 30×30×0.6 mm. With the slotted plate, the openings created on the plate surface effectively allow the sample to shear within itself at the slot-sample interface, thus preventing wall sip to occur. To prevent wall slip, a slotted plate with a slot area to plate area ratio of 0.67 was employed (Zhu *et al.*, 2001). To strengthen that the sample filling the slots is static, with no secondary flow and with shearing occurring only at the edge of the slots, the ratio of the height of a slot over the thickness of the plate was made smaller than 3. The plate was hung from the balance through a very thin stainless steel wire (0.127 mm in diameter). The velocity of the plate varied from 0.003 to 60 mm/min and was precisely set by controlling the speed of the platform through a controlling computer.

All measurements were performed at room temperature (25°C). The sample was first loaded in a 400 ml beaker (70 mm high and 90 mm in diameter) and allowed to equilibrate. The plate and wire were sample coated by repeated dipping before being vertically inserted into the sample. The beaker was placed on the platform, the plate was suspended from the balance and, the plate was allowed to reach equilibrium. Following this procedure the platform was lowered at a controlled and constant rate. In this experimental setup, as the platform is lowered the sample in the beaker exerts a force at the plate. The plate in turn exerts a force on the balance, and this continuously changed force is recorded. Measurements were performed at various controlled constant plate speeds to obtain the sample's rheological properties as a function of shear rate.

2.3. Measurement with a rheometer

Rheological measurements were also performed at 25°C using a TA Instruments AR-2000 rheometer. The measuring flow geometries employed include a 40 mm cone and plate (cone angle 1°, gap 0.028 mm), double concentric cylinder (rotor outer diameter 21.96 mm, rotor inner diameter 20.38 mm, stator inner diameter 20.0 mm, rotor length 59.5 mm) as well as a vane-cup attachment (vane diameter 14 mm, vane length 42 mm, cup diameter 15 mm). A solvent trap was used to prevent sample evaporation during measurements.

3. Results and discussion

3.1. Measurement using the slotted plate device

In the slotted plate device, the platform was designed for extremely low speed motion to produce very slow movement of the slotted plate, allowing for the determination of the sample's rheological properties at very low shear rates. A typical force response recorded as a function of time is presented in Fig. 1 for silicone oil tested at a plate speed of 0.014 mm/s. The measured force is the reading taken at the balance when the plate is moving. The measured force increases rapidly and reaches steady state. This behavior is typical for various different plate speeds and all other samples (Newtonian and non-Newtonian fluids). The steady

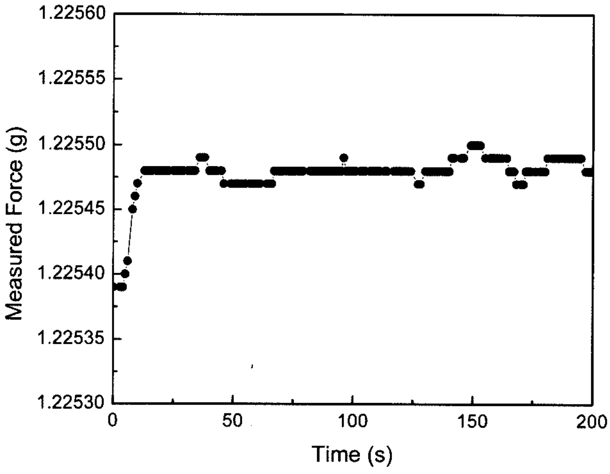

Fig. 1. Measured force at the balance as a function of time for silicone oil. The plate speed is 0.014 mm/s.

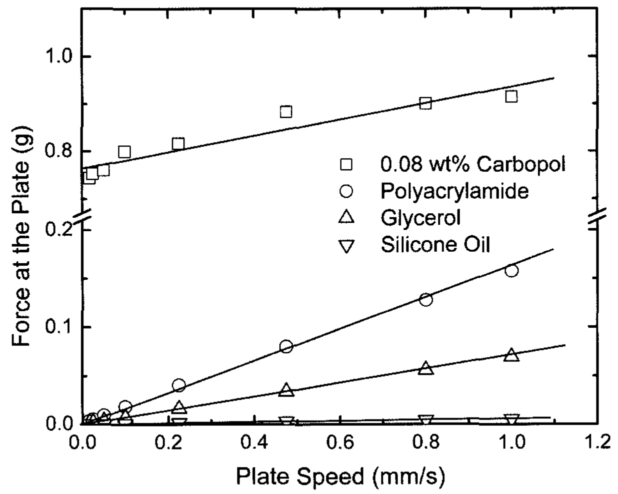

Fig. 2. Force at the plate as a function of plate speed for various samples.

state value was used to obtain the sample's viscosity at the corresponding plate speed.

The steady state measured force, F, is the reading at the balance and has to be corrected by subtracting the initial force, F_i, to obtain the force at the plate, F_z.

$$F_z = F - F_i \tag{1}$$

where, F_i is the combined plate and wire weight less the buoyant force.

The force at the plate as a function of the plate speed is presented in Fig. 2 for various samples. The results for silicone oil and glycerol (Newtonian fluids) show that there is a linear relation between the force at the plate and the plate speed, and the lines connecting data points at different plate speeds extrapolate to the origin, indicating the absence of yield stress. The linear relation between the force at the plate and the plate speed suggests that the slotted plated device can be employed as a good viscosity measurement tool.

The data for the polyacrylamide solution also shows a linear relation between the force at the plate and the plate speed, similar to the observations with the Newtonian fluids. The reason for the linear behavior for the polyacrylamide solution, which is known to be a shear thinning fluid, will be discussed later.

In Fig. 2, data obtained for the 0.08 wt.% aqueous Carbopol dispersion displays the characteristics of yield stress fluid behavior, as indicated by a finite extrapolated force at zero plate speed. The force intercept can be considered to be a measure of the dynamic yield stress of the Carbopol dispersion.

3.2. Data analysis for the slotted plate method

The stress on the plate can be computed from

$$\sigma_z = \frac{F-F_i}{\text{plate area}} = \frac{F_z}{\text{plate area}} = \frac{F_z}{2LW} \tag{2}$$

where σ_z is the stress associated with the plate surface, L is the length of the plate, and W is the plate width. To obtain the sample's viscosity from σ_z data at the corresponding plate speed, which is related to the shear rate, we employ the dimensional analysis.

The dimensional analysis (drag) over a submerged body gives

$$F_z = \frac{1}{2} C_D \rho v_s^2 A \tag{3}$$

where C_D is the drag coefficient, v_s is the plate speed, and A is the surface area of the submerged body. For the slotted plate, the area A is $\sim 2LW$. In the slotted plate device, since the platform speed is extremely low, the Reynolds number is consequently very small; i.e., $\text{Re} = L v_s \rho / \eta < 1$. The drag coefficient can thus be presented as a function of the Reynolds number, analogous to creeping flow over a sphere, i.e.,

$$C_D = k/\text{Re} \tag{4}$$

where k is a constant which depends on the geometry of the plate.

The force at the plate is then given by

$$F_z = k \eta v_s W \tag{5}$$

where η is the apparent fluid viscosity.

The result is consistent with the data obtained for Newtonian fluids shown in Fig. 2, which displays a linear relation between F_z and v_s. Wu and Thompson (1996) showed that a linear creeping flow model could predict drag coefficients for shear thinning fluids flowing past a flat plate. The constant, k, was determined to be 17.5.

To estimate the shear rate corresponding to the plate velocity, the boundary-layer thickness of the slotted plate is assumed (as for the boundary-layer theory) to be:

$$\delta = 4.64 \sqrt{\frac{\eta z}{\rho v_s}}. \tag{6}$$

The average boundary-layer thickness over the plate length, L, is

$$\delta_{avg} = 3.093 \sqrt{\frac{\eta L}{\rho v_s}} \tag{7}$$

and therefore, the shear rare at the plate speed, v_s, is

$$\dot{\gamma} = \frac{v_s}{\delta_{avg}} = \frac{v_s}{3.093}\sqrt{\frac{\rho v_s}{\eta L}}. \tag{8}$$

The experimental data from the slotted plate device were analyzed using Equations (5 and 8) to determine the apparent viscosity data as a function of shear rate. Viscosity as a function of shear rate is presented in Fig. 3 for the var-

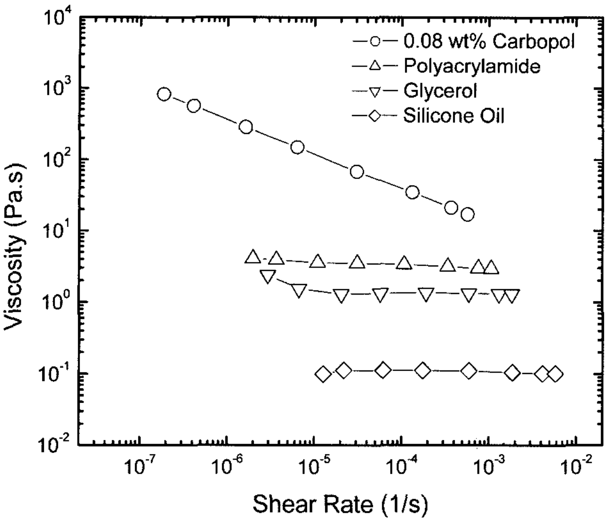

Fig. 3. Viscosity as a function of shear rate for various samples.

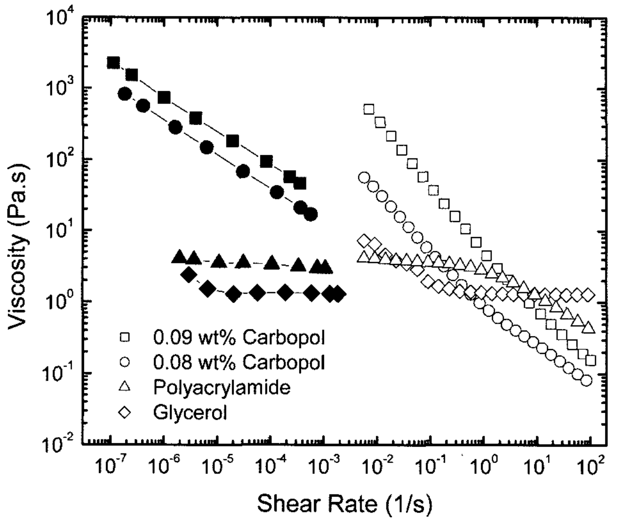

Fig. 4. Viscosities from the slotted plate device and the AR-2000 as a function of shear rate for various samples. The filled symbols refer to the slotted plate device and the open symbols are data from the AR-2000 rheometer.

ious samples tested. The viscosity of Newtonian fluids (silicone oil and glycerol) is constant over the entire shear rate range, confirming that the slotted plate device can be used to measure rheological properties, especially at very low shear rates. The measured apparent viscosity of the polyacrylamide solution, a shear thinning fluid, is also constant. We believe that the constant viscosity of polyacrylamide is likely the zero shear viscosity, considering the very low shear rates at which the viscosity was obtained. This will be further discussed in the next section with the apparent viscosity data from the commercial rheometer. The apparent viscosity of the aqueous Carbopol dispersions decreases with shear rate, over a shear rate range of ~10^{-7} s^{-1} to 10^{-3} s^{-1}. The results in Fig. 3 demonstrates that the slotted plate device can be used as a rate-controlled rheometer for measuring viscosities at very low shear rates (~10^{-7} s^{-1} for aqueous Carbopol dispersions) due to its ability to control the platform speed to extremely low rates.

3.3. Comparison with commercial rheometer data

The rheological properties (apparent viscosities) of the various samples studied also were measured with an AR-2000 rheometer using different geometries for comparison with the data from the slotted plate device. Viscosity data obtained using the slotted plate device and AR-2000 as a function of shear rate are presented in Fig. 4. For glycerol, the slotted plate device shows a constant viscosity at shear rates as low as ~10^{-5} s^{-1}, but the AR-2000 using a cone and plate geometry (1°, 40 mm) shows the same constant viscosity only for $\dot{\gamma} > 10^{-1}$ s^{-1}. For lower shear rates, the measured viscosity from the AR-2000 appears to increase with decreasing shear rate. The lowest shear rate obtainable with the AR-2000 is around 10^{-3} s^{-1}, which is quite large compared to the slotted plate device capability.

For the 1.45 wt.% polyacrylamide solution (shear thinning fluid), the slotted plate device measures a constant viscosity for $10^{-6} < \dot{\gamma} < 10^{-3}$ s^{-1}. Measurements using the AR-2000 using a cone and plate geometry (1°, 40 mm) gives a constant viscosity of similar magnitude for ~$10^{-3} < \dot{\gamma} < 10^{-1}$ s^{-1}, followed by a viscosity which decreases with shear rate for $\dot{\gamma} > 10^{-1}$ s^{-1}. Thus the constant viscosity measured using the slotted plate device indeed represents the zero shear viscosity of the polyacrylamide solution.

In Fig. 4, the data obtained for the 0.08 and 0.09 wt.% aqueous Carbopol dispersions using the slotted plate device and the AR-2000, using a cone and plate geometry (1°, 40 mm), show similar behavior in apparent viscosity, which decreases with increasing shear rate. The slotted plate device measures the viscosity in the shear rate range of 10^{-7} to 10^{-3} s^{-1}, while the AR-2000 measures the viscosity for $\dot{\gamma} > 10^{-3}$ s^{-1}. Over a shear rate range of 10^{-2} to 10^{1} s^{-1}, corresponding to a viscosity range of 10^{1} to 10^{3} Pa.s, a significant difference in the results between the two techniques can be observed. This inconsistency may be attributed to the yield stress behavior of the Carbopol solutions, and will be discussed next.

Fig. 5 shows the viscosity data as a function of shear stress for the aqueous Carbopol dispersions tested using the AR-2000 rheometer under the stress-controlled mode, using a 14 mm vane geometry. Our data agree well with those previously reported by Roberts and Barnes (2001) for dispersions of Carbopol 980. The viscosity of the aqueous Carbopol dispersions is constant at low stress values, but decreases dramatically with increasing shear stress to finally reach a constant value at higher shear stresses. The critical stress where the slope of the viscosity-shear stress plot is a maximum corresponds to the yield stress (Roberts

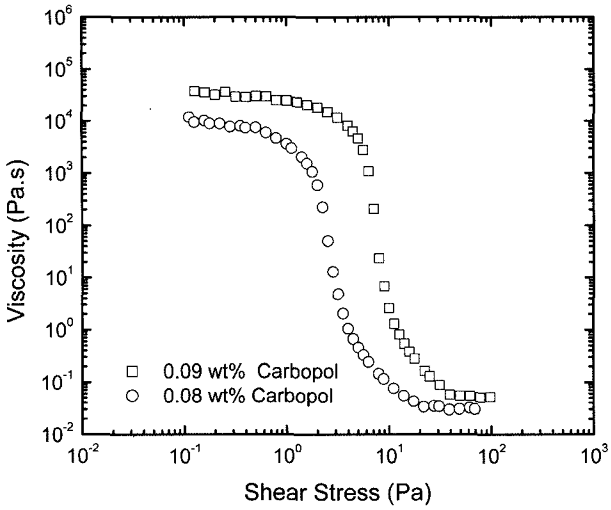

Fig. 5. Viscosity of Carbopol dispersions as a function of shear stress. The data were obtained with the AR-2000 using a vane geometry.

and Barnes, 2001). As seen in Fig. 5, this occurs in a viscosity region ranging from approximately 10^1 to 10^3 Pa·s, which coincides with the region where disagreement between the slotted plate data and the AR-2000 data exists (see Fig. 4). Thus in this region, a very small change in the shear stress during the measurement can cause a large change in viscosity. The inconsistency in the data from the AR-2000 in the range $\sim 10^{-2} < \dot{\gamma} < \sim 10^1$ s^{-1} shown in Fig. 4 seems to arise from the substantial change in viscosity with a very small change of shear stress (or shear rate). By comparison, the slotted plate device would provide more reliable and accurate measurements with yield stress fluids in the extremely low shear rate region down to 10^{-7} s^{-1}.

4. Conclusions

The slotted plate technique is a successful method to determine a static yield stress of suspensions since it does not rely on an assumed yield surface and is more reliable for evaluating smaller yield stress values. In this study, we also established the usefulness of the slotted plate device as a rheometer, especially at very low shear rates, by taking advantage of the extremely low speed motion of the slotted plate device. Rheological properties of various fluids (Newtonian, shear thinning, and yield stress fluids) were measured using the slotted plate device and their results were compared with those from a commercial rheometer. The relationship between the force at the plate and the viscosity for the slotted plate device is obtained by combining dimensional analysis (drag) with the idea of the boundary layer thickness. It predicts the linear relation between the force at the plate and the plate speed, consistent with the obtained experimental data. The apparent viscosity versus shear-rate data obtained from the slotted plate device are complementary to the data from a commercial rheometer. The slotted plate measures viscosities over the shear rate range $10^{-7} < \dot{\gamma} < 10^{-3}$ s^{-1}, while the commercial rheometer (AR-2000) measures viscosities for $\dot{\gamma} > \sim 10^{-3}$ s^{-1}. The slotted plate device also allows for the determination of a dynamic yield stress for yield stress fluids.

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