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Measurement of the Limiting Viscosity with a Rotating Sphere Viscometer

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Synopsis

The rotating sphere viscometer is found to be a superior method of measuring the viscosity at zero shear rate of moderately viscous non-Newtonian fluids. The effects of inertia and container walls are studied experimentally. The shear stress-shear rate relationship is found to be linear at the low shear rates studied; thus yielding the limiting viscosity. This measured value of the limiting viscosity can be used in empirical models that are applicable over a wide range.

Introduction

Although many fluids possess a viscosity that is dependent on the invariants of the rate of strain tensor, a constant value of the viscosity is generally approached at very low deformation rates. If the viscosity function is to be described over a wide range of deformation rates by an empirical model, it is necessary to know the limiting viscosity, η_0. Typical rotational viscometers give the shear stress-shear rate relationship for a range of about 10-1000 sec.$^{-1}$ in shear rate. In the upper portion of this range, many fluids are observed to follow power law behavior. That is,

$$\mathbf{\Gamma} = \beta(^1/_2\mathbf{p}:\mathbf{p})^{(\alpha-1)/2}\mathbf{p} \tag{1}$$

where $\mathbf{\Gamma}$ is twice the symmetric part of the velocity gradient, α and β are material constants, and $\mathbf{p}$ is the shear stress dyadic given by

$$\mathbf{S} = p\mathbf{I} + \mathbf{p} \tag{2}$$

where $\mathbf{S}$ is the stress dyadic; p, the pressure; and $\mathbf{I}$, the idemfactor. However, in the lower portion of this range the viscosity's dependence on deformation rate is not as strong as indicated by eq. (1), since the viscosity is now beginning to approach its limiting value. For many applications, such as flow in porous media, knowledge of the viscosity function for shear rates less than 10 sec.$^{-1}$ is required. One way of acquiring this information is to measure the limiting viscosity and use it along with the viscometric data to fit an empirical rheological model. This model can then be used with confidence in the 0-10 sec.$^{-1}$ shear rate range as well as over the entire viscometric range. One such model commonly used is the Ellis model (see following page);

$$\boldsymbol{\Gamma} = [\phi_0 + \phi_1(^1/_2\mathbf{p}:\mathbf{p})^{(\alpha-1)/2}]\mathbf{p} \tag{3}$$

where ϕ_0 is the reciprocal of the limiting viscosity and ϕ_1 and α are material parameters. The Ellis model has been used with success in describing flow past a sphere,[1] flow in annuli,[2] and other applications.[3]

Several methods have been used to determine η_0 (and thus ϕ_0). The simplest method is to fit eq. (3) to the standard viscometric data and use the "best-fit" value of ϕ_0. This procedure is acceptable if only shear rates over the viscometric range are encountered in a particular application. However, some doubt must be attached to its use for deformation rates below the viscometric range, since there is no assurance that the extrapolation is valid. An alternate procedure is to measure the limiting viscosity independently and to use this measured value (as ϕ_0^{-1}) to fit eq. (3) to the viscometric data. Bird and co-workers (see, for example, Turian[4]) have used a falling-sphere viscometer to measure the limiting viscosity. Corrections for bottom, wall, and inertial effects for this viscometer can be found in Brenner[5] and Turian.[4] Briefly, the method involves measuring the terminal velocity of falling spheres of small diameters in the fluid in question, correcting for the effects mentioned above, and extrapolating a plot of log η_0 versus $p_{r\theta}|_{\max}$ to zero shear stress. The chief inaccuracy of this method is that for practical purposes a minimum stress of about 30 dynes/cm.^{2} is obtainable (sphere diameter of 0.025 in.). Thus, except for very viscous fluids, the measured shear rate is too large for an accurate extrapolation.

Another method involves the use of a rotating sphere viscometer. While this instrument dates back to Stokes, Savins and Walters[6] appear to be the first to have suggested its use for measuring the limiting viscosity. Their study was primarily directed toward the measurement and observation of elastic effects at higher shear rates, however. This investigation studies in more detail the use of the rotating sphere device as a means of measuring the limiting viscosity. In particular, the experimental limits and accuracies are established.

Experimental

The basic measurement to be made is the couple acting on the sphere as a function of rotational speed. A plastic ball of diameter 5.430 cm. was the primary sphere used; however, steel balls of diameters 3.000 and 2.528 cm. were used for some measurements.

The couple measuring method is that used by Savins and Walters.[6] The sphere was connected to the dynamometer head of a Brookfield viscometer by means of steel shaft to diameter 0.0711 cm. A threaded connection secured the shaft to the sphere, while the other end of the shaft was hooked to a balanced clasp which, in turn, was fastened to the dynamometer head. A variable-frequency driver in conjunction with the Brookfield gear train allowed the ball to be rotated from 0.2 rpm to 120 rpm.

Three circular cylinders of diameters 21.3, 15.5, and 10.13 cm. were used as containers for the fluids. The heights of all cylinders were greater than their diameters. In this manner the bath/sphere diameter ratio necessary for the "infinite sea" assumption could be ascertained. In all cases, the sphere was mounted in the center of the container.

All fluid samples were also run on the Couette viscometer at the same temperature. A wider range of properties could be obtained from this measurement.

Glycerol solutions (with water) of 85, 91, and 96% were tested in all containers using all three of the spheres. In this manner the bath wall effects, inertia effects, and internal consistency of the results could be checked. Solutions of $^1/_4$, $^1/_4$, $^3/_4$, and 1% Natrosol 250H (a highly substituted hydroxyethyl cellulose marketed by Hercules Powder Company) in water were tested for the largest sphere in the largest bath.

Analysis and Results

The mathematical description of the physics of slow rotation of a sphere in an infinite sea of a Newtonian fluid about an axis containing a diameter is well known.[7] If the particle paths are assumed to be circles about the axis of rotation (i.e., $V_r = V_\theta = 0$, $V_\phi = r \sin \theta\omega$), a solution of the equations of change exists provided the nonlinear inertial terms and normal stress terms are negligible. This solution may be written as a relationship between the couple acting on the sphere C and the speed of rotation Ω.

$$C = 8\pi R^3 \eta_0 \Omega \tag{4}$$

It will be convenient later to rewrite eq. (4) in terms of the Reynolds number, N_{Re}

$$\begin{aligned} C &= (8\pi R \eta_0^2/\rho)(\rho R^2 \Omega/\eta_0) \\ &= (8\pi R \eta_0^2/\rho) N_{\mathrm{Re}} \end{aligned} \tag{5}$$

Since all fluids approach Newtonian behavior at very low shear rates, the above equations should apply to these fluids at low speeds of rotation. Thus, a plot of C versus Ω should be linear and the slope can therefore be used to find η_0.

First, it was necessary to check for the effects of the containing cylinder's walls. Brenner and Sonshine[8] have solved the equations describing slow viscous rotation of a sphere in a cylindrical container. Experiments were performed using glycerol solutions for radii ratios from 2.05 to 11.2. For ratios higher than about 3.5 the instrument accuracy was not sufficient to perceive the effect of the cylinder wall. At lower values of the radii ratio, torque deviations in good agreement with those predicted by Brenner and Sonshine were observed. For example, the measured viscosity of the 91% glycerol for a radius ratio of 2.05 was found to be 9.1% higher than the mean of the measured values for radii ratios greater than 3.5. This mean value of viscosity was in good agreement with handbook values.

It was also necessary to determine when inertial effects become of importance The data from the 85% glycerol solution and the $^1/_2$% Natrosol

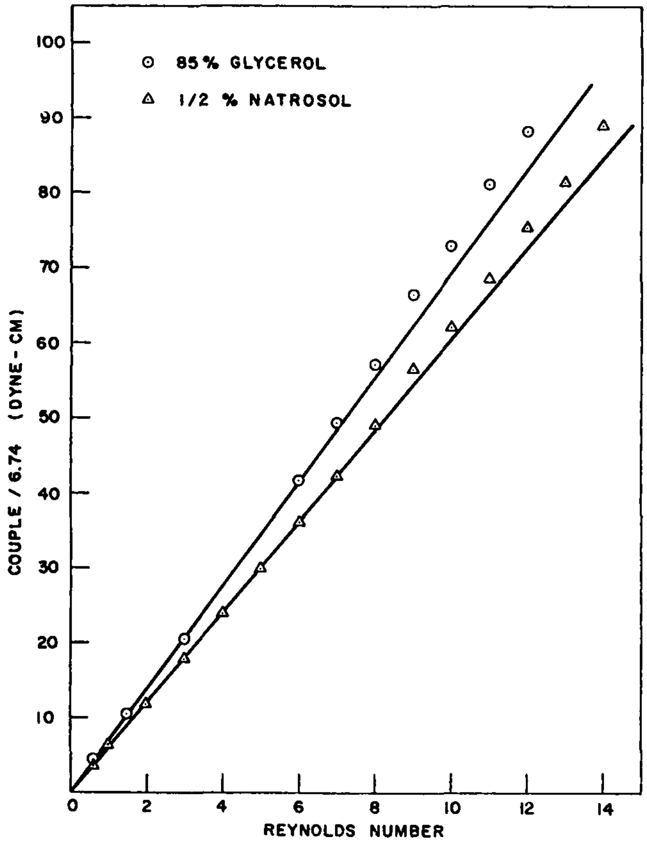

Fig. 1. Inertial effects in rotating sphere device.

solution indicate that inertial effects become significant at a Reynolds number of about 7. Figure 1 shows a plot of measured torque versus Reynolds number for these two solutions. For the 91% glycerol, 96% glycerol, $^3/_4$% Natrosol, and 1% Natrosol, a full-scale deflection on the dynamometer was attained before the inertial region was reached. Figure 2 shows the results of these experiments. The relationship is seen to be linear over the entire range studied. Therefore, the limiting viscosity can be accurately obtained. The $^1/_4$% Natrosol solution's limiting viscosity was too low for a meaningful torque reading to be obtained without rotational speeds that produced Reynolds numbers greater than 7. The minimum limiting viscosity that can be measured practically with this instrument is about 25 cp.

Since this technique is designed to measure the viscosity at zero shear rate, it is desirable to know the approximate shear rates at which the measurements are being made. For a slowly rotating sphere the shear rate is given by

$$\Gamma(r,\theta) = r \sin \theta \omega'(r) \qquad (6)$$

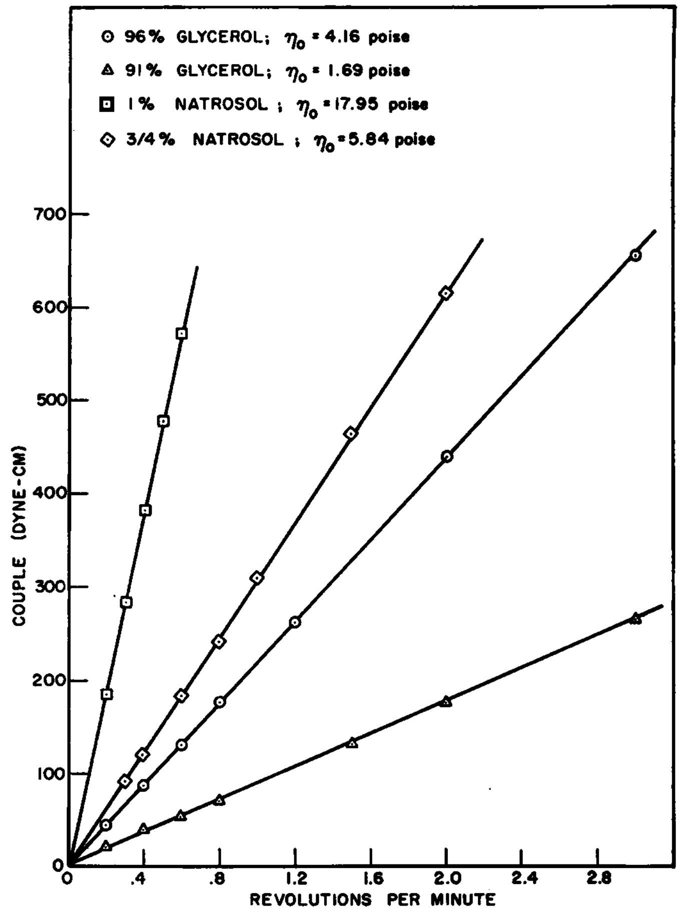

Fig. 2. Measured couple vs. rotational speed for fluids studied

The solution of the equations of motion gives

$$\omega(r) = (R^3/r^3)\Omega \tag{7}$$

Thus

$$\Gamma = 3 \sin \theta\Omega(R/r)^3 \tag{8}$$

Therefore, on the surface of the sphere, the maximum shear rate is

$$\Gamma_{\max} = 3\Omega \tag{9}$$

while the corresponding maximum shear stress is

$$p_{r\phi|\max} = 3C/8\pi R^3 \tag{10}$$

The average shear rate on the surface is

$$\bar{\Gamma} = \int_0^{2\pi}\int_0^{2\pi} \Gamma(R,\theta)R \sin \theta d\phi R d\theta = 6\Omega/\pi \tag{11}$$

and the corresponding average shear stress is

$$\bar{p}_{r\phi} = 3C/4\pi^2R^2 \tag{12}$$

In the experiments performed, the angular velocities were generally under 5 rpm. Thus, the average shear rate was less than 1 sec.$^{-1}$. This measurement then gives values at considerably lower shear rates than the falling sphere device. For example, for the 1% Natrosol solution (η_0 = 17.95 poise) couples were measured for five angular speeds, the maximum of which was 0.6 rpm. Thus, at the highest rotational speed the maximum shear rate was only about 0.2 sec.$^{-1}$. If the falling sphere viscometer of McEachern[9] was used for this fluid, the minimum shear rate obtainable would be about 2 sec.$^{-1}$.

The usefulness of a measured limiting viscosity in empirical constitutive equations was also studied for the Natrosol solutions. The study was limited to one common lower-limiting-viscosity model, the Ellis model [eq. (3)]. Data over a shear rate range of about 10–1000 sec.$^{-1}$ was taken on the Couette viscometer at the same temperature and time that the corresponding limiting viscosity measurements were made. The viscometric data were converted to shear stress–shear rate data by using the method of Krieger and Elrod.[10] These data along with the independently measured value of ϕ_0 were fitted by a least-squares technique to the Ellis model, thereby finding the best-fit values for ϕ_1 and α. The average absolute deviation of the experimental shear rates from those calculated from the Ellis model fits was 2.3% for the Natrosol solutions. This deviation is within the accuracy of the viscometric measurements.

Conclusions

The rotating sphere viscometer is a superior method of measuring the viscosity at zero shear rate. For practical reasons the instrument is limited to substances whose limiting viscosities are greater than 25 cp. The use of the measured limiting viscosity in conjunction with the usual viscometric data yields empirical models that can be used with confidence over a wide range of shear rates.

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Résumé

Le viscosimètre à sphère tournante est une méthode supérieure de mesure de viscosité à tension de cisaillement nulle pour des fluides modérément visqueu non-Newtoniens. Les effets d'inertie et des parois de l'appareil sont étudiées expérimentalement. Les rapports tension-cisaillement est linéaire pour les vitesses de cisaillement étudiées. Ceci permet d'obtenir la viscosité limite. Cette valeur mesurée des viscosités limites peut être utilisée dans des modèles empiriques qui sont applicables sur une vaste gamme.

Zusammenfassung

Das Rotationskugelviskosimeter erweist sich als ein ausgezeichnetes Hilfsmittel zur Messung der Viskosität mässig viskoser, nicht-Newton'scher Flüssigkeiten bei der Schubgeschwindigkeit Null. Trägheits- und Gefässeinflüsse werden experimentell untersucht. Bei den angewendeten, niedrigen Schubgeschwindigkeiten besteht eine lineare Beziehung zwischen Schubspannung und Schubgeschwindigkeit, was eine Bestimmung der Grenzviskosität erlaubt. Dieser gemessene Grenzviskositätswert kann in empirischen, über einen weiten Bereich anwendbaren Modellen herangezogen werden.

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