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SEDIMENTATION OF PARTICLES THROUGH QUIESCENT SUSPENSIONS

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ABSTRACT

Studies of falling-ball rheometry in concentrated suspensions, embodying a combination of analysis, experiment, and numerical simulation, are discussed. Experiments involve tracking small balls falling slowly through otherwise quiescent suspensions of neutrally buoyant particles. A theory has been developed relating the average ball velocity to the macroscopic suspension viscosity, and, for dilute suspensions, agreement is obtained with Einstein's sheared suspension viscosity. Detailed trajectories of the balls, obtained either with new experimental techniques or by numerical simulation, are statistically interpreted in terms of the mean settling velocity and the dispersion about that mean. We show that falling-ball rheometry, using small balls relative to the suspended particles, can be a means of measuring the macroscopic zero-shear viscosity without disturbing the original microstructure significantly; therefore, falling-ball rheometry can be a powerful tool to study the effects of microstructure on the macroscopic properties of suspensions.

INTRODUCTION

The microstructure of a concentrated suspension influences the macroscopic flow properties of that suspension. In turn, the flow of the suspension influences the microstructure in a tightly coupled process. Evidence of this can be seen in the diffusion-like movement of marked particles in suspensions undergoing inhomogeneous shear. Several studies have shown that particles migrate to regions of low deformation rate, that large particles migrate faster than small particles, and that ordered structure can be formed.[1-3] If such phenomena are caused by shearing a suspension, then one must confront the need to probe the properties of a suspension without *changing* the suspension properties through the very act of *measuring* them. Conventional viscometers employ flow fields that tend to influence the microstructure of the suspension. For example, suspended fibers, if not subject to strong, randomizing, rotary Brownian forces, will tend to align in shearing flow; hence, the measurements will be performed on suspensions having flow-induced anisotropy.

New techniques to track balls in concentrated suspensions (which are normally opaque) allow the use of falling-ball rheometry to determine the macroscopic viscosity of a suspension with little effect on the microstructure of the suspension. If the size of the probe (the falling ball) is of the order of the

characteristic length of the suspended particles, the ball disturbs the original microstructure of the quiescent suspension only slightly as it falls. We show that falling-ball viscosity measurements agree well with independent measurements in conventional shear rheometers for moderately concentrated (solids volume fraction, ϕ, less than about 0.30) suspensions of spheres. These measurements can also be used to probe the viscosity of suspensions of rods without unduly modifying the orientation of the suspended particles. In addition, with detailed particle tracking, we may also investigate the microstructure of a suspension by studying the fluctuations in the falling ball's velocity.

Numerical simulation of a sphere settling in quiescent suspensions can lend additional insight into the effects of suspension microstructure on the apparent viscosity of the suspension. In some instances it is far easier to simulate an ideal suspension of known particle configuration than it is to create such a suspension in the laboratory. We show that the use of boundary element methods can lead to successful modeling of falling-ball rheometry in suspensions.

In the following sections we first discuss a theoretical basis for measuring the macroscopic viscosity of a suspension using a falling ball that is about the same size as the suspended particles. We next describe techniques used to enable us to perform falling-ball experiments in the laboratory. Results from studies of model suspensions of spheres and rods are reviewed. Third, we discuss the possibility of using the fluctuations in the ball's terminal velocity, as the ball interacts with individual suspended particles or clusters of suspended particles, to give information about the suspension microstructure. Finally, we outline current numerical modeling efforts using boundary element methods.

THEORETICAL BASIS FOR FALLING-BALL RHEOMETRY IN SUSPENSIONS OF SPHERES

Einstein derived his well-known formula for the apparent viscosity of a dilute suspension of spheres by subjecting the suspension to an irrotational flow field.[4,5] We, on the other hand, have considered a suspension that is macroscopically at rest except for being perturbed by a steady point force or Stokeslet.[6] This singularity is asymptotically equivalent in its far field behavior to a sphere animated by an externally imposed body force (for example, a ball settling under the influence of gravity). We have derived an expression for the effective viscosity of a quiescent suspension of neutrally buoyant spheres by two independent, but closely related methods: (1) by calculating the suspension-scale Stokeslet velocity and pressure fields arising from a steady point force acting on a fixed interstitial fluid point of the suspension and comparing these fields with those arising in a hypothetical homogeneous Newtonian fluid continuum of viscosity μ; (2) by calculating the retardation in Stokes settling velocity of a ball falling through the suspension under the action of a net gravity force, as compared with the mean velocity achieved by the ball when settling through the particle-free homogeneous fluid of viscosity μ_o. Wall effects were included in the latter calculation.

Einstein's classic result $\mu = \mu_o(1 + \frac{5}{2}\phi)$ for the suspension viscosity was reaffirmed in both cases. Moreover, the falling-ball wall correction to Stokes law for a dilute suspension bounded within a hollow sphere was found to be identical to that for a homogeneous Newtonian liquid. This result strongly suggested that well-known, homogeneous Newtonian fluid, wall corrections for other bounding geometries could equally well be applied to suspensions. In particular, one may apparently determine the viscosity of a suspension from falling-ball rheometry data (taken in circular cylinders) by assuming that the classical, Newtonian, cylindrical wall corrections to Stokes law[7,8] also apply to suspensions, as we have confirmed experimentally.[9]

The suspension viscosity result was shown to be independent of the relative radii of the settling to suspended spheres (even when the sedimenting sphere is smaller than the suspended spheres), as well as of the degree of polydispersity of the suspended spheres. These analyses demonstrated that Stokes law *without slip* applies on the suspension scale to the mean velocity of a ball settling through a (dilute) suspension. One might expect, especially in the case where the settling sphere is small compared with the sizes of the suspended spheres or the mean distance between them, that

'Knudsen-type' effects would be obtained, leading to an effective slipping motion at the surface of the settling sphere when viewing its motion on the suspension scale. As we will show in the next section, no such slip is observed in falling-ball experiments in suspensions of moderate concentrations ($\phi < 0.30$). At very high concentrations (e.g., $\phi = 0.50$) anomalous behavior does occur;[10] however, these concentrations are obviously far beyond those for which our dilute suspension theory is rigorous.

FALLING-BALL RHEOMETRY IN SUSPENSIONS

Real-time Radiography and Index of Refraction Matching

The movement of particles in concentrated suspensions is difficult to measure because observation of the interior of a flow field is obstructed by the high particle density. Most suspensions are opaque, even at relatively low particle concentrations. Radiography using penetrating radiation such as x rays is one method to image opaque suspensions. If the imaging is on a fluor, the image may be electronically amplified, observed by a video camera, and recorded on videotape. This technique allows the movement of high Z-number tracer particles within the suspension to be followed.

The use of two complete x-ray systems focused on the same point provides a stereo view of the tracer particles for three-dimensional tracking. The video systems we use can support two cameras simultaneously: this ensures the synchronization of the two images. With this stereo view, we obtain four screen coordinates to determine three spatial coordinates. This is an overdetermined systems problem that lends itself to linear regression analysis. We have used the process described by Walton[11] to determine the laboratory-fixed coordinates from the camera coordinates.

The accuracy in laboratory space depends on the field of view, determined by the needs of the particular experiment. In the experiments discussed in the following subsections, an area of about 150 × 75 mm is imaged on each split screen. This implies that the accuracy in the measured position of the particle is within 0.2 mm. (However, note that with an x-ray microfocus, a typical field of view is only 10 mm × 10 mm, and very small particles can be tracked accurately. We have successfully tracked steel balls with diameters of 432 μm.[12])

Although real-time radiography has the distinct advantage that it can be used with any opaque suspension, the tracking of tracer particles can be accomplished optically in a transparent suspension. We have developed a three-component, Newtonian liquid that matches both the refractive index and the density of polymethyl methacrylate (PMMA). Therefore, transparent suspensions of neutrally buoyant PMMA particles can be made. This suspending liquid is a solution of practical grade 1,1,2,2 tetrabromoethane (TBE) from Eastman Kodak (14.07% by weight); UCON oil (H-90,000), a polyalkylene glycol made by Union Carbide (35.66% by weight); and Triton X-100, an alkylaryl polyether alcohol from J. T. Baker (50.27% by weight). Similar solutions with various viscosities can be made by using different UCON oils, which can be purchased in a wide range of viscosities.

With transparent suspensions, the use of high-energy x rays is no longer required; thus productivity is increased by decreasing the complexity of the measurement process. However, mapping the camera coordinates to laboratory coordinates now requires correction for the nonlinear term arising from Snell's law at the non-index-matched surfaces. We have developed a new algorithm, with this correction, to provide tracking which is comparable in accuracy to that using radiography.

Falling-Ball Experiments in Moderately Concentrated Suspensions of Spheres

We have used both of the methods discussed above to track the path of a dense sphere settling slowly through a quiescent suspension of spheres.[9,13] We have measured the viscosities of suspensions of 5% to 55% by volume of uniform PMMA spheres in various density-matched Newtonian liquids, usually a mixture of UCON oil and TBE. Suspended spheres from 3.18 mm to 12.7 mm in diameter have been used. A ball composed of any metal such as brass, nickel, or tungsten carbide is a sufficient x-ray attenuator to produce an x-ray image that can be tracked as the ball falls through the suspension. Balls of opaque plastic, aluminum, and corundum have also been used in transparent suspensions. Typically, we use balls of a size fairly close to that of the suspended spheres. The suspensions are held in temperature-controlled cylindrical columns and are stirred before each experiment to achieve a uniform distribution of suspended particles.

The discrete nature of the suspension is readily apparent in falling-ball experiments. One expects a very large ball to fall smoothly through a suspension of tiny particles and its velocity to appear fairly constant. However, when we observe the passage of a ball of the same diameter as large suspended particles we see that actually the velocity is not constant. Periods of almost no motion, as the falling ball approaches and "rolls off" suspended particles, alternate with periods of almost free fall in the interstices between suspended spheres. However, a statistical analysis reveals that the *average* terminal velocity of the ball, measured over a distance (usually between 100 and 1000 suspended particle diameters), is reproducible.

Furthermore, if this average terminal velocity, corrected for Newtonian wall effects, is translated into a viscosity, this viscosity is independent of the size of the falling ball relative to the diameter of the suspended spheres over a wide range of falling-ball sizes. (Anomalous behavior can occur with very small or very large balls, though.[10,13]) For moderately concentrated suspensions ($\phi < 0.30$), the average relative viscosity (μ_r, the viscosity of the suspension normalized by the suspending fluid viscosity), agrees well with independent measurements taken in shear and capillary rheometers.[14] These rheometers generally indicate that moderately concentrated suspensions of spheres behave as Newtonian fluids, without the anomalous strain-dependent results of the higher concentrations. Falling-ball rheometry, using relatively small balls, then can determine the bulk shear viscosity of a suspension *while only slightly modifying the suspended particle distribution.*

Falling-Ball Experiments in Suspensions of Rods

An illustration of the use of falling-ball rheometry as a tool to measure viscosity without unduly influencing the microstructure of the suspension can be found in recent work with suspensions of rodlike particles.[15–17] For suspensions of non-Brownian rods in Newtonian fluids, viscometric and elongational flows (the usual tools of rheologists) induce an alignment of the rods. With falling-ball rheometry the initial orientational distribution of the rods can be controlled and fiber-aligning effects of the flow field are minimal.

Experiments were performed to determine the macroscopic viscosity of suspensions of randomly oriented rods.[15,16] The particles were large (typically 1.596-mm diameter), neutrally buoyant, and well characterized. The measured viscosities of suspensions of aspect-ratio-19.83 particles varied linearly with ϕ below a volume fraction of about 0.125 and cubicly above that. This critical volume fraction where the transition between dilute (linear) and (semi)concentrated behavior occurs is remarkably similar to that predicted for solutions of rodlike macromolecules, as is the dependence of the specific viscosity on the cube of ϕ after this transition.[18–20]

Unlike with suspensions of spherical particles, here we cannot compare the falling-ball measurements with shear flow measurements because the latter measurements cannot be done on a similar suspension of *randomly distributed* rods. The flow necessarily sets up a different structure of particles in the suspension. However, theoretical predictions by Brenner and by Haber and Brenner exist for dilute suspensions of rods with sustained random distributions.[21,22] The intrinsic viscosity $[(\mu_r - 1)/\phi]$ predicted by these theories for suspensions of rods of this aspect ratio is 29.2, and the experimental result of 27.6 differs from this by only 5.8%. Further experiments with other aspect ratio rods also show very good agreement with theory.[16]

In a subsequent series of experiments, the suspended rods were approximately aligned hydrodynamically before each measurement so that the suspensions were anisotropic.[17] Here, the falling-ball measurements yielded an apparent viscosity (in the direction parallel to the axis of the cylinder) that was substantially less than that for a suspension having the same volume fraction of the same rods in a random configuration. For example, at $\phi = 0.05$, the relative viscosity of an oriented suspension was 1.52, whereas the randomly oriented suspension had a relative viscosity of 2.37. In addition, the viscosities measured for the suspension of aligned rods closely correlated with the viscosities of suspensions of short fibers (having similar aspect ratios and concentrations) measured in shearing flows.[23] This implied that such alignment may mimic the flow-induced orientation found in rotational rheometers. These results showed the possibility of using falling-ball rheometry to determine a viscosity dependent on the measurement direction (a viscosity *tensor*) for anisotropic suspensions.

DISPERSION OF A SETTLING SPHERE AS A PROBE OF MICROSTRUCTURE

We are exploring the possibility of using the fluctuations in the terminal velocity, as the ball interacts with individual suspended particles or clusters of suspended particles, to give information about the suspension microstructure. Whereas the mean settling velocity predicts the continuum behavior of the suspension, the dispersivity around the mean velocity allows insight into the non-continuum behavior of the suspension caused by the presence of the macroscopic suspended spheres.

To date, detailed tracking of several sizes of falling balls relative to the suspended spheres (ratios from 0.5 to 4.0) have been performed for several suspended sphere sizes (3.18 mm to 12.7 mm diameter a) and two concentrations ($\phi = 0.30$ and 0.50). After a characteristic time interval (usually equivalent to the time needed for a ball to settle four to six suspended-sphere diameters), the experimental data can be interpreted via an analogy to the long-time root-mean-square statistical behavior of a colloidal particle undergoing Brownian motion, namely $(\Delta x)^2 \approx 2Dt$ (where Δx is displacement, t is time, and D is the dispersivity). The dispersivity is then rendered dimensionless by using the size of the suspended spheres and the mean settling velocity as respective characteristic length and time scales.

The three-dimensional traces of the falling-ball paths indicate the random structure of the suspensions studied. The long-time behavior is, indeed, Fickian in nature, as witnessed by the data's linear relationship between the square of the displacement and the time. Furthermore, Fourier transforms of the paths reveal no statistically significant frequency peaks. Around $v/2a$ and $v/4a$ (v the mean settling velocity) there appear small peaks, but they are not significantly different from the noise level. Only three experiments out of 30 have been investigated in detail and further examination may reveal these peaks as significant, but in no case are they a dominant feature of the particle motion.

Experiments at $\phi = 0.30$ and $\phi = 0.50$ show qualitatively different dimensionless dispersivity behavior. The data at $\phi = 0.30$ display dispersivity in both the vertical and horizontal directions that is independent of ball size, whereas the data at the higher concentration reveal a larger horizontal dispersivity for the smaller settling balls (diameters less than a). Also, here, the vertical dispersivity increases then decreases when the settling ball is smaller than the suspended spheres. We believe that this transition signals a change in the bulk suspension characteristics, since the smaller settling balls also predict a significantly lower suspension viscosity than do the larger ones.[10] Interestingly, in all cases the measured dispersivity parallel to gravity is 10 to 20 times that perpendicular to the settling path.

To see if the same vertical dispersion characteristics were also present in the many tests where only the average settling velocities were measured, we related the variance in the mean settling velocity to the dispersivity in the vertical direction. The main conclusion was that these dimensionless dispersivities, for settling balls two to six times larger than the suspended spheres, appeared to be constant at a given volume fraction ($0.05 \leq \phi \leq 0.50$), in agreement with the results of the detailed tracking experiments above. On the other hand, when the settling sphere was even larger compared to the suspended spheres, the dimensionless dispersivity decreased by a factor of 2. However, because the settling sphere in this limit was also fairly large compared to the size of the containing cylinder, an experiment with small suspended spheres in a large cylinder ($d_{cyl}/a \approx 400$) will be needed to determine if the effect is due to the containing walls or due to approaching the continuum limit. Note that the length scale on which the dispersivity is observed is the size of the suspended particle, which is why a sphere settling in a suspension of very small particles (such as paint) would appear to settle evenly.

NUMERICAL SIMULATIONS OF FALLING-BALL RHEOMETRY

Only a very limited class of problems in multiphase hydrodynamics possesses closed-form, analytic solutions. Other problems (such as the theory discussed earlier in this paper) may be solved by approximating boundary conditions by considering constraints associated with one boundary at a time and iterating successively. Because these calculations are arduous, solutions have been limited to simple geometries. One means of solving multibodied problems with little or no restriction on geometry or boundary conditions is to take advantage of high-speed digital computers and discretization methods such as boundary element methods. Boundary element methods offer new promise to simulate multiphase flows without the need to discretize the interior of the problem.[24,25] Thus, mesh generation is relatively easy for problems involving moving particles.

We have used a boundary element method to simulate the hydrodynamic interaction among suspended particles in a falling-ball experiment.[25] The method couples the quasi-static Stokes equations for the fluid with the equilibrium equations for the particles. We modeled a dilute ($\phi = 0.02$) suspension of neutrally buoyant spheres subjected to the flow caused by the fall of a heavier, but otherwise identical, sphere. The instantaneous drag on the falling ball, due to 40 suspended spheres and the containing walls, was calculated for many different initial configurations of the suspended spheres. The results showed the expected sensitivity of the heavy sphere's velocity to the positions of the suspended spheres. By calculating the resistance to the fall of the heavy ball, as neutrally buoyant spheres were added one at a time, we demonstrated that only the suspended balls in the neighborhood of one cylinder diameter above and below the heavy ball significantly affect the heavy ball's fall velocity. Significant computational time could be saved by modeling this "near field" only. As in our experiments, an apparent viscosity of the suspension could be calculated from the heavy sphere's average velocity. This viscosity was very close to that predicted by Einstein's relationship.

A similar falling-ball rheometer for suspensions of neutrally buoyant rods ($\phi = 0.01$ and particle aspect ratio $= 5$) has been modeled. Twenty-five suspended rods can be initially oriented randomly or aligned either parallel or perpendicular to gravity. Good agreement with experimental measurements for the randomly oriented rods has been obtained. As expected from the experiments described earlier, suspension of aligned rods are predicted to have an effective viscosity parallel to the rods' axes that is distinctly different from that perpendicular to the axes. However, no data currently exist for rods of this aspect ratio when aligned parallel with gravity, and no data exist for any aspect-ratio rods when aligned perpendicular to gravity.

Studies on the effects of the rod orientations are expected to be much easier in this "numerical rheometer" than in the laboratory. In addition, dynamic simulations may lead to more understanding of the relationship among fluctuations in the falling-ball velocity and the microstructure of the suspensions, as discussed in the previous section.

CONCLUSIONS

Conventional rotational rheometers may change the particle distribution in a suspension and, hence, produce measurements on suspensions having flow-induced anisotropy. The falling-ball measurements discussed in this paper, however, have little effect on the structure of the suspension because the probe (the falling-ball) size is fairly small compared with the size of the suspended particles. Analytical studies have placed such measurements on a firm theoretical basis, and experiments on dilute and moderately concentrated suspensions of spheres further emphasize that the falling-ball rheometer is indeed measuring the macroscopic shear viscosities of these suspensions. Experiments using suspensions of rods illustrate that falling-ball rheometry may be a useful tool to isolate the effects of particle orientation on the bulk viscosity. Furthermore, by carefully studying the detailed path of the ball, both in actual experiments and in numerical simulations, we expect to use falling-ball rheometry to probe the relationship between the suspension microstructure and its macroscopic properties.

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