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Concentration dependence of the linear viscoelastic properties of particle suspensions

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Abstract The response under small amplitude oscillatory deformations of a suspension of non-Brownian spheres dispersed in a viscoelastic fluid is investigated. The correspondence principle of linear viscoelasticity is used to derive a simple constitutive model from a model for a suspension in a Newtonian liquid. The theory predicts that for a specific particulate system the concentration dependence of the viscoelastic properties should collapse to a single master curve when the values are normalized with those of the carrier fluid alone. Measurements with the micro-Fourier rheometer using oscillatory squeeze flow are carried out on two suspensions of 60 and 80 μm sized particles dispersed in polymeric fluid and in silicon oil, and the master curve is verified.

Key words Suspensions · Linear viscoelasticity · Concentration dependence · Constitutive equation

Introduction

There has been interest recently in the development of constitutive models to describe the rheological behavior of suspensions, which consist of rigid particles dispersed in a carrier fluid. The rheology of suspensions can be quite complex since it can be affected by particle Brownian motion, shape anisotropy, buoyancy and sedimentation forces as well as a variety of interaction forces between the particles (Russel et al. 1991). In this work, we focus on one of the simplest systems, consisting of near-spherical particles of identical size in the micron range, where Brownian motion effects can be neglected and the dominant inter-particle interaction is the hydrodynamic force transmitted by the surrounding fluid.

Up to now, most investigations have been carried out on particles suspended in a Newtonian liquid, and for these systems there have been steady advances in our theoretical understanding (Frankel and Acrivos 1967; Brenner 1972; Nunan and Keller 1984; Brady 1993; Phan-Thien 1995), and in computer simulations (Brady and Bossis 1988; Kim and Karrila 1991). Experimental studies have been carried out for many years, and presently quite comprehensive summaries are available (e.g. Kamal and Mutel 1985; Mewis 1996). Although the behavior is very rich, one of the general trends observed is that the presence of the particles will increase the flow resistance of the material, which would be expected on simple physical grounds since the adhesion of the fluid at the particle-liquid interface will increase the viscous dissipation in the liquid.

There has been less attention given to the rheological behavior of particles suspended in a viscoelastic carrier fluid, despite the industrial importance of these materials (e.g. the processing of particle-filled plastics). One reason for this is that the Newtonian case itself contains many unresolved issues, and the viscoelastic case is even further complicated by the dependence of the carrier fluid's response on the deformation history. However, if we focus on the response to small strains, a simple physical model will enable us to understand many aspects of the behavior of suspensions in viscoelastic fluids. An experimental investigation of the small strain

behavior has been reported by Ek et al. (1987), who measured the stress relaxation after the application of a small step shear (0.1%) to an HDPE plastic filled with calcium carbonate particles (size 10 μm, volume fraction 30%). They found that the relaxation curves for the filled materials and the plastic alone superposed very well when normalized by the corresponding initial stress value. They did not investigate the effect of other filler concentrations or matrices. An empirical law for the creep behavior of a filled plastic has been proposed by Nielsen (1969), which involves dividing the creep compliance curve of the unfilled plastic by the independently measured increment in elastic modulus caused by the presence of the filler. Nielsen confirmed this rule by carrying out measurements on polyethylene filled with kaolin. However, to our knowledge there has been no systematic study of the possible similarity in the concentration dependence of the viscoelastic response functions, when carrier fluids with different linear viscoelastic characteristics are used.

In this work, we investigate the response of a concentrated suspension in a linear viscoelastic fluid to small strain deformations. We develop a simple constitutive model, which predicts that for a certain particulate system the concentration dependence of the increment in the rheological properties (which we write as $f(\phi)$) should be similar even when fluids with different viscoelastic properties are used. We have carried out experiments on two different systems that support this hypothesis. We note that for suspensions in a Newtonian fluid, a collapse of the reduced viscosity versus concentration curves was observed in the data compilation of Thomas (1969), and Krieger (1972) has suggested a widely used empirical power-law fit to the curves.

This paper is organized as follows. In the next section, we present a theoretical model for the linear viscoelastic behavior of concentrated suspensions of monodisperse spheres, which is derived via the correspondence principle of linear viscoelasticity from the pair lubrication theory developed by Goddard (1977) and van den Brule and Jongschaap (1991). The following section describes the micro-Fourier rheometer and the materials used in our experimental investigations. The results and a discussion are then presented, and in the final section we present our conclusions.

Theory

Constitutive equation

We will proceed in two steps: (1) we briefly review a constitutive equation for a suspension in a Newtonian liquid (Goddard 1977; van den Brule and Jongschaap 1991); (2) we then use the general correspondence result between Newtonian and viscoelastic fluids (Walters 1974) to obtain the constitutive equation for a suspension in a viscoelastic liquid under small strain oscillations, based on the Newtonian liquid result.

Consider a system of particles of radius a dispersed in a Newtonian liquid of viscosity η_0 with a volume fraction ϕ, under a macroscopically applied flow field described by the rate of deformation tensor $\mathbf{D}$. In the present work, we focus on small strain response. For high ϕ the interparticle gap (ε) between a generic pair of neighboring particles will be small (Fig. 1). We write the center-center vector as $\mathbf{R} = R\mathbf{p} \simeq 2a\mathbf{p}$, where $\mathbf{p}$ is a unit vector in the same direction. The dominant mechanism for momentum transfer will be the hydrodynamic squeezing force $\mathbf{F}_{sq} = \frac{3}{2}\pi\eta_0 a\varepsilon^{-1}\dot{R}\mathbf{p}$ which is obtained from the Stoke's equations (ignoring inertia) with the assumption of a small gap and no slip boundary conditions on the particle-liquid interface (see, for example, Kim and Karrila 1991). Thus for a collection of N particles occupying a volume V, the particle contributed stress σ^p will given by:

$$\sigma^p = N/V\langle\mathbf{R}\mathbf{F}_{sq}\rangle \tag{1}$$

where $\langle\ldots\rangle$ is an average over the volume V containing a large number of particles. To estimate $\dot{R}$, the rate of approach of the two generic particles, we assume that the particle centers move affinely with the imposed deformation, giving $\dot{R} = R(\mathbf{D} : \mathbf{pp}) \simeq 2a(\mathbf{D} : \mathbf{pp})$. We obtain from Eq. (1):

$$\sigma^p = f_1\eta_0\langle\mathbf{D} : \mathbf{pppp}\rangle \tag{2}$$

where f_1 is a dimensionless function indicating the magnitude of the squeezing force contribution. f_1 is a function of some characteristic gap size ($\langle\varepsilon\rangle/a$), which can be calculated from the volume fraction ϕ for a given packing of the spheres [e.g. for an isotropic packing $\langle\varepsilon\rangle/a = k\phi^{-\frac{1}{3}}$ where k is a constant $O(1)$]. Thus from now on we will write this function as $f_1(\phi)$. The fourth order
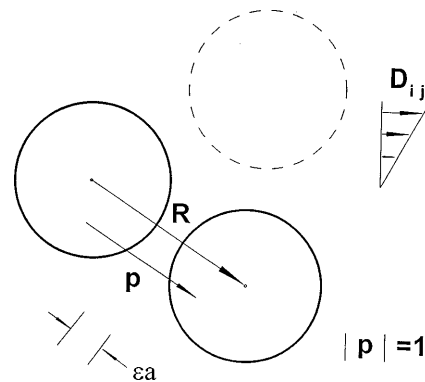

Fig. 1 Schematic diagram of a generic pair of neighboring spheres (radius a) in the flow field with a rate of deformation tensor D_{ij}. $\mathbf{R}$ is the vector joining the two centers and $\mathbf{p} = \mathbf{R}/|\mathbf{R}|$. The gap between the particle surfaces is εa

tensor $\langle \mathbf{pppp} \rangle$ describes the suspension's microstructure, and will in general be a function of time. In this work, we restrict ourselves to the case of small deformations from a random isotropic state. If we assume that there is negligible change in the particle configurations, then we can write:

$$\langle p_i p_j p_k p_l \rangle = \tfrac{1}{15}(\delta_{ij}\delta_{kl} + \delta_{il}\delta_{jk} + \delta_{ik}\delta_{jl}) \tag{3}$$

giving us the particle-contributed stress

$$\boldsymbol{\sigma}^{\mathrm{p}} = \tfrac{2}{15}f_1(\phi)\eta_0 \mathbf{D} \ . \tag{4}$$

There is also a contribution to the stress from the carrier liquid $\boldsymbol{\sigma}^0$, which can be added separately to the particle-contributed stress (Bachelor 1970)

$$\boldsymbol{\sigma}^0 = 2\eta_0 \mathbf{D} \ . \tag{5}$$

Thus the total stress for a concentrated suspension in a Newtonian liquid under a small strain is given by:

$$\boldsymbol{\sigma}^{\mathrm{tot}} = \boldsymbol{\sigma}^0 + \boldsymbol{\sigma}^{\mathrm{p}} = f(\phi)2\eta_0 \mathbf{D} \tag{6}$$

where

$$f(\phi) = 1 + \tfrac{1}{15}f_1(\phi) \tag{7}$$

is the increment in the stress due to the presence of the particles. We note that the suspension still behaves as a Newtonian liquid, but that the viscosity has been increased by the factor $f(\phi)$, where $f(\phi) = 1$ in the absence of the particles. In this present work we leave $f(\phi)$ as a function to be determined from experiment, which may depend on the nature of the particulate system.

If the deformation is a small amplitude oscillatory flow, so that $\mathbf{D}(t) = \hat{\mathbf{D}}\mathrm{Re}(e^{i\omega t})$, the stress will be given by

$$\boldsymbol{\sigma}^{\mathrm{tot}}(t) = f(\phi)2\eta_0 \hat{\mathbf{D}}\mathrm{Re}(e^{i\omega t}) \ . \tag{8}$$

We now generalize the above result to describe the small strain oscillatory response of a suspension in an incompressible linear viscoelastic fluid, characterized by the complex viscosity $\eta_0^*(\omega)$. We use the result of Walters (1974), which states that, under small strain oscillatory deformations, the solution to a boundary value problem (i.e. prescribed forces/displacements on the spatial boundaries) for a viscoelastic material can be obtained from the corresponding Newtonian solution (here Eq. 8). All that needs to be done is to replace the Newtonian viscosity η_0 with the viscoelastic complex dynamic viscosity $\eta_0^*(\omega)$ in the final equation, and to recognize that the stresses and displacements may also be complex quantities (i.e. may possess in-phase and 90° phase-shifted components). This correspondence arises from the similarity of the governing equations of the Newtonian fluid and the Fourier-transformed equations of the viscoelastic fluid. To carry out this substitution in Eq. (8), the boundary conditions in the two problems must be identical, so that we need to assume:

1. There is continuity of velocity at the particle-fluid interface, as in the Newtonian case (no-slip boundary condition).
2. The spatial distribution of particle positions is statistically similar to the Newtonian system. We will again assume in the viscoelastic case that small deformations are applied to a random isotropic system which remains in that state.

With these assumptions we obtain directly from Eq. (8):

$$\boldsymbol{\sigma}^{\mathrm{tot}}(t) = f(\phi)2\hat{\mathbf{D}}\mathrm{Re}(\eta_0^* e^{i\omega t}) \tag{9}$$

where the concentration dependence $f(\phi)$ is the same as for the Newtonian case (Eq. 7). The analogy between Eqs. (8) and (9) is clear. Note that η_0^* is related to the storage modulus $G_0'(\omega)$ and the loss modulus $G_0''(\omega)$ of the carrier fluid by $\eta_0^* = (G_0'' - iG_0')/\omega$. We see that the constitutive equation for a suspension in a viscoelastic fluid is of the usual form for a linear viscoelastic material but, as in the Newtonian case, involves the prefactor $f(\phi)$ describing the increment due to the particles. We see that the increment of the viscoelastic properties described by the function $f(\phi)$ does not depend on the nature of the carrier material, provided that we are in the linear viscoelastic regime.

Governing equations for small amplitude oscillatory squeeze flow

Using the constitutive relation Eq. (9) we now write the governing equations for the micro-Fourier rheometer.

In the squeeze flow field (Fig. 2), we assume that a viscoelastic material is sandwiched between two parallel plates and that the upper plate is oscillated vertically about a mean position with a frequency of ω and displacement amplitude of αh_0:

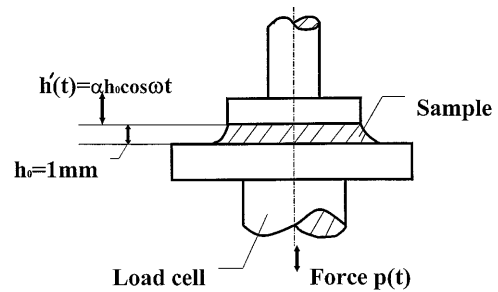$$h'(t) = \alpha h_0 \mathrm{Re}(e^{i\omega t}) \quad \text{where} \quad \alpha \ll 1 \ . \tag{10}$$

Fig. 2 Sketch of the test cell of the micro-Fourier rheometer. The sample is sandwiched between the two plates (mean gap h_0), and the upper plate is perturbed by a small amount $h'(t)$. The force $p(t)$ is detected by the load cell, enabling the viscoelastic properties of the sample to be determined

We measure the signal of the force $F(t)$ transmitted through the fluid, spilt into its in-phase and 90° phase-shifted components:

$$F(t) = \text{Re}\{[F'(\omega) + iF''(\omega)]e^{i\omega t}\} \tag{11}$$

Knowledge of the force and displacement signals enable the storage and loss moduli of the sample $G'(\omega), G''(\omega)$ to be found via a constant factor related to the rheometer geometry. The equations are (Field et al. 1996):

$$F'(\omega) = \alpha \frac{3\pi a^4}{2h_0^2} G'(\omega) \tag{12}$$

$$F''(\omega) = \alpha \frac{3\pi a^4}{2h_0^2} G''(\omega) \ . \tag{13}$$

If the sample is a suspension, then we immediately obtain from the constitutive relation Eq. (9) the following equations:

$$F'(\omega) = \alpha \frac{3\pi a^4}{2h_0^2} f(\phi) G_0'(\omega) \tag{14}$$

$$F''(\omega) = \alpha \frac{3\pi a^4}{2h_0^2} f(\phi) G_0''(\omega) \ . \tag{15}$$

Thus information on the concentration dependence factor $f(\phi)$ can be obtained from measurements of the response under small amplitude oscillatory squeezing. The case for a Newtonian carrier fluid is handled by letting $G_0''(\omega) = \omega\eta_0'(\omega) = \omega\eta_0$ and $G_0'(\omega) \equiv 0$.

Experimental

Random squeeze flow using the micro-Fourier rheometer

The small strain amplitude and quick measurement capability of the micro-Fourier rheometer make it most suitable for the experimental investigation of the linear viscoelasticity of suspensions.

This instrument has recently been developed in a collaborative project between the CSIRO and the Department of Mechanical and Mechatronic Engineering of the University of Sydney. Details have already been presented elsewhere (Field et al. 1996) and so only a brief introduction to the operating principle will be given here. As shown in Fig. 2, the sample is placed between the two parallel circular plates and the upper plate is perturbed vertically about the mean position, the motion being driven by a Terfenol giant magnetostrictive shaker connected to a signal generator with feedback. The instantaneous position of the upper plate is detected by a fibre optic device, giving the displacement $h'(t) = h_0 - h(t)$. The piezoelectric load cell beneath the stationary lower plate measures the instantaneous normal force $F(t)$. The device is constructed to have a very large stiffness, so that the variations of $F(t)$ during oscillations can be considered as arising from the sample.

The radii of the upper and lower plates are 12.5 and 15 mm respectively, and both plates are made of nickel-plated brass. Parallelism of the plates is routinely checked with feeler gauges. The mean gap between the plates (h_0) was usually set at 1.5 mm. As indicated in Fig. 2, the sample is allowed to have a small meniscus

at the outer edge, but this edge effect is not considered to be large (Field et al. 1996). The upper plate is forced to undergo small amplitude oscillations ($\alpha \ll 1$) with an angular frequency ω as in Eq. (10). The limit of linear viscoelasticity was determined by examining the dependence of the viscoelastic moduli G' and G'' on the strain α. It was found that the higher concentration samples had a very low critical strain amplitude, with a value of $\alpha = 0.002$ for the $\phi = 0.4$ suspensions. Therefore we typically set α to 0.001, since our aim was to investigate the behavior under small strains.

To measure the storage and loss moduli, a band-limited pseudorandom noise is used as the input displacement signal, covering a frequency range of 0.5–100 Hz. The linear nature of the material response enables the telescoping of tests at different frequencies into a single excitation sequence (Field et al. 1996). Fourier decompositions of the force signal $F(t)$ and displacement signal $h'(t)$ give a complex number transfer function $TF = \bar{F}(\omega)/\bar{h}'(\omega) = (F' + iF'')/\alpha h_0$, which is directly proportional to the complex modulus $G^* = G' + iG''$ as shown in Eqs. (12) and (13). This enables very quick determination of G' and G'' for the suspensions, with a typical measurement averaging over eight excitation sequences completed in less than 1 min with a high degree of reproducibility.

The measured transfer function will be affected by imperfections in the instrument response. To correct for this, the micro-Fourier rheometer uses a procedure known as frequency domain equalization, which consists of forming the ratio of the transfer functions for the sample fluid and for a known reference material, enabling the transfer function representing the instrument response to be automatically cancelled (Field et al. 1996). The transfer function of the reference material must be independently determined, and in the present study a small compression spring of known spring constant and mass is used.

Materials

Two particulate systems are used: particulate A consisting of polyethylene particles of average diameter 80 μm, and particulate B with 60-μm diameter polyethylene particles. The shapes of both particles are close to spherical, but they are not perfectly round. For both particulate A and B, two types of carrier fluid are used to make suspensions: 1.0-Pas silicon oil and 3% weight Separan polymer solution. The particles are easily dispersed in the carrier fluids with an electric mixing apparatus and there is no apparent particle clumping or evidence of air bubbles. The distribution of the particles appears stable during the short duration of the measurements, with no evidence of sedimentation or buoyancy effects.

Results and discussion

For particulate A, the concentration dependence for the dynamic viscosity $\eta'(= G''/\omega)$ at 10 Hz with silicon oil is shown in Fig. 3 (the value is almost constant up to 100 Hz). As expected, there is a dramatic increase in η' as ϕ is increased. For the polymeric carrier fluid, the variation with ϕ of the storage and loss moduli at different frequencies is shown in Figs. 4 and 5. It is clear that for all frequencies, the values of G' and G'' increase as ϕ is raised. The above theory (Eqs. 14 and 15) predicts that a single master curve corresponding to the function $f_A(\phi)$ should be obtained if we plot the ϕ-dependence of the following quantities, which are normalized with respect to the carrier fluid alone: η'/η_0' for the silicon oil system, and $G'/G_0', G''/G_0''$ for the Separan system at
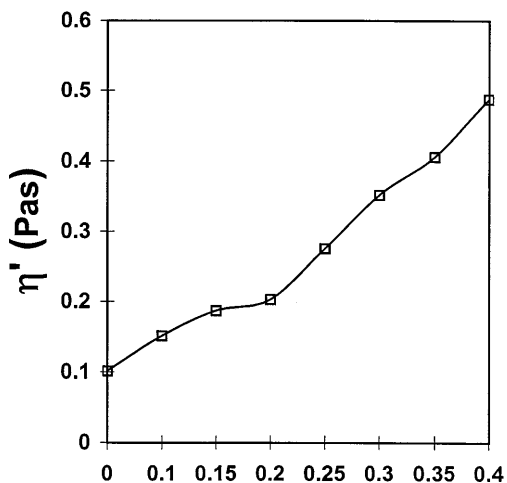
Fig. 3 The dependence of η' at 10 Hz on volume fraction ϕ, for the suspension of particulate A dispersed in silicon oil
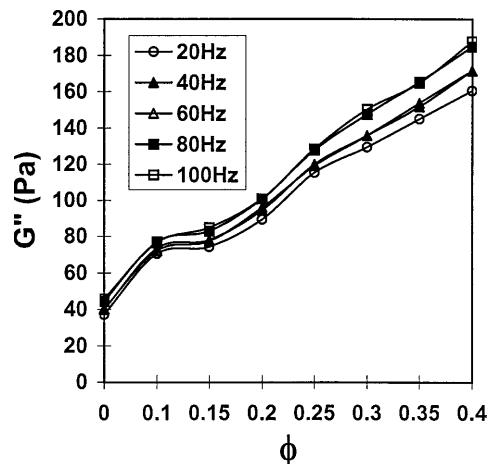

Fig. 5 G'' at various frequencies against volume fraction ϕ, for the suspension of particulate A dispersed in Separan
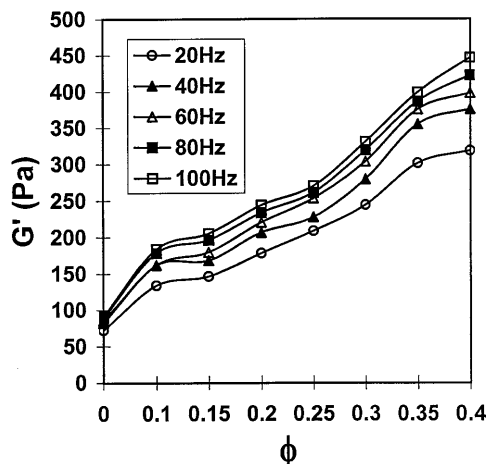

Fig. 4 G' at various frequencies against volume fraction ϕ, for the suspension of particulate A dispersed in Separan

Fig. 6 Variation with volume fraction ϕ of normalized values η'/η'_0 (suspension in silicon oil), G'/G'_0, G''/G''_0 (suspension in Separan) for particulate A. Reasonable collapse to the master curve $f_A(\phi)$ is observed

various frequencies. These normalized values are plotted in Fig. 6 and we see that the data collapse is quite good, lending credence to the basic theory.

Similarly for particulate B, Fig. 7 shows the concentration dependence of η' at 10 Hz in the silicon oil, and Figs. 8 and 9 describe the ϕ dependence of G' and G'' in the polymeric solution. As before, if we plot the normalized quantities η'/η'_0 (for silicon oil), G'/G'_0 and G''/G''_0 (for Separan at various frequencies) as a function of ϕ, we find reasonable data collapse to the function $f_B(\phi)$ (Fig. 10) as predicted by the theory. Note that this function $f_B(\phi)$ need not be the same as $f_A(\phi)$ since factors such as particle shape and the broadness of the size distribution would be expected to produce different results. Thus we see that there is reasonably good collapse of the data for particle systems A and B, lending
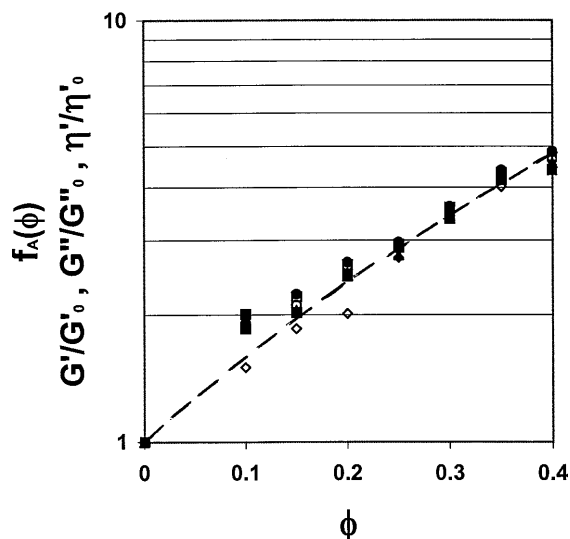
support to the hypothesis that there is a generality in the concentration dependence of the linear viscoelastic properties for a particular suspension.

The present work focusses on the small strain linear viscoelastic response. As noted previously, linear viscoelasticity was found only up to a very small strain (amplitude up to 0.002), agreeing with the observations of Aral and Kalyon (1997) on concentrated suspensions. This departure from linear viscoelastic behavior with increasing strain is thought to be related to the formation of a non-isotropic microstructure, and this issue will be explored separately in a forthcoming paper.
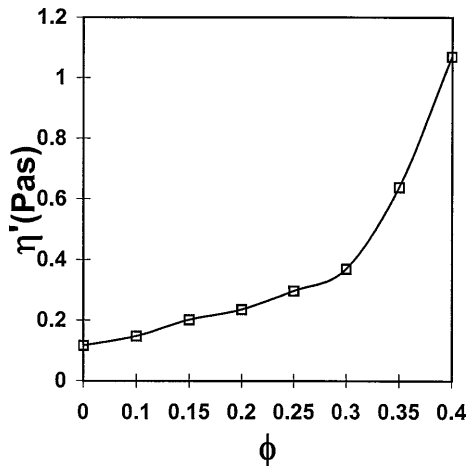
Fig. 7 The dependence of η' at 10 Hz on volume fraction ϕ, for the suspension of particulate B dispersed in silicon oil
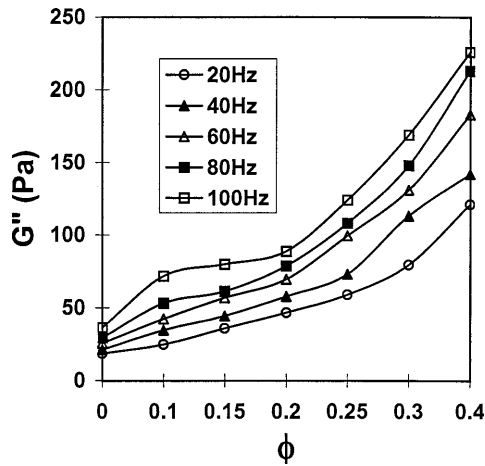

Fig. 9 G'' at various frequencies against volume fraction ϕ, for the suspension of particulate B dispersed in Separan
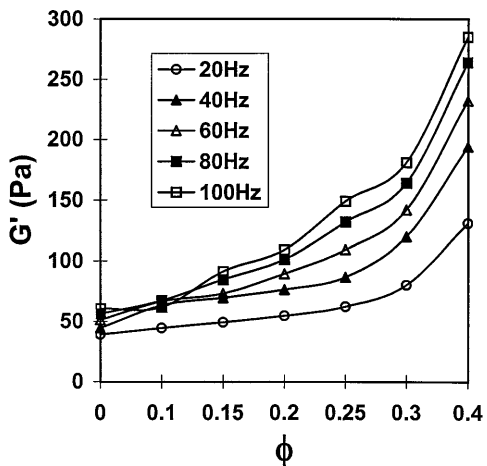

Fig. 8 G' at various frequencies against volume fraction ϕ, for the suspension of particulate B dispersed in Separan
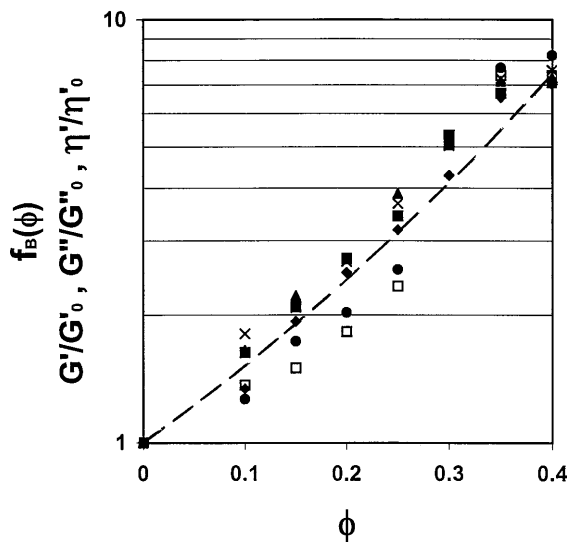

Fig. 10 Variation with volume fraction ϕ of normalized values η'/η'_0 (suspension in silicon oil), G'/G'_0, G''/G''_0 (suspension in Separan) for particulate B. Reasonable collapse to the master curve $f_B(\phi)$ is observed

While the agreement between the experimental results and theory is encouraging, it is appropriate to clarify the limits of the present argument. A key assumption that has been made is the perfect adhesion at the interface between the fluid and particle surfaces. In particular, if the carrier material is more solid-like than the liquids used in the present work, then local detachment at the material-particle interface ("de-wetting") may occur even at small strains, and indeed Nielsen (1969) attributes the deviations from his empirical law mentioned in the Introduction to a lack of adhesion. We also note that since the shear rate is not uniform in squeezing flow, there is the possibility that the particles will migrate to regions of lower shear rate, as has been observed by Gadala-Maria and Acrivos (1980) in concentric cylinder rotational flow and theoretically explained by the model of Phillips et al. (1992). This

migration is predicted to occur even in low amplitude oscillatory flows (see Huilgol and Phan-Thien 1997). In the present work however, the strains are kept very small and the measurements are completed in a short time, so it is thought that migration effects would be minimal.

Conclusions

We have investigated the response of a suspension of particles in a viscoelastic carrier liquid under small amplitude oscillatory squeeze flow. We derive a simple constitutive equation for an isotropic suspension of

monodisperse spherical particles, by extending the lubrication-type model for suspensions in a Newtonian liquid to those in a viscoelastic fluid, through the correspondence principle of linear viscoelasticity. This theory predicts that the increment in the viscoelastic properties due to the presence of the particles will be given by a universal function $f(\phi)$, depending only on the particulate system itself. We have carried out experiments with two types of particulate systems using the micro-Fourier rheometer, and find reasonably good collapse of the curves when the viscoelastic functions, normalized by the values for the carrier fluid alone, are plotted against the volume fraction.

In this paper, we have attempted to elucidate some of the basic mechanisms of the rheology of suspensions in viscoelastic fluids; not only will such an understanding assist in the prediction of the flow behavior of composite materials in many industrial processes, it could possibly lead to methods for characterizing in situ the viscoelasticity of a carrier material, eliminating the need to physically separate the particles and the carrier fluid.

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