Effects of Microstructure on the Compressive Yield Stress

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The effects of microstructure on the compressive properties of aggregated alumina suspensions are determined by intentionally introducing heterogeneities into the suspension. Suspensions are prepared at a high volume fraction and diluted with low shear hand mixing to a series of initial concentrations. As the initial concentration is increased, larger heterogeneities are introduced, and the suspension becomes more compressible relative to the compressive yield stress of the uniform suspension. A simple model is proposed in which the heterogeneous suspensions compress by rearrangement of the dense aggregates until a critical concentration (ϕ_c, which coincides with the volume fraction prior to dilution) is reached. Above ϕ_c, the suspensions consolidate identically to the uniform suspension. With a single fitting parameter (the size of the heterogeneities), the model shows semiquantitative agreement with the experimental data.

Introduction

The behavior of flocculated suspensions under compressive loads affects a wide variety of industrial processes. The compressive properties of suspensions control the effectiveness of solid-liquid separation processes, ceramic fabrication, and the ability of structural bodies and soils to support static loads. Understanding the origin of the compressive properties will enable improved prediction and control of the response of aggregated suspensions to applied loads.

Buscall and White (1987) describe a constitutive model for the compressive behavior of aggregated suspensions. They define a compressive yield stress P_y, which is analogous to the shear yield stress in that it is a critical stress below which the stress is stored elastically by the particle network. Above the compressive yield stress, the suspension consolidates irreversibly until the new volume fraction is capable of supporting the applied load elastically. For a given set of conditions, P_y is a monotonically increasing function of volume fraction ϕ.

Miller et al. (1996) and Green and Boger (1997) show that, for a wide variety of materials, the measured compressive yield stress is independent of the compressive history of the sample. Therefore, a wide variety of loading mechanisms can be used to experimentally determine $P_y(\phi)$. Two of the simplest loading mechanisms are pressure filtration and centrifugation. In pressure filtration experiments, the suspension is subjected to a constant applied load. After a period of time dependent on the filter resistance, viscosity of the continuous phase, particle size, and strength of the interparticle bonds, the bed compacts to a steady-state height. Since each point in the bed experiences the same stress, the equilibrium filter cake has a constant volume fraction. At this point, the applied load is the compressive yield stress at that volume fraction (Lange and Miller, 1987; Landman and White, 1994). In centrifugation, compressive yield stress data can be obtained by two methods: (1) measuring the volume fraction profile of the centrifuge bed at a single speed (Bergström et al., 1992; Miller et al., 1996), or (2) measuring the equilibrium height at a series of speeds (Buscall, 1982; Buscall and White, 1987). Studies on numerous materials indicate that $P_y(\phi)$ is a reproducible constitutive parameter and, thus, can be used in process design and control.

In order to consolidate a particulate network, the bonds between individual particles or aggregates must be broken and reformed. The force required to compress the network, therefore, will depend upon both the strength of the interparticle pair potential (that is, the strength of the bonds), and

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upon the microstructure of the network, which will affect the average number of bonds which must be broken during consolidation. Despite the appealing simplicity of this picture, models for predicting the magnitude of P_y remain in a poor state of development (Buscall et al., 1997).

Bergström et al. (1992) studied the compressive behavior of alumina particles suspended in decalin. Buscall et al. (1988) and Meeten (1994) present data on the compressive behavior of polystyrene latex, silica, and two clays, bentonite and attapulgite. Miller et al. (1996) have investigated the compressive properties of aqueous zirconia, alumina, and kaolin suspensions, and Miller et al. (1995) measured the compressive yield stress for calcium aluminate and portland cement pastes. Green et al. (1994) investigated the compressive yield stress of Bauxite Residue (that is, "red mud"). In all of these diverse systems, qualitatively similar volume fraction dependencies are observed. As the volume fraction of solids approaches the gelation volume fraction ϕ_g from above, P_y decreases to 0, and the compressive yield stress diverges as the system approaches some maximum packing fraction (ϕ_{max}, determined by particle geometry). Here, the gelation volume fraction is defined as the concentration at which the suspension undergoes a transition from a liquid-like behavior (well-defined zero shear rate viscosity) to a solid-like behavior (well-defined low shear rate plateau or yield stress). While the term gelation is more precisely used for systems with interparticle interactions on the order of a few kT, it is often applied more broadly to systems where there is a liquid to solid transition, even in the presence of much stronger attractions (Buscall et al., 1988). In the volume fraction region intermediate to these two limits, P_y often displays a power law dependence of the particle volume fraction. Similar effects are also observed for a variety of systems as the strength of the particle network is varied (Channell and Zukoski, 1997; Green and Boger, 1997).

One of the central difficulties in measuring P_y and a feature typically ignored in developing models for P_y is the potentially variable nature of suspension microstructure in aggregated beds. Unlike suspensions of stable, Brownian particles where microstructures are determined by equilibrium statistical mechanics (Russel, 1987), in aggregated suspensions there is no guarantee that microstructures are reproducible from sample to sample. This property of aggregated suspensions results from the extremely long structural relaxation times produced by the extended networks in the suspensions and the strengths of the interparticle bonds. As mixing and loading procedures used in determining compressive (or shear) properties of aggregated beds may introduce heterogeneous microstructures, determining the sensitivity of P_y to density fluctuations becomes an important experimental concern. Stated a different way, if heterogeneities strongly influence P_y, there may be process variables which can be manipulated to greatly enhance solid-liquid separation techniques.

The repeatability of the compressive yield stress and the independence of $P_y(\phi)$ on the compressive history reported by Miller et al. (1996) suggest that the compressive yield stress may in some way be a material property. However, in these experiments great care was taken to ensure that the samples started with the same initial microstructure. In a separate series of studies, Miller et al. (1995) found that the compressive yield stress measurements depend on the initial volume fraction of the suspensions studied for both cement pastes and aqueous alumina suspensions. These studies motivated us to investigate the role of heterogeneities in the particle network on the compressive behavior of aggregated suspensions.

Potanin and Russel (1996) present a model for consolidation of weakly aggregating fractal networks. Their constitutive model incorporates the viscous deformation of the network at small static loads. In the Potanin-Russel model, the suspension will deform in a viscous manner for $P < P_y$ at times longer than a characteristic relaxation time t_c. They estimate t_c to be

$$t_c = \frac{\delta}{k_o^{1/2} U_c} 6\pi \eta_c a^2 \exp\left(\frac{zU_c}{k_b T}\right) \phi^{d_1/(3-d_f)} \tag{1}$$

where δ is the spacing between particles in the network, η_c is the continuous phase viscosity, a is the particle radius, z is the average number of bonds per particle, d_1 is the fractal dimension of the load bearing chains ($1 \leq d_1 \leq 1.6$), and d_f is the overall fractal dimension of the network (typically $1.6 \leq d_f \leq 3.0$). U_c and k_o describe the depth and curvature of the interparticle pair potential. For aggregated suspensions, Potanin and Russel (1996) use a value of 100 for k_o. For attractions arising from Van der Waal's forces, $U_c \approx Aa/12\delta$, where A is the Hamaker coefficient (5–10 k_bT). Choosing $a = 0.65$ μm, $\eta_c = 10^{-3}$ Pa·s, $\phi = 0.30$, $d_1 = 1.6$, $d_f = 2.0$, $\delta = 5$ nm, and $z = 3$, we estimate U_c to be 50–100 k_bT. Thus, for the alumina system studied here, t_c is very large (greater than 10^{12} years). These estimates indicate that if heterogeneities are produced in a network, they are unlikely to relax out in a reasonable time frame. This will complicate comparison of samples used, for example, to compare shear and compressive properties.

In this study, we investigate the microstructural effects on the compressive yield stress for a series of aqueous alumina suspensions. Large aggregates or other heterogeneities were intentionally introduced into the microstructure, and the compressive yield stress of the suspension was measured via centrifugation and pressure filtration. The presence of heterogeneities in the microstructure was found to affect the values measured for the compressive yield stress at relatively low loads. P_y was found to be insensitive to these heterogeneities at higher pressures. In addition, the effects could be reduced or eliminated altogether through different sample preparation techniques which produced more homogeneous microstructures. Finally, a simple model to describe the qualitative trends observed in the data was developed.

Experimental Procedure

Compressive rheology experiments were carried out using suspensions of conventional α-alumina particles (AKP-15, Sumitomo Chemical, New York, NY). The alumina particles have a density of 3.984 g/cm^3, as determined by helium pycnometry, and a median particle diameter of 1.3 μm. Velamakanni and Lange (1991) measured the zeta potential of AKP-15 powders and determined that the zeta potential reaches a plateau at a pH of approximately 4.0 and that the isoelectric point of these particles is at pH 9.0. Suspensions were prepared at three different pHs (4.0, 7.0, and 9.0), and all of the suspensions studied were flocculated by the addition of 1.0 M NH_4Cl. All of the pH adjustments were made using HNO_3 and NH_4OH.

In order to study the effects of the microstructure on the compressive properties, sample preparation had to be designed to specifically introduce microstructural differences into the suspensions. As a control, a homogeneous suspension was prepared for each pH. This was accomplished by mixing the suspension at concentrations well below the gel point ($\phi \sim 0.10$) so that the suspension would exist as individual particles or flocs. The suspension was prepared at a pH of 4.0 in order to help stabilize these primary particles. The suspension was then subjected to very high shear conditions using a Power Gen homogenizer operating at 20,000 RPM for 15 min in order to break up any flocs present. The suspensions were then titrated to the desired pH and rehomogenized at 20,000 RPM for 15 min to provide a uniform microstructure.

In order to introduce heterogeneities in the microstructure, suspensions were prepared at a high solids volume fraction ($\phi \sim 0.45-0.50$). These suspensions were homogenized at a pH of 4.0 (20,000 RPM, 15 min), titrated to the desired pH with NH_4OH, and re-homogenized. The suspensions were then diluted to various initial volume fractions with 1.0 M NH_4Cl via low shear hand mixing. In order to study the effects of the mixing procedures on the compressive yield stress, samples were also prepared by substituting for hand mixing both ultrasonication (using a Fisher Sonic Dismembrator Model 300 at 60% of maximum power for 15 min) and high shear mixing (20,000 RPM, 15 min).

Centrifugation was used in order to determine the compressive yield stress of the alumina suspensions. Centrifugal loads were applied using a Beckman J2-21M/E centrifuge. Samples were loaded into a JS-13.1 swinging bucket rotor, and spun at a constant speed (typically 1,000 RPM) until equilibrium was reached. The beds were then manually dissected to determine the volume fraction profiles (Meeten, 1993). Using a spatula constrained so that only material above a selected height was removed, the final bed was sectioned into slices of thickness 0.5 mm–2.0 mm so that 15–20 total slices were obtained. The volume fraction of each slice was measured via weight loss upon drying, and the stress which each slice experienced was determined. At each point in the bed, the suspension must support the weight of all material of smaller s, where s is the distance from the top of the bed

$$P_y = \int_0^S \Delta \rho g(s') \phi(s') ds' \quad (2)$$

This method allows the measurement of $P_y(\phi)$ for a wide range of pressures with a single experiment.

The shear modulus of the alumina suspensions was measured in a Bohlin VOR rheometer using a C25 couette geometry (Bob Diameter = 25 mm, Cup Diameter = 27.2 mm). In order to eliminate air bubbles and ensure good rheological measurements, the suspensions were subjected to a standard pre-shear (116 s^{-1} for 5 min). The suspensions were then allowed to relax to remove any residual stress. The elastic modulus was measured at low strains (that is, in the linear viscoelastic region) by an oscillatory shear at a frequency of 1 Hz.

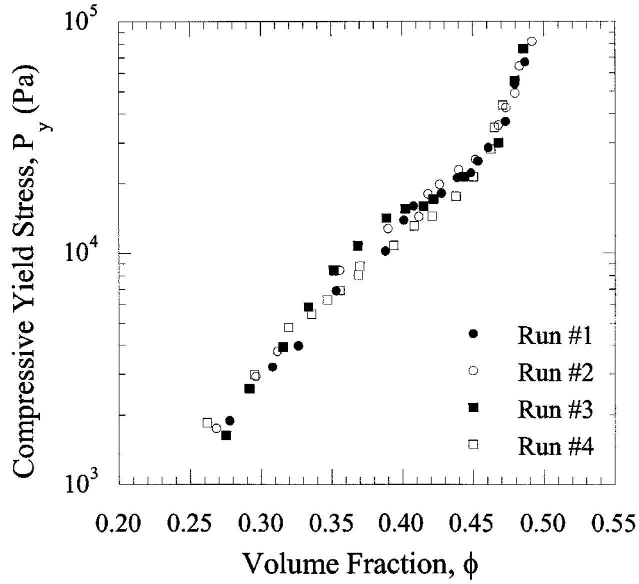

Figure 1. Compressive yield stress for flocculated AKP-15 suspensions at pH 9.0 (the isoelectric point) prepared at $\phi < \phi_g$ to provide a homogeneous microstructure.

The gelation volume fraction is approximately 0.14.

Results

The compressive yield stress represents the critical pressure at which a particle network can no longer support a compressive load elastically and starts to undergo irreversible deformation. Miller et al. (1996) show that the compressive yield stress measurements are reproducible if care is taken to ensure the sample preparation is unchanged. This observation was confirmed here (Figure 1). In addition, when the samples are exposed to high shear prior to compressive tests, the values obtained are independent of the sample preparation method. However, when samples are prepared and diluted to the volume fraction of interest without intense shear, the compressive yield stress varies systematically with the initial volume fraction. In these studies, we use this dilution method to probe the origin of this initial volume fraction dependence, and compare these diluted samples to samples prepared below the gel point so that they have homogeneous microstructures. Due to the insensitivity of the P_y data to initial volume fraction or volume fraction from which the initial volume fraction was created, we use the short hand of calling samples exposed to high shear as having a uniform microstructure.

Figure 2 shows the compressive yield stress curves for the uniform microstructure samples at each of the three pH's studied (4.0, 7.0, 9.0). As with all of the earlier studies, as the interparticle potential becomes more attractive (that is, as pH increases), the suspensions become more difficult to compress. In addition, the S-shaped curve is characteristic of the compressive behavior observed in other systems (Bergström

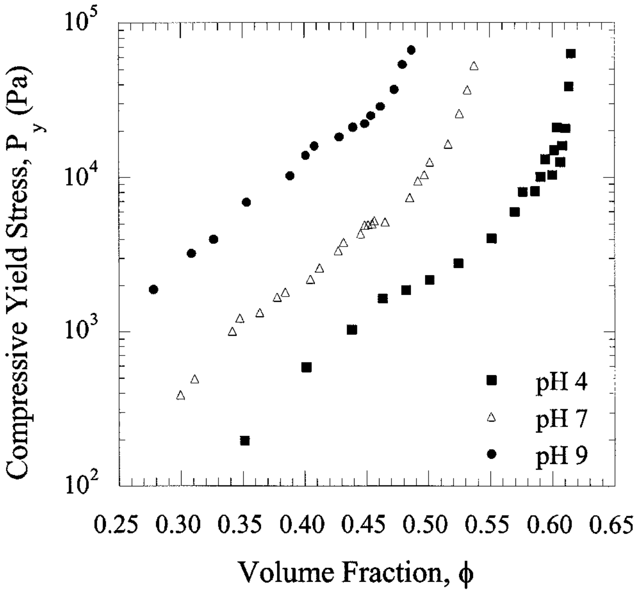

Figure 2. Effects of interparticle pair potential on the compressive yield stress of flocculated alumina suspensions.

The curves presented are for suspensions with homogeneous microstructures (that is, P_y^u).

et al., 1992; Channell and Zukoski, 1997; Miller et al., 1996; Green and Boger, 1997). At low volume fractions, $P_y(\phi)$ approaches zero as $[\phi/\phi_g - 1]^{2.6}$, while at high volume fraction, $P_y(\phi)$ diverges as the suspensions approach close packing. Here, ϕ_g is the gelation volume fraction, which is dependent upon the interparticle pair potential. For the system studied here, ϕ_g was estimated from a curve fit of the low concentration ($\phi < 0.4-0.5$) compressive data for the homogeneous suspensions and qualitative verified via settling experiments. For the pH 4.0, 7.0, and 9.0 AKP-15 suspensions, ϕ_g is approximately 0.24, 0.21, and 0.14, respectively.

When the samples are mixed at high solids loading and diluted to the volume fraction of interest using hand mixing, the compressive yield stress depends upon the initial volume fraction systematically (Figures 3a and 3b). In these experiments, the suspension was initially prepared at $\phi_i = 0.46$. The samples were then diluted to volume fractions of 0.25, 0.31, and 0.36 which we refer to as the initial volume fraction ϕ_o, used in the compressive experiments. The pH 4.0 suspensions were found to be too compressible to allow detailed analysis of the curves, and are not included in this discussion. Even at very low pressures, the pH 4.0 suspensions were compressed to volume fractions approaching maximum packing, and the sensitivity of the measurements was insufficient to distinguish between the different initial volume fractions. However, both the pH 7.0 and the pH 9.0 suspensions increased in compressibility as the volume fraction to which they were diluted increased.

As shown in Figure 4, the shear modulus G' does not show the initial volume fraction differences observed in the compressive experiments. Even for the suspensions diluted to intentionally introduce microstructural differences, the storage modulus is within experimental uncertainties of that obtained for the uniform microstructure suspension. This is likely due to the pre-shear procedure used in loading the suspensions into the rheometer. In order to eliminate air bubbles in the

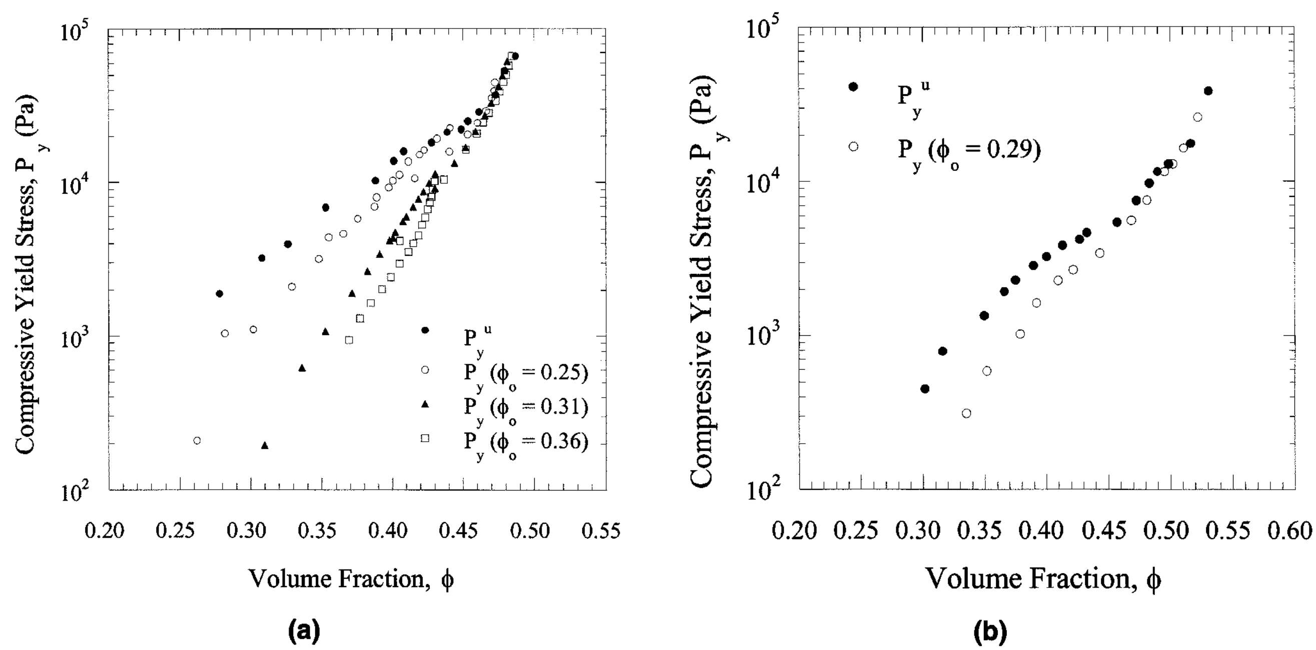

Figure 3. Comparison of the compressive yield stress curves for the alumina suspensions prepared below the gel point (P_y^u) with those for suspensions prepared at high volume fraction and diluted to various initial concentrations using hand mixing.

The data presented are for (a) pH 9.0 and (b) pH 7.0. Note that the curves converge near the volume fraction prior to dilution ($\phi \approx 0.45$).

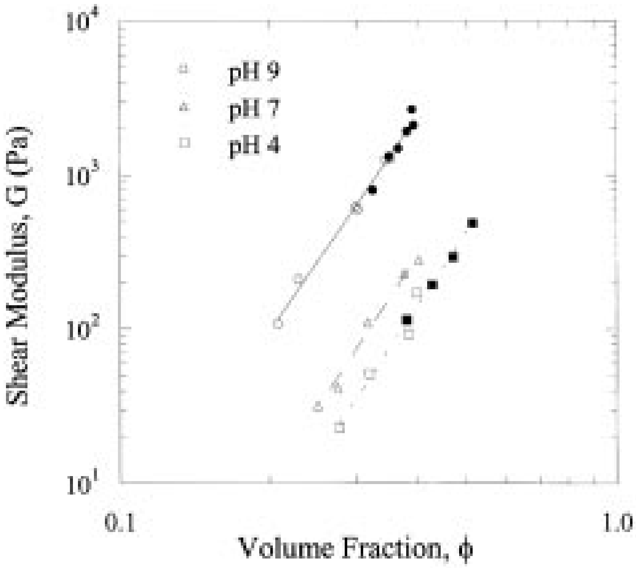

Figure 4. Effects of volume fraction and interparticle potential on the shear modulus for flocculated AKP-15 suspensions.

The solid symbols represent samples prepared with uniform microstructures and compacted to the desired volume fraction, and the open symbols represent samples prepared at high concentration and diluted with hand mixing.

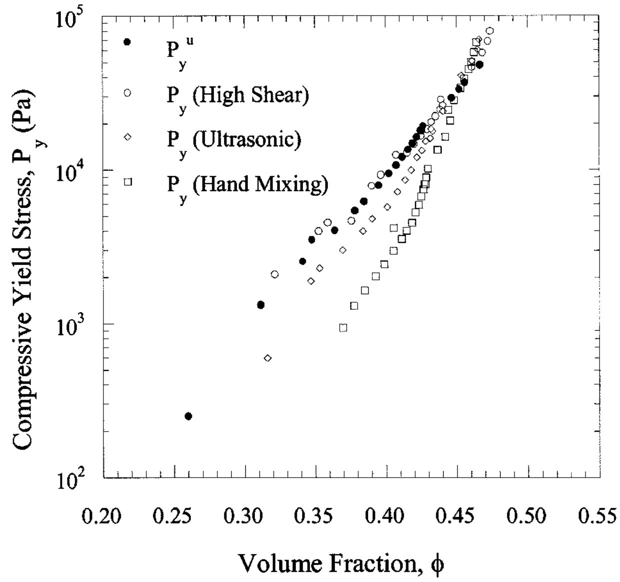

Figure 5. Comparison of the uniform compressive yield stress curve with the curves for suspensions prepared at $\phi = 0.45$ and diluted to $\phi = 0.36$ with various mixing techniques.

Note that high shear effectively returns the suspension to the homogeneous microstructure. All suspensions were prepared at a pH of 9.0 in 1.0 M NH_4Cl.

Couette cell, the suspensions were sheared at 100 s^{-1} for 5 min before the measurements were performed. This shear field seems to be effective at eliminating the initial concentration effects and thus also at eliminating the heterogeneities from which such concentration effects arise.

The effects of different types of mixing upon the suspensions were investigated. Figure 5 shows the uniform compressive yield stress curve (P_y^u) for the pH 9.0 suspensions, as well as the curves for a suspension with an initial volume fraction of 36% prepared by dilution from $\phi_i = 46\%$ with hand mixing, ultrasonication, or high shear mixing. High shear mixing resulted in the same curve as the uniform suspension, while the other two mixing methods showed evidence of microstructural differences. The ultrasonicated sample approached that of the uniform sample, but was somewhat more compressible.

Discussion

Since the high shear mixing resulted in a curve that was, within experimental uncertainty, the same as that we call uniform, we postulate that the initial volume fraction dependence in the compressive yield stress may have been caused by heterogeneous microstructures produced by diluting and poor mixing. Less vigorous mixing procedures would be less effective at breaking up the aggregates and would result in larger inhomogeneities. Miller et al. (1996) found that the suspensions became more compressible as the particle size was increased. Based on this observation, we hypothesize that the presence of large, relatively incompressible agglomerates could qualitatively account for the initial volume fraction effects observed.

As shown in Figure 6, this model is based on the hypothesis that when the suspension is diluted from a volume frac-

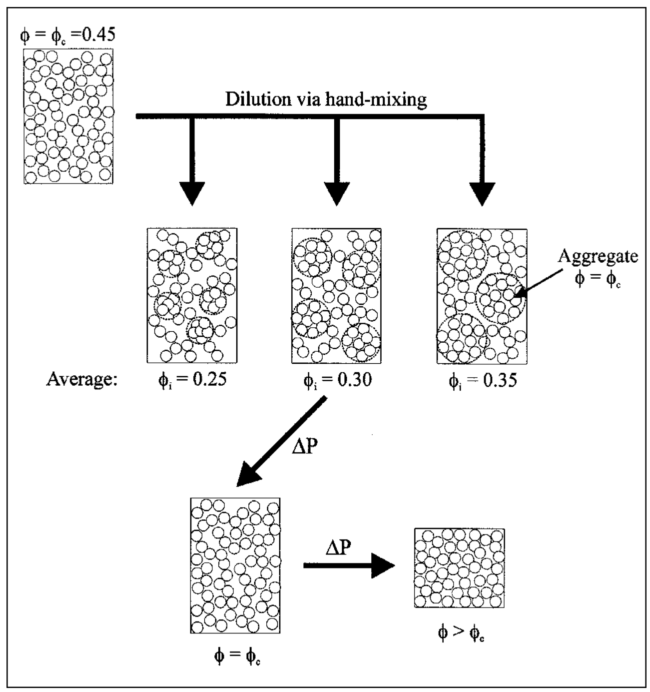

Figure 6. Effects of heterogeneities on the microstructure of flocculated alumina suspensions.

Dilution from high concentration introduces relatively dense ($\phi = \phi_c$) heterogeneities into the microstructure. These aggregates rearrange upon compression until the overall volume fraction of the compression reaches ϕ_c, after which the suspension consolidates as a uniform suspension.

tion ϕ_i, if mixing is poor, the resulting suspension will be composed of agglomerates of volume fraction ϕ_i which have aggregated to form a space filling network. The size of these agglomerates will depend on both the degree of mixing to which the diluted suspension has been exposed and ϕ_o. If the degree of shear is held approximately constant, we expect the size of the agglomerates to decrease as ϕ_o decreases. Our model postulates that the agglomerates are rigid up to the point where the applied pressure exceeds their compressive yield stress. Below this pressure, the suspension consolidates through rearrangement of the agglomerates.

To test this hypothesis, a simple model was developed in which the particle network was considered to consist of uniform agglomerates of size ξ. These aggregates will compress as solid spheres until some critical volume fraction ϕ_c is reached. Above ϕ_c, the aggregates will deform and the suspension will consolidate as a uniform suspension. The compressive yield stress should be related to

$$P_y(\phi) = \left(\frac{\text{energy}}{\text{bond}}\right)\left(\frac{\text{bonds}}{\text{volume}}\right) f(\phi) \approx \epsilon \frac{\phi}{\xi^3} f(\phi) \qquad (3)$$

where $f(\phi)$ reflects the interconnected structure of the network, ϵ is the average energy per bond, and ξ is the characteristic size of the consolidating unit. A consolidating bed, therefore, has two resistances to consolidation: (1) rearrangement of the aggregates, and (2) consolidation within the individual aggregates. Consolidation of the bed is assumed to occur as if these two resistances act in parallel, yielding

$$P_y(\phi) = P_y^u \left[\frac{P_y^u(\phi)}{P_y^u(\phi_{\text{floc}})} \frac{\phi_c}{\phi} \left(\frac{\xi}{\xi_o}\right)^3 + 1 \right]^{-1} \qquad (4)$$

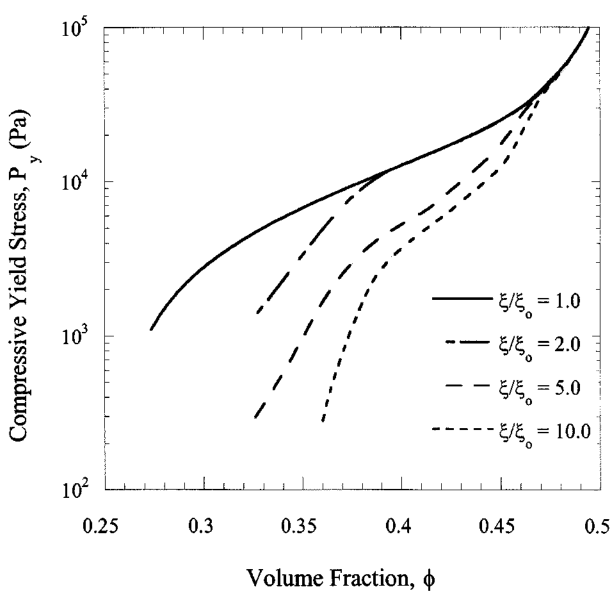

Figure 7. Predictions of the heterogeneity model for alumina suspensions at a pH of 9.0 for various sizes of aggregates within the microstructure.

Note that the curves converge as $\phi \to \phi_c$ ($\phi_c \approx 0.45$).

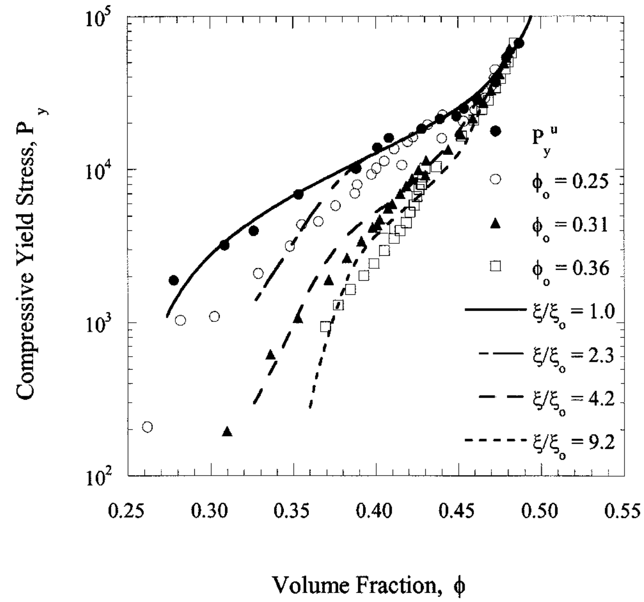

Figure 8. Comparison of the experimental data and the model predictions for the compressive yield stress of AKP-15 suspensions at pH 9.0.

where P_y^u is the compressive yield stress of the uniform suspension. The floc volume fraction (ϕ_{floc}) is calculated by assuming that the flocs reach random close packing when the overall solid volume fraction reaches the volume fraction prior to dilution [$\phi_{\text{floc}} = 0.64\ (\phi/\phi_c)$].

In order to predict compressive behavior from this model, the uniform compressive yield stress is measured. From the curve of P_y^u, the effect of different size ratios of heterogeneities can be determined. ϕ_c is the volume fraction of particles within the individual flocs, and is assumed to be equal to the volume fraction of the stock solution prior to dilution ϕ_i. At ϕ_c, the curves converge, and the compressive yield stress is described by the uniform suspension curve $P_y^u(\phi)$.

Figure 7 shows the predicted compressive yield stress curves for various values of the size ratio (ξ/ξ_o) based on the uniform compressive yield stress curve for the pH 9.0 suspensions. The volume fraction prior to dilution ϕ_c for these experiments was 46%. The model predicts qualitative behavior similar to that observed for the experimental data in Figure 3. Figure 8 displays the experimental data from Figure 3a as well as the model predictions using the size ratio as a single fitting parameter. The model captures the qualitative behavior well. However, due to the low level of microstructural characterization used in the model, quantitative agreement is not achieved. In order to predict the quantitative behavior accurately, issues such as polydispersity in both the primary particles and the aggregates, as well as particle shape must be taken into account. In addition, in a more detailed model, ϕ_c could be predicted as the volume fraction at which the average number of bonds between aggregates is the same as the average number of bonds between particles within a single aggregate.

The use of a high shear field seems to be effective at breaking up the aggregates and, therefore, at eliminating the observed initial volume fraction dependence. It is important to note that while ultrasonication seems to be somewhat effective at removing the aggregates, high shear mixing is very effective. This may be due to the fact that while ultrasonication vibrates the aggregates apart, the lack of bulk mixing allows local density variations to persist and may encourage the re-formation of aggregates.

While this model only captures the effects of heterogeneities in a qualitative manner, several significant conclusions can be drawn. First, in studying the compressive properties of aggregated networks, the method of sample preparation is significant. Here we show that for the same suspension, changes in P_y of an order of magnitude can be achieved by altering the sample preparation method. Secondly, once the heterogeneities are destroyed by high shear or by consolidation to a volume fraction equal to or above the volume fraction of the heterogeneities (ϕ_c in the model), compressive properties are insensitive to the preparation technique. Finally, shear is an extremely effective mechanism of destroying the heterogeneities. While the P_y curves are sensitive to ϕ_o, if the same samples are loaded into a shear rheometer and exposed to a shear rate of 100 s^{-1}, the shear mechanical properties are insensitive to ϕ_o. Thus, while for large samples, high shear mixing appears necessary to produce a uniform microstructure, in small samples exposed to a uniform shear rate, much smaller shear rates are adequate. The ability of small shear stresses to disrupt heterogeneities is a result of the shear yield stress being much smaller than the compressive yield stress (Channell and Zukoski, 1997) and the continuous nature of the shearing action.

Summary

The microstructure of the particle network plays an important role in the compressive behavior of flocculated suspensions. Heterogeneities such as large voids or aggregates in the suspension change the response of the particle bed to a static load. For this reason, in order to quantitatively predict the consolidation behavior of a suspension, the microstructure must be characterized. Care must also be taken during sample preparation in order to prevent the introduction of heterogeneities into the suspension microstructure. Regardless of the sample preparation method, however, measurements of the compressive yield stress show good repeatability when the same preparation recipe is used for all samples. Therefore, once the microstructure of the suspension prepared by a particular recipe is characterized, predictions can be made without further characterization.

In this work, we present a simple model to qualitatively account for the effects of heterogeneities in flocculated alumina suspensions. The model qualitatively predicts the trends observed for a series of samples which were diluted with low shear mixing in order to create and preserve relatively large aggregates in the network. Low shear hand mixing and even ultrasonic mixing are unable to completely remove the aggregates from the suspension, but high shear mixing is effective at removing these heterogeneities. This provides some hope that the compressive yield stresses measured in the laboratory can be used to accurately predict the consolidation behavior of suspensions in industrial processing situations.

The sensitivity of P_y to microstructure is also of technological significance. For example, if process conditions could be manipulated to purposely introduce heterogeneities, solid-liquid separations could be carried out with greater ease.

Acknowledgments

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