

MINERAL-CONTENT AND PARTICLE-SIZE EFFECTS ON THE COLLOIDAL PROPERTIES OF CONCENTRATED LATERITIC SUSPENSIONS

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Abstract—The rheological behavior of concentrated lateritic suspensions from Cuba is affected by mineral composition and particle size. Electrophoretic mobility and yield stress were considered. The lateritic samples were found to be mostly composed of mixtures of serpentine and goethite in varying proportions. The flow properties of the lateritic suspensions are strongly affected by the mineral composition and particle size. This result was determined by comparison of flow properties of the bulk sample and the colloidal fraction. The electrokinetic curves suggest that heterocoagulation is present in all samples, with a zeta potential minimum at the isoelectric point (IEP), which varies with the serpentine to goethite ratio. A relationship between yield stress (τ_0) and the sample volume fraction (ϕ) and particle size (d) was obtained at the IEP from the expression $\tau_0 = k\phi^3/d^{0.5}$, with the constant k dependent on the sample serpentine to goethite ratio.

Key Words—Concentrated Suspensions, Heterocoagulation, Interfacial Mobility, Lateritic Suspensions, Mineral Content of Suspensions, Rheology, Yield Stress.

INTRODUCTION

Structural iron released from silicates in highly weathered soils is precipitated in the form of ferric-oxide minerals. Thus, iron oxides provide excellent aggregate stability (Golden and Dixon, 1985). This stability is attributed to a close association between the iron oxides and clay minerals, although direct evidence of this is limited (Schwertmann and Taylor, 1977). To understand the nature of this association, many studies were performed on iron oxide-silicate interactions. For kaolinite see Ma and Pierre (1997) and Arias *et al.* (1995). However, no similar studies were performed for lateritic soils where iron oxides coexist with serpentine minerals (Cerpa *et al.*, 1996).

Lateritic sediments are of great economic importance for the recovery of Ni and Co (Avotins, 1979; Avramidis and Turian, 1991). One step prior to the extraction of both elements is the preparation of concentrated aqueous suspensions (25–48 wt. % of solids) of the lateritic sediments. The recovery process requires an adequate study of the stability and flow properties of the suspensions. These properties are influenced by many factors, such as surface chemistry, particle density, concentration and size, and shape of the suspended particles (Hunter, 1987; Padmanabhan and Mermut, 1995). For lateritic suspensions, some factors are not easily controlled, and consequently, this behavior is not well understood (Avramidis and Turian, 1991). Cerpa *et al.* (1996) showed that lateritic sediments have a wide range of stability with pH, since their isoelectric point (IEP) ranges from 4 to 9. The IEP range depends on the mineral composition, which in this case is the serpentine/goethite ratio.

In this study the effect of the mineral composition and particle size on rheological properties is emphasized. For this purpose, the flow behavior of the bulk ($\leq 80 \mu\text{m}$) and the colloidal ($\leq 2 \mu\text{m}$) fractions of lateritic samples with different serpentine/goethite ratios are compared. Also, the effects of particle concentration and size on the yield stress at the IEP of each sample is analyzed following the work of Leong *et al.* (1995). Viscosity is commonly used to describe how suspensions flow, but this is a shear rate-dependent property. Thus, we use the yield stress, since it is independent of shear rate and can be used as a parameter for describing the flow properties of the lateritic suspensions (Ramakrishnan *et al.*, 1996). Moreover, the effect of the surface chemistry of the particles was tested by changing the solution pH, which may control the degree of association between particles of different constituents.

MATERIALS AND METHODS

Sample origin and characterization

The lateritic sediments, which developed from ultrabasic rocks rich in olivine, are from the north coast of the Oriente province, Cuba (Vera, 1979). Three lateritic samples from the Yamanigüey and Atlantic deposits were studied. The samples were selected based on different behavior during the sedimentation process. They are referred to as SG, GS, and G according to the goethite/serpentine ratio. Samples SG and G come from Yamanigüey, whereas GS was extracted from the Atlantic deposit.

In addition to the bulk sample ($\leq 80 \mu\text{m}$), the colloidal fraction ($\leq 2 \mu\text{m}$) obtained by sedimentation was also studied for all samples. It was found that the bulk

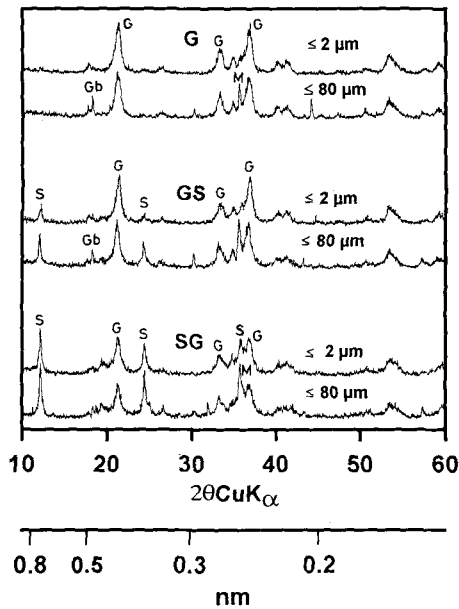


Figure 1. XRD patterns from random-powder mounts for the bulk and colloidal fraction of lateritic samples SG, GS, and G. (Diffraction peaks: G = Goethite; S = Serpentine; Gb = Gibbsite; M = Maghemite)

sample is dominated by the colloidal fraction (65–70 wt. %). However, note that minerals have different densities which affect rheological properties. For example, serpentine and goethite have different particle density and despite equal particle sizes, they may behave differently during their sedimentation process (Padmanabhan and Mermut, 1995). Light scattering (Coulter LS 130) measurements and transmission electron microscopy (TEM, Philips EM300) observations were performed to determine particle size and shape of the samples.

Mineral identification was performed by X-ray diffraction (XRD) using a Philips PW1130 diffractometer (graphite monochromated $\text{CuK}\alpha$ radiation). XRD patterns were obtained for random and oriented clay aggregate powders (air-dried, ethylene glycol solvated, and heated at 300 and 500°C for 3 h). The oriented clay aggregate powders were only used for the analyses of the colloidal fraction. Semi-quantitative estimates of the minerals present were obtained from XRD random-powder patterns, using intensity factors reported by Schultz (1964). Thermogravimetric (TG) and differential thermal (DTA) analyses (Netzsch STA409) were also performed on the colloidal fraction.

Colloidal and rheological properties

The IEP of the solids was determined by measuring electrophoretic mobilities of aqueous dispersions as a function of pH, in a Delsa Coulter 440 apparatus. For electrophoretic mobility determinations, 15 mg of

Table 1. Semi-quantitative mineral composition (relative wt. % between samples) for the lateritic samples. tr: traces, —: not detected.

Sample	Fraction	Goethite	Serpentine	Maghemite	Gibbsite	Quartz
SG	$\leq 80 \mu\text{m}$	32	41	20	tr	7
	$\leq 2 \mu\text{m}$	50	43	4	3	tr
GS	$\leq 80 \mu\text{m}$	60	19	16	3	2
	$\leq 2 \mu\text{m}$	79	15	3	tr	3
G	$\leq 80 \mu\text{m}$	75	—	13	7	5
	$\leq 2 \mu\text{m}$	91	—	—	tr	9

sample of the colloidal fraction was dispersed in 100 cm^3 of 0.01 M NaCl solution to maintain a constant ionic strength, the pH was varied by adding HCl or NaOH as needed.

The flow properties of the suspensions were measured at 25–36 wt. % concentrations and varying pH values, using the Haake Rotovisco RV20 concentric cylinder viscosimeter. The above equipment allows the direct determination of the yield stress (Schramm, 1994).

RESULTS AND DISCUSSION

Mineralogical characterization

Figure 1 shows XRD patterns of select samples for both the bulk and the colloidal fraction. Diffraction peaks at 0.418 (G), 0.269 (G), 0.244 (G), 0.72 (S), 0.36 (S), and 0.460 (S) nm correspond to goethite (G) and serpentine (S) phases, the latter being only observed in samples SG and GS. Corroboration of both goethite and serpentine in the colloidal fraction of all samples was also obtained by infrared (IR) spectroscopy (Cerpa *et al.*, 1996).

The Al content in the lateritic samples was between 7–9 wt. %, which was clearly greater than that estimated by the relative intensity of the XRD peaks of gibbsite (at 4.85 Å). This result suggests that some Al occurs by isomorphous substitution in the serpentine or goethite structures. The presence of Al in the serpentine phase was confirmed by energy dispersive X-ray analysis (EDX) (data not shown). This result, along with the platy morphology observed by TEM, suggests that lizardite is the main serpentine phase present in these laterites (Dixon, 1977). In contrast, the presence of Al in goethite appears limited, as indicated by XRD and IR spectroscopy (absorption band at 890 cm^{-1}) (Cornell and Schwertman, 1996). We note that an enrichment of goethite with respect to serpentine is observed as particle size decreases in samples SG and GS (Table 1; Figure 1). In addition to these phases, small amounts of maghemite (XRD peak at 0.251 nm), gibbsite, and quartz were detected in all samples.

Therefore, the nearly constant mineral compositions observed in the bulk and colloidal fraction and the large amount of the $<2\text{-}\mu\text{m}$ size fraction present

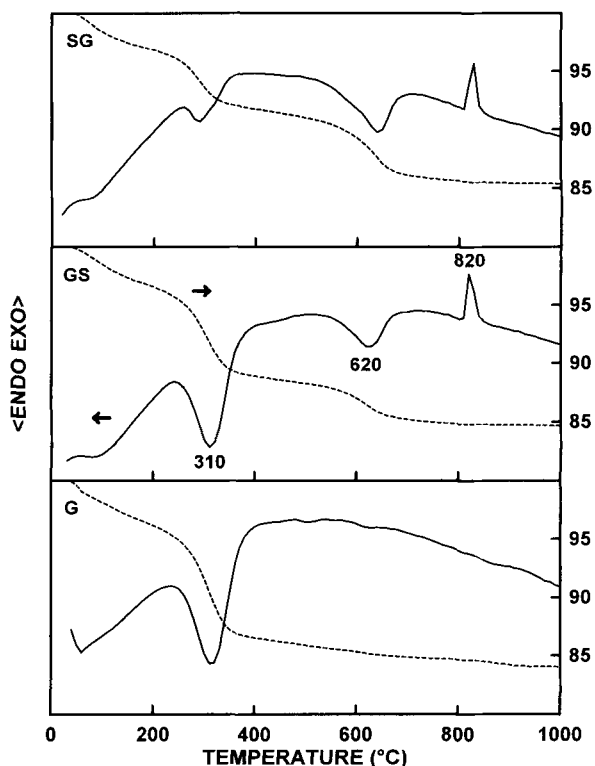


Figure 2. DTA/TG curves for the colloidal fraction of samples SG, GS, and G.

(~70%) suggested that the rheological behavior of the lateritic samples is mostly dependent on the serpentine to goethite ratio of the colloidal fraction. In polydisperse systems, the rheological behavior is usually dominated by the fine particles (Leong *et al.*, 1995).

The DTA curves of the colloidal fraction for the three samples (Figure 2) show two apparent endothermic peaks at 310 and 620°C, due to dehydroxylation of goethite and serpentine, respectively, whose intensities vary with the relative proportions of both phases in each sample. In addition, an exothermic peak at 820°C is present due to crystallization of forsterite. Based on the TG weight loss at 310 and 620°C, an estimate of the serpentine to goethite ratio was obtained which compares well with that obtained by XRD (Table 1).

Particle morphology as observed by TEM is illustrated for sample GS (Figure 3). Platy crystals of serpentine with sizes smaller than $0.3 \times 0.2 \mu\text{m}$, intermixed with acicular crystals of goethite of length smaller than $0.2 \times 0.02 \mu\text{m}$ are clearly observed. Light-scattering measurements for the colloidal fraction of the three samples (Figure 4) give a broad peak with maxima at 0.4, 0.4, and $0.3 \mu\text{m}$ for samples SG, GS, and G, respectively, and a shoulder at larger sizes occurs probably due to aggregate particles. For bulk samples of the sediments, the mean-particle diameter

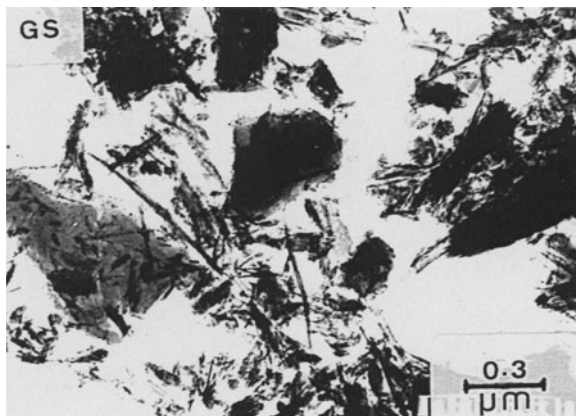


Figure 3. TEM micrograph for the colloidal fraction of sample GS.

(d_{50}) was 3.5, 3.1, and $2.4 \mu\text{m}$ for samples SG, GS, and G, respectively. It is noteworthy that reasonable agreement was found between the results obtained by light scattering and TEM for the colloidal fraction of each sample.

Rheological and electrokinetic study

Serpentine minerals are not stable in acids and they decompose by releasing Mg and Si ions at a rate strongly dependent on pH (Luce, 1964; Bates and Morgan, 1985). However, the colloidal properties of the samples studied here were nearly constant. A detailed study of the change in the colloidal properties of serpentine with time will be published elsewhere.

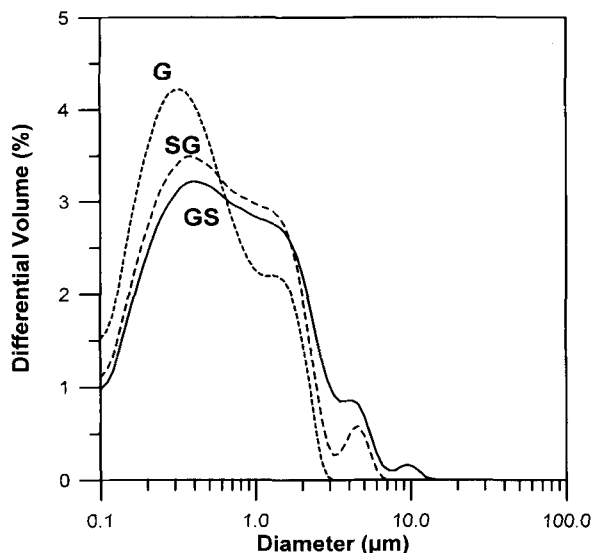


Figure 4. Differential volume (%) vs. diameter (μm) for the colloidal fraction of the samples studied.

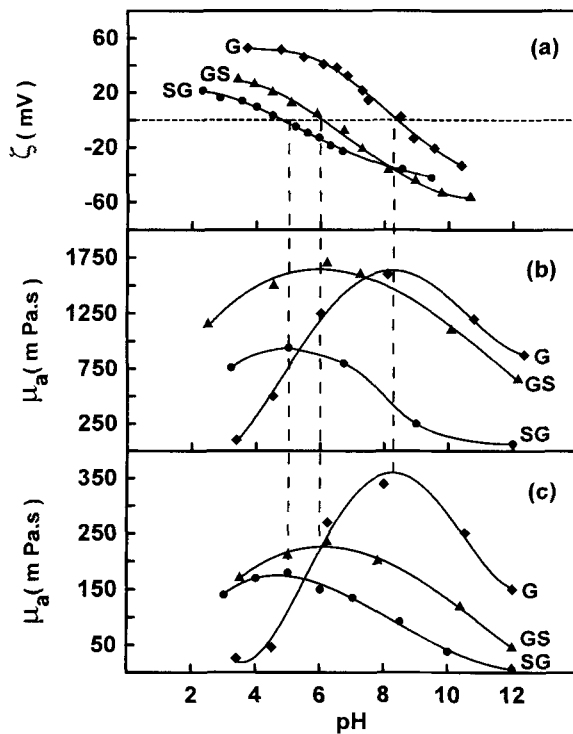


Figure 5. (a) Zeta potential as a function of pH suspensions for the colloidal fraction of samples SG, GS, and G; (b) Apparent viscosity ($\gamma = 200 \text{ s}^{-1}$) vs. pH for suspensions of the colloidal fraction containing 36 wt. % of solids; (c) Apparent viscosity ($\gamma = 200 \text{ s}^{-1}$) vs. pH for suspensions of the bulk samples containing 36 wt. % of solids.

Effect of pH

The most effective method for modifying the flow properties of a suspension is to modify the surface chemistry of the suspended particles, *i.e.*, the surface-charge density and the ionic strength, which is accomplished by changing pH and the electrolyte concentration of the suspensions (Hunter, 1987; Hiemenz, 1977). In this section, we examine the influence of pH on the stability and flow properties of concentrated suspensions of <36 wt. %, a value of suspension common in industrial processing of lateritic suspensions (Avotins, 1979; Valdés, 1984).

Figure 5a shows the effect of pH on the zeta potential for the colloidal fraction of the samples. The curves show a shape similar to those of single-phase solids, regardless of the serpentine-goethite mixture. In addition, as the goethite content increases, the IEP values shift toward that of “pure” goethite, 8.4 (sample G). This behavior is characteristic of systems involving heterocoagulation between two phases in solution. This behavior was previously observed in several types of suspensions (Yong and Ohtsubo, 1987; McLaughlin *et al.*, 1994). Heterocoagulation between kaolinite and iron hydroxides (*e.g.*, ferrihydrite, Yong

and Ohtsubo, 1987) occurs at certain pH values. Moreover, a coating reaction occurs between silica sand and different iron oxides (*e.g.*, goethite) at specific pH values (Scheidegger *et al.*, 1993). This relationship was assumed to occur from the positive charge of the adsorbed goethite particles by neutralizing the negative charge of the silica surface. We believe that a similar situation between goethite and serpentine must occur in the lateritic samples.

The IEP value (4.8) of the sample richest in serpentine (sample SG) is much lower than expected for a pure serpentine mineral (near 10). However, note that the pronounced weathering incurred by these samples (Vera, 1979) must promote an enrichment of Si with respect to Mg in the outer layers of the serpentine particles (Luce *et al.*, 1964; Bates and Morgan, 1985). Thus, the IEP is shifted ($\sim 1\text{--}3$) toward that of “pure” SiO_2 (Parks, 1965). In fact, measurements of the IEP of pure serpentine from the same deposits as the laterites range from 3.5 to 2.8 depending on the Si/Mg ratio, which suggests intense weathering. Figure 5a also illustrates the important effect that the mineral composition, *i.e.*, serpentine to goethite ratio, has on the stability of the suspensions. To fully understand the colloidal behavior, the degree of weathering of the serpentine component must be also considered.

For each sample, the maximum value of viscosity (Figure 5b) appears at a pH value close to the IEP where interparticle interactions are governed only by attractive forces. Therefore, maximum flocculation occurs. For samples SG and GS, this maximum is at pH (IEP) = 4.8 and 6.1, respectively, whereas for sample G, pH (IEP) = 8.4. Moreover, the magnitude of positive or negative surface charges (surface potential) increases as pH deviates from the IEP. Therefore, the suspensions will experience progressive deflocculation with decreasing pH below the IEP, and with increasing pH above the IEP (Hunter, 1987). For this reason, the lowest viscosity appears in all samples when the maximum value of the zeta potential is observed. In samples SG and GS, this value occurs at pH > 10 whereas in sample G, the lowest viscosity appears at acid conditions (pH = 3–5). It is noteworthy that strong thixotropy was observed at high concentration for the colloidal fractions, in particular at pH values close to the IEP of each sample. Under these conditions, the maximum value of viscosity is always subject to uncertainty (Schramm, 1994). This difficulty was more severe for sample G, which has the smallest particle size.

Similar behavior was observed for apparent viscosity as a function of pH between the bulk and colloidal fractions (Figure 5b and 5c). Therefore, the serpentine/goethite ratio also determines the flow behavior of lateritic suspensions in the bulk samples. The main difference between both fractions is that the viscosity values are always higher for the colloidal fraction, which is obviously due to smaller particle size, in agreement

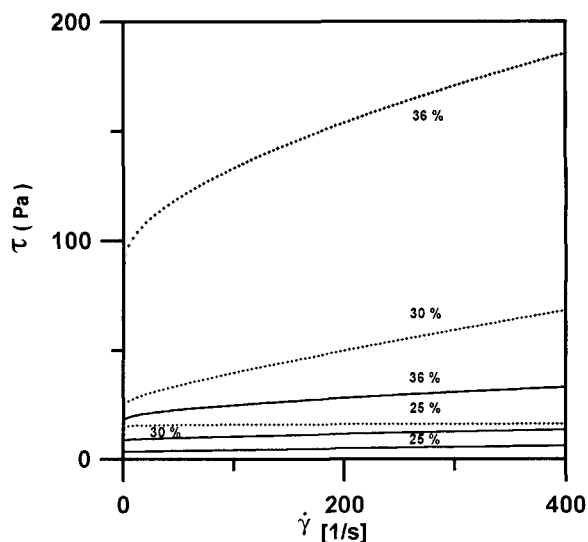


Figure 6. Shear stress vs. shear rate for suspensions of the sample SG for colloidal fraction ; bulk samples —.

with the idea that the rheological properties of the suspensions are mainly dominated by the fine particles. The maximum value of the apparent viscosity for the bulk samples (Figure 5c) increases as $SG < GS < G$, consistent with the trend in mean-particle diameter (d_{50}) determined by light scattering of 3.5, 3.1, and 2.4 μm for samples SG, GS, and G, respectively. However, it is noteworthy that suspension-viscosity dependence on particle size is not monotonic. Above $\sim 1\text{--}2\ \mu\text{m}$, which is the upper end of the colloidal range, the viscosity of suspensions increases with increasing particle size due to hydrodynamic effects rather than to surface effects. Therefore, large particles present in the bulk samples are apparently mostly aggregates composed of smaller particles.

The effect of particle size and solid concentration

The effect of particle size on flow properties is illustrated in Figure 6 by the differences found between the bulk and colloidal fraction of the sample richest in serpentine, sample SG. As expected, the shear stress is always greater for the colloidal fraction, the effect being more pronounced as the solid concentration increases. This behavior was shown in samples G and GS (data not shown), and it was observed previously in alumina suspensions (Velamakanni and Lange, 1991), and strongly flocculated clay systems (Buscall *et al.*, 1987).

Figure 7 shows the evolution of the apparent viscosity ($\gamma = 500\ \text{s}^{-1}$) with solid concentration for bulk samples at $\text{pH} = 6.0 \pm 0.3$, a value commonly found in industrial processes. The same exponential behavior also occurred in the colloidal fraction of each sample (data not shown), and this behavior was observed previously in many other systems (Avotins, 1979; Thom-

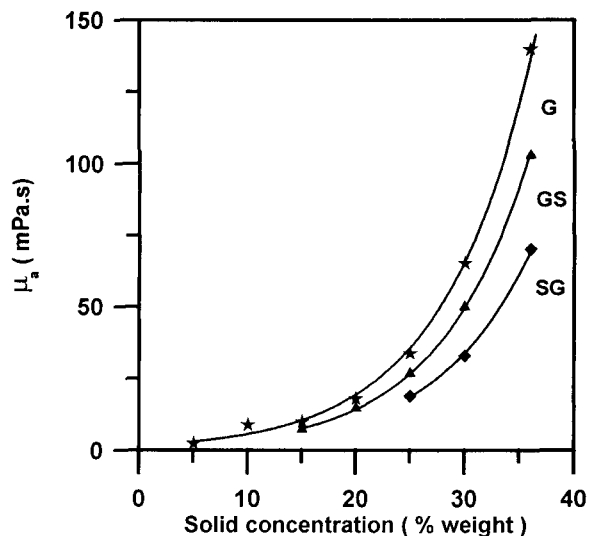


Figure 7. Apparent viscosity ($\gamma = 500\ \text{s}^{-1}$) vs. solid concentration for the bulk samples at $\text{pH} (6.0 \pm 0.3)$.

as, 1961). It is noteworthy that the value of the apparent viscosity increases here with the amount of goethite present in the samples, an effect that is more apparent as the concentration increases (Figure 7). This result occurs because goethite has a smaller particle size than serpentine.

At the IEP, only the van der Waals attractive forces govern the interactions of the particles. The yield stress (τ_0) was shown to follow a power relationship with concentration and particle size of the type (Leong *et al.*, 1995):

$$\tau_0 = K\phi^b/d^c \quad (1)$$

where K , b , and c are constants, ϕ is the volume fraction of the solids, and d is the particle diameter.

The effect of the concentration of each sample (through the volume fraction, ϕ) on τ_0 , for both the

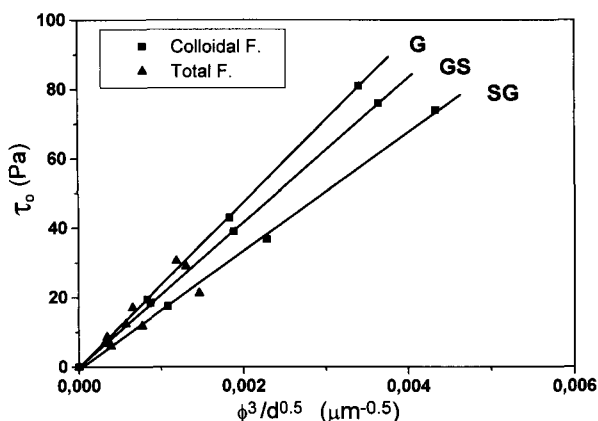


Figure 8. Plot of the maximum yield stress (τ_0) and $\phi^3/d^{0.5}$ for each lateritic sample.

Table 2. Comparison between calculated and experimental values of yield stress, τ_0 (Pa) as a function of sample concentration.

Solid conc. (wt. %)	Samples								
	SG ¹			GS ¹			G ¹		
	Calc.	Exper.	% dev.	Calc.	Exper.	% dev.	Calc.	Exper.	% dev.
	colloidal fraction								
25	17.6	17.5	0.7	18.0	18.4	2.2	20.0	19.5	2.5
30	37.3	36.8	1.3	38.8	39.0	0.5	43.8	43.0	1.8
36	71.0	74.0	4.2	75.3	76.0	0.9	81.6	81.0	0.7
	bulk samples								
25	6.4	6.0	6.3	6.5	6.8	4.6	8.7	8.6	1.1
30	12.6	11.8	6.3	11.8	12.4	5.0	15.5	17.1	10.3
36	23.9	21.2	11.3	27.0	28.9	7.0	28.2	30.5	8.1

¹ For the calculated values [equation (2)], $K = 1.64 \times 10^4$ for sample SG, $K = 2.07 \times 10^4$ for sample GS, and $K = 2.4 \times 10^4$ for sample G.

bulk and colloidal fraction of each laterite, shows a linear relationship with an exponent of ~ 3 in all samples, suggesting the predominant effect of the colloidal fraction in the value of τ_0 . This exponent is identical to that obtained for a number of oxide suspensions by Thomas (1961), but smaller than that reported ($b = 4$) in other concentrated suspensions (Leong *et al.*, 1995; Ramakrishnan *et al.*, 1996).

Finally, note the linear trend passing through the origin in the plot of τ_0 vs. $\phi^3/d^{0.5}$ for all samples (Figure 8). Thus for the lateritic samples, equation (1) takes the form:

$$\tau_0 = K\phi^3/d^{0.5} \quad (2)$$

with the K value decreasing with the serpentine to goethite ratio of the sample. A comparison between the experimental τ_0 values and those calculated by equation (2) is shown in Table 2, with the appropriate K value obtained for each lateritic sample. The agreement is in general good, giving an average percentage deviation of ± 4.3 . The exponent determined for the particle diameter, 0.5, is somewhat smaller to those reported in other oxide suspensions, 1–2 (Leong *et al.*, 1995; Ramakrishnan and Malghan, 1996). However, the existence in the lateritic samples of two types of solids heterocoagulated at the IEP can not be ruled out for the differences found.

CONCLUSIONS

Lateritic sediments from Cuba studied here are mostly composed of serpentine and goethite in different proportions. The rheological behavior of these samples is a function of the serpentine to goethite ratio of the colloidal fraction ($\leq 2 \mu\text{m}$) since this fraction constitutes $\sim 70\%$ of the solid components in the bulk sample. In each sample, the maximum of the apparent viscosity occurs at a pH value close to the IEP, at which interparticle interactions are mainly governed by attractive forces and thus, maximum flocculation occurs. The observed electrokinetic curves and the correlation between the IEP value and the serpentine

to goethite ratio suggest that heterocoagulation occurs in the studied samples. A relationship between the yield stress (τ_0) and the sample-volume fraction (ϕ) and particle size (d) was obtained at the IEP by the expression $\tau_0 = K\phi^3/d^{0.5}$, with the constant, K, depending on the mineral composition.

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