

Geraldo de F. Maciel  
maciel@dec.feis.unesp.br

Hamilton Kiryu dos Santos  
hamilton@dec.feis.unesp.br

Fabiana de O. Ferreira  
fabiana.of@gmail.com  
São Paulo State University-UNESP  
College of Engineering of Ilha Solteira  
15385-000, Ilha Solteira, SP, Brazil.

# Rheological Analysis of Water Clay Compositions in Order to Investigate Mudflows Developing in Canals

*This paper describes the physical and rheological property mixtures of water and kaolinitic clay that form the interstitial fluid found in muds from inundations or torrential lava. Several compositions with varying volume concentrations ( $C_v$ ) were tested in a high-precision R/S rheometer (shear rate  $\times$  shear stress). The rheometric parameters such as critical stress, apparent viscosity and outflow curves were established. A detailed analysis of the possible thixotropic fluid (shear thinning) and preparation of the mixtures were carried out. Such mud-type mixtures were well adjusted to the Herschel-Bulkley rheologic model at 03 parameters namely  $\tau = \tau_c + K\dot{\gamma}^n$ , where  $\tau$  is the shear stress;  $\tau_c$  the yield stress (or initial rigidity);  $k$  is the consistency term;  $n$  the flow index; and  $\dot{\gamma} = du/dy$  the deformation rate or shear rates. Considering that this is a thorough research that seeks to infer laws of friction on a canal taking into account the non-Newtonian rheology of the flowing material, it is observed that the tested mixtures specifically keep away from the Newton model, though less for the Bingham model, and adjusted well to the Herschel-Bulkley model, especially for lower deformation rates. Finally, from a refined literature review on the subject, it was appropriate to describe in detail a wider range of deformation rates and overall behavior laws of the various parameters of the Herschel Bulkley as a function of the concentration by volume ( $C_v$ ).*

**Keywords:** non-Newtonian fluid, Herschel-Bulkley, Mud, rheology

## Introduction

Mixtures resulting from Newtonian fluids and solid particles frequently appear in nature and in industry. These mixtures concede, as a rule, non-Newtonian properties. Generally, in the approach to these compositions, one finds residual sludge flows (sewage sludge), liquid charcoal, mud from drilling processes, fresh concrete, agricultural food fluids and torrential lavas, Fig. 1 – this real "sea of mud" and debris have periodically caused destruction and devastation with a substantial death toll in the national and international scenario.

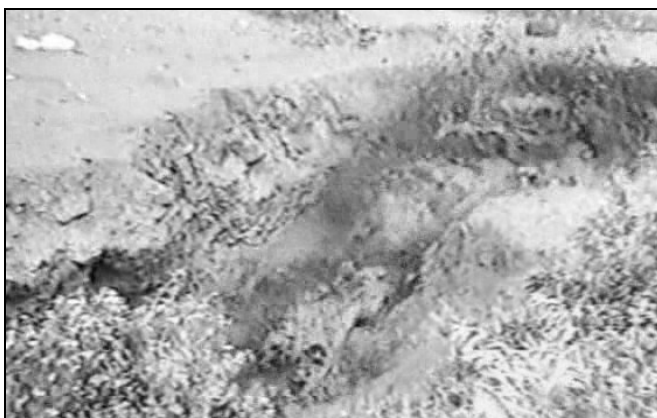


Figure 1. Mudflow due to rain causing erosion.

For these reasons, our work team has diagnosed more consistently and with engineering commitment the physical and rheological properties of the non-Newtonian clay matrix (water+clay mixtures – Fig. 2), which once characterized can be included in the dynamic study of mudflow in canals inferring on friction laws more

appropriate to flows with laminar behavior as in mudflows or turbulent behavior as in urban floods. Recent studies with water+clay+inert material mixtures (sands with varied grain sizes) are also in progress by the work team investigating a new friction law in waterways.

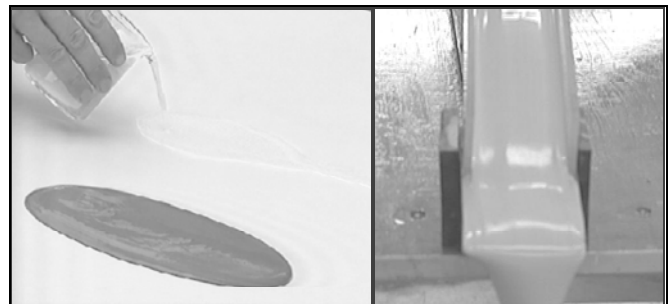


Figure 2. Water+kaolinitic clay mixture (non-Newtonian fluid) and water with colorant (Newtonian fluid) flowing at inclined planes.

## Nomenclature

- $C_v$  = Volume concentration (%)
- $k$  = Adjust parameter – consistency index ( $\text{Pas}^n$ )
- $n$  = Flow index
- $\rho$  = Density of mixture ( $\text{kg/m}^3$ )
- $\dot{\gamma}$  = Shear Rate ( $\text{s}^{-1}$ )
- $\mu$  = Newtonian viscosity (Pas)
- $\mu_{ap}$  = Apparent viscosity (Pas)
- $\tau$  = Shear stress (Pa)
- $\tau_c$  = Yield stress (Pa)

## General Considerations

It is known that a fluid with a small concentration of sediments retains its Newtonian properties; however, it can show variation in its viscosity, which then depends on this concentration. When the

sediment concentration increases, such that the particles begin to interact with each other, especially for sediments containing clay particles, the mixture no longer behaves as a Newtonian fluid, taking on non-Newtonian properties. Subsequently, this concentration varies with the grain size and composition of the sediment's mineral, as well as the water quality (Einstein (1956), Wang et al. (1994)).

There have been various attempts to establish a better explanation for the global and intrinsic dynamics of these mixtures in the last decade. The series of lab tests in which several rheological behavior laws have been discussed can be cited from the modeling attempts in literature, from the rare field data that includes numerical modeling. Among these attempts, the most common in literature is the Bingham plastic rheological model. However, recent studies, based on physical experimentation, demonstrate that all these clayey matrix and hyperconcentrated suspensions generally obey the Herschel-Bulkley viscoplastic non-linear rheological model (cf. Coussot (1992, 1997); Coussot et al. (1996); Piau (1996); Huang & Garcia (1998)), in which case a "binghamian" proposal becomes a particular case, and also occasionally interesting and convenient in certain engineering applications (Maciel et al., 1997).

### Rheology of Clay – Water Mixtures

Given that clay-water suspensions contain small particles, they can easily be tested with conventional laboratory rheometers. Additionally, these clay-water systems exhibit various behavior types and therefore often used as model fluids. The literature shows extensive studies on clay-water systems, yet our knowledge of their behavior remains incomplete. For sufficiently low shear rates, the mechanical behavior of clay-water mixtures or fine mud suspensions is always considered as being stable and homogeneous.

Dilute clay-water suspensions, for which hydrodynamic or Brownian effects may play a significant role, have rarely been studied. Concentrated suspensions, in which colloidal effects become predominant under usual flow conditions, can demonstrate nearly all the complex behavior types generally encountered in the field of rheology: yield stress, shear-thinning, thixotropy, viscoelasticity, etc. This paper begins by reviewing these characteristics from a general point of view and examines the influence of some parameters (clay type, pH, electrolyte, concentration, temperature and manner of preparation) on the behavior of the suspensions. In the second part, we review the theoretical models attempting to explain or predict the rheological behavior from internal processes.

The following synthesis will certainly seem critical of many previous works. Indeed, it is plausible that when considering the substantial amount of work carried out in this field over the last 50 years, only a relatively small fraction has substantiated important or relevant rheological information. We believe that this is due to the difficult task in obtaining reliable and interesting rheometrical data with concentrated suspensions rheometry, which requires numerous precautions; various disturbing effects that must be controlled may occur (fracture, slip, crack evolution, sedimentation, etc.). In addition, considering the accurate non-Newtonian character of most clay-water suspensions and the inherent uncertainty of rheometrical measurements, behavior transition or evolution with material characteristics (pH, concentration, temperature, etc.) can only be seriously regarded when, for a given material, a wide range of tests under different conditions has been carefully performed. For example, in order to have an idea of simple shear behavior in steady conditions, a simple apparent viscosity measurement at a given shear rate is insufficient. Measurements within a wide shear rate range are indispensable, since apparent viscosity depends on shear rate.

The shear-thinning behavior of these suspensions has often been noticed in the literature (Street, 1956; Weymann et al., 1973; Local and Demers, 1988; Akae and Low, 1988; Huifang et al., 1991; Coussot and Piau, 1994) and appears more directly visible in a linear scale diagram. Though shear-thinning is often associated with floc breakage, there is actually no microstructural theory capable of even predicting these curves qualitatively. To compare suspension properties or predict flow characteristics, the best one can do is to fit an empirical model to the experimental data and compare the values of the corresponding parameters.

The simplest theoretical model for yield stress fluid, i.e. the Bingham model, cannot be fitted to flow curves in the range of tested shear rates. Obviously, it is possible fitting this model to the experimental data in a narrow range of large shear rates, as many authors already have. However, very important material properties are overlooked when using this method. For example, the yield stress deduced from fitting this model to some data (plotted in a linear scale diagram for this fitting) in the range [20; 200 s<sup>-1</sup>] is at least twice as high as the real yield stress (for which an estimation is found by extrapolating the shear stress value at very low shear rates in logarithmic scale). As soon as the Bingham model is used to predict any flow, for which another range of shear rates (for example flow initiation) plays a major role, significant errors will be made. In addition, the yield stress value obtained by fitting a Bingham model does not specifically represent the strength of the suspensions, but only considers an arbitrary amount of viscous dissipations, which depends on the particular shear rate range used.

Consequently, it was proposed (Nguyen and Boger, 1983; Chhabra and Uhlherr, 1988; Doraiswamy et al., 1991; Coussot and Piau, 1994; Sherwood, 1994; Maciel et al., 1996) fitting a Herschel and Bulkley (1926) model to the experimental data for the whole experimental shear rate range.

$$\tau = \tau_c + k\dot{\gamma}^n \quad (1)$$

Where  $\tau$  is the shear stress magnitude,  $\dot{\gamma}$  the shear rate magnitude, and  $\tau_c$ ,  $k$ , and  $n$  the positive parameters. Clearly here  $\tau_c$  is an empirical yield stress. Other models can be used (see the review of Lapasin (1985)) but they contain more parameters or do not represent experimental flow curves in a wide shear rate range. Apart from the usual numerical fitting procedures, the following practical fitting method may be used. On plots  $(\tau - \tau_c)$  against  $\dot{\gamma}$  on a logarithmic scale diagram with different tests for the values of  $\tau_c$ , until it is possible to draw a satisfactory straight line through these points. The slope and position of this straight line give  $n$  and  $k$ . It is worth noting that the shear stress level corresponding to a shear rate of about 0.01 s<sup>-1</sup> is generally very close and just above the real yield stress (Coussot et al., 1993; Coussot and Piau, 1994; Mas and Magnin, 1994; Coussot and Boyer, 1995). Finally, using a Herschel-Bulkley model fitted to values in a wide shear rate range, including values as small as 0.01 s<sup>-1</sup>, provides a theoretical yield stress that is very close to real yield stress, with an uncertainty that is sufficiently small for conventional practical applications. However, for some suspensions that display a peculiar behaviour, this method can be quite inappropriate (such as bentonite-water mixture exhibiting a minimum in flow curve (Coussot et al., 1993)).

An important question is whether the Herschel-Bulkley model (or the corresponding parameters) does or does not express any physical mechanism occurring within the material during the flow. For yield stress the answer was given above: it actually reflects the strength of a continuous network of interaction. Concerning the part of the shear stress expression, which is shear rate dependent in equation (1), the question remains open. It has often been

erroneously considered that the Bingham model found for clay-water mixtures reflects the sum of the energy needed to break the particle network, by means of yield stress and viscous dissipations, using the second term identical to the stress for a Newtonian fluid. To simplify the description of internal phenomena we can consider that, during motion, there are essentially two sources of energy dissipation: (a) ruptures and restorations of links between clay particles within the material, and (b) usual hydrodynamic dissipations due to the shear of interstitial free water. As soon as there is a flow, the structure is continuously broken and restored in part, even though each particle obviously occupies a new position.

The complete network (with all the links) is only obtained after a sufficiently long (in theory infinite) resting time. Thus, at each instant a transient network or a set of clusters exists and the shear stress intensity to be applied to maintain the shear may therefore be not far from the yield stress value. In all likelihood, since the complete network no longer exists, the stress needed to break and restore a certain number of links is different from the yield stress. Thus, physically, during flow, the shear stress is not the sum of a constant force needed to break the static network and an additional force that is needed to maintain the shear of the suspension of broken flocs, even if a constant fluid "yield stress" appears in the flow curve. In fact, the total shear stress, either for the Bingham model or the Herschel-Bulkley model, expresses a complex sum of viscous dissipations related to the deformations of the transient network or to the overlapping or friction of the flocs. The following critical example clearly demonstrates the aforementioned statements. In the case of concentrated dispersed systems in a low molecular weight matrix, it was shown that a minimum in the flow curve can exist. This means that, beyond yielding, the shear stress necessary to maintain a flow can either be higher or lower than the yield stress, depending on the characteristics of the material. Finally, both terms of equation (1) are not independent at all as long as there is actually a flow and both of these express the two types of phenomena (a and b) (Coussot et al., 1993; Coussot and Piau, 1994). Consequently, and this is even more noticeable for a Herschel-Bulkley fluid, none of the parameters  $k$  and  $n$  a priori represent a simple specific property of the fluid, unlike viscosity in Newtonian cases or yield stress.

## Rheometry

### Generalities

It is known that, under the same initial and boundary conditions, the characteristics of a flow depend solely on the material composition (in flow). This fact led to the development of empirical methods (such as slump test), in which the materials are subjected to forces and movements and react differently according to their composition. Thus, the different reactions presented by the materials were used for a comparison between them. These techniques are usually standardized in fresh concrete or in the food industry, for example. However, in the case of fluid flows, these methods do not provide relevant information, since the conditions of these flows are complex and far from being controllable; therefore, the data collected in the test portray the flow behavior in a particular moment, thus not reliable for the fluid's characterization.

Rheometry aims at determining the fluid characteristics from measurements performed in simple and controlled flows. In these tests, the fluid is stressed in a simple manner, such that few components from its stress tensor differ from zero. Thus, from the components of shear stress and shear rate, one can obtain a characteristic equation.

In practice, in order to measure a fluid's viscosity, the fluid should be confined between some devices, with determined edge

conditions (one fixed and another with fixed speed). The fluid should be confined in a tube, between parallel plates, between concentric cylinders, etc. These devices are defined as rheometers.

The most appropriate choice of geometry for complex materials, like mud, is an extremely important point. In practice, for any fluid, it is very difficult to find the geometry that will provide a perfectly homogeneous shear rate between the plates (cones, cylinders, disks). Therefore, in the majority of cases, there is a heterogeneous shear rate, and conventional rheometric data only shows the average flow characteristics (torque, rotation speed, pressure) at the edges. In such cases, some calculations are required to obtain the shear stress  $\times$  shear rate data from the torque  $\times$  rotation speed values. The calculation methods differ for each geometry.

### Measuring Strategies

From the proposition of a model that could explain the rheological behavior of the tested mixtures, together with the experimental data in hand, it is necessary defining the method to be used for accurately calibrating the parameters of the model in question.

For the Herschel-Bulkley model, for example, an option would be to plot  $(\tau - \tau_c) \times \dot{\gamma}$  in a log-log scale for different  $\tau_c$  value attempts until a satisfactory curve is traced from the points obtained experimentally, as commented earlier. The position and curvature of this characteristic curve would supply the values of  $k$  and  $n$  (Herschel-Bulkley parameters).

In the case under study, there is no need to look for a curve with better adaptation to the experimental data, since the Rheo2000 software used together with the rheometer provides the tuned calibration of the Herschel-Bulkley parameters, as well as the characteristic flow curve of each tested material. The rheometer used is a R/S Rheometer (shear stress  $\times$  shear rate) manufactured by Brookfield Engineering Laboratories, equipped with heat bath (Fig. 3). The geometry chosen uses coaxial cylinders as in the Fig. 4.

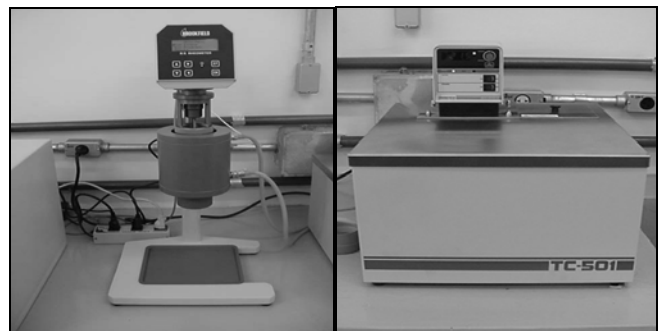


Figure 3. R/S Rheometer and heat bath.

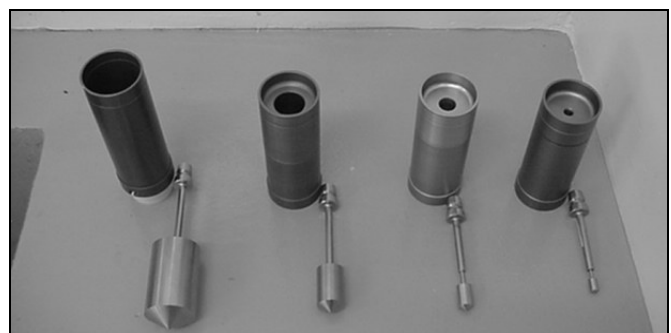


Figure 4. Spindles used.

## Experimental Procedure

The procedure used in the rheometric tests is simple and repetitive, and can be summarized as follows:

- First, the entire apparatus is connected (rheometer, heat bath and microcomputer);

- Next, the heat bath temperature is set up. The working principle of this equipment is to maintain the temperature of the circulating fluid by means of heating and cooling fins. This fluid circulates through the "heat jacket" (accessory coupled to the rheometer in Fig. 3), which envelops the spindle, maintaining the sample temperature constant. Depending on the temperature range at which it is working, a type of circulating fluid is used. For temperatures varying between 5 and 80°C, the fluid is water. The experiments were run at about 25°C.

- The chosen spindle containing the previously prepared sample is fixed to the rheometer. The spindle is chosen according to the sample's "consistency", since each accessory covers a viscosity range in which it can work. When the chosen spindle is unable to conduct the test, it should be replaced.

- In the software, the entire test procedure is configured: the option for shear stress or shear rate controlled test is made, the spindle used should be indicated, the test time and the shear rate or stress imposed is defined (for gathering shear stress or shear rate data, respectively). Next, the rheometer is activated from the software, and the test starts.

- To obtain the results, close to 70 experimental tests were conducted.

Due to the rheometer's high sensitivity, calibration should be performed weekly, for continuous reliable data.

## Results

All tests were conducted in the rheometer (R/S) using the shear stress variation method and measuring the respective shear rates. One can also opt for a variation of shear rate and shear stress measurement; however, we decided to maintain a single protocol and regularity in the tests as a prudent measure.

### Physical Characterization – Chemistry of the Clay Used

The physical-chemical characteristics of the clay used, supplied by the mining company, were controlled *a posteriori* by the soil team of the Soil Mechanics Lab from FEIS/UNESP, and can be listed as follows:

- Ore: Kaolinitic clay (with non-expansible properties);
- Real density (dry): 1.5 to 1.65 g/cm<sup>3</sup>;
- Ground apparent density: 0.5 to 0.65 g/cm<sup>3</sup> (325 mesh);
- Apparent porosity: 16.00 to 18.50%;
- Water absorption: 9 to 10%;
- Particles below 5 microns: 65 to 70%;
- pH: 5 to 6.

### Temperature Variation Analysis

The results below are to elucidate certain characteristics of the tested mixture (water+kaolinitic clay, in different volume concentrations -  $C_v$ ) in relation to temperature variation. For a certain sample, with a volume concentration ( $C_v$ ), pH and pre-established density, the temperature was varied with the aid of the heat bath in a range of approximately 10°C to 80°C. Figure 5 represents the variation of shear stress x shear rate curves as a function of this parameter.

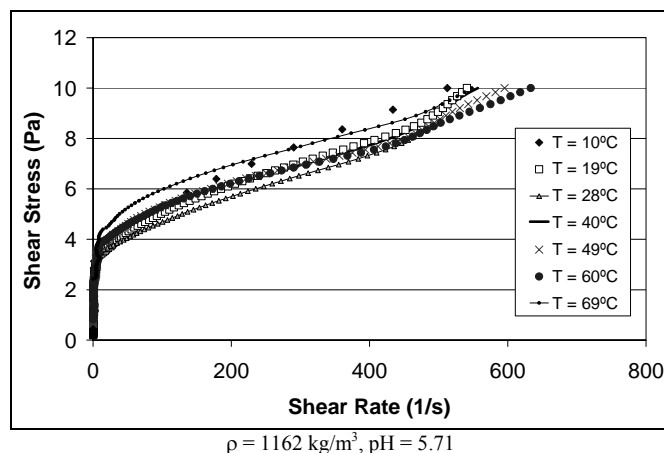


Figure 5. Variation of flow curves in function of temperature for  $C_v = 10\%$ .

As analyzed, the temperature variation in the sample did not seem to significantly influence the mixture's flow curve, especially if a temperature range, including the room temperature, is considered ( $20^\circ\text{C} < T < 30^\circ\text{C}$ ). In addition, the tests were conducted in an acclimatized lab (temperatures below  $20^\circ\text{C}$  were not registered in all the tests). Due to the material's low sensitivity to temperature variation, its control was not regarded, since the room temperature variations did not significantly influence the flow curve.

### Analysis of the Mixture's Ionic Potential Variation

According to Coussot (1992), the rheological characteristics of water+clay mixtures may or may not undergo significant changes with a variation in the number of ions and pH of the mixture. These variations can influence the type of cationic interaction in the double-chamber effect (effect responsible for interaction between colloidal particles inside the dispersing liquid). Thus, various changes in behavior can occur depending on the type of clay used and the type of ions added.

In addition, according to Pandolfelli (2000), the ionic potential and pH variation are interconnected, since ionic potential variations can influence the pH and vice-versa.

Therefore, tests were conducted to determine the variation influence in the number of ions in the interaction of particles inside the mixtures, in addition to the consequent influence on their rheological behavior.

The ion variations were a result from faucet water (Hydrology Lab from FEIS/UNESP) and distilled water variations, as well as the addition of NaCl to the solution. To measure the variation in the number of free ions in the mixtures, the pH variations (pH of a solution indicates the number of free H<sup>+</sup> ions in this solution; thus, variation in the number of other free ions in the mixture can cause a variation in hydrogenionic potential) were measured.

Figures 6 and 7 show the rheological behavior differences of the mixtures as a function of the ionic potential of the mixtures for two different volume concentrations ( $C_v = 10\%$  and  $C_v = 15\%$ ).

The  $C_v = 10\%$  and  $C_v = 15\%$  concentrations clearly indicate the Herschel-Bulkley rheologic behavior expected for these clay mixes. Concentrations higher than  $C_v = 20\%$  would jeopardize the experimental results due to the rheological torque used.

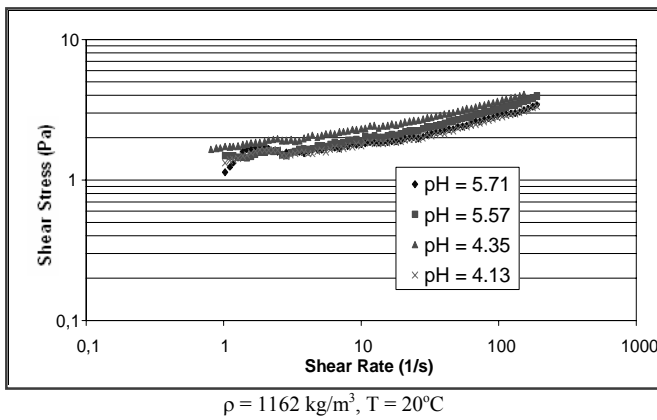


Figure 6. Variation of flow curves as a function of pH variation for  $C_v = 10\%$ .

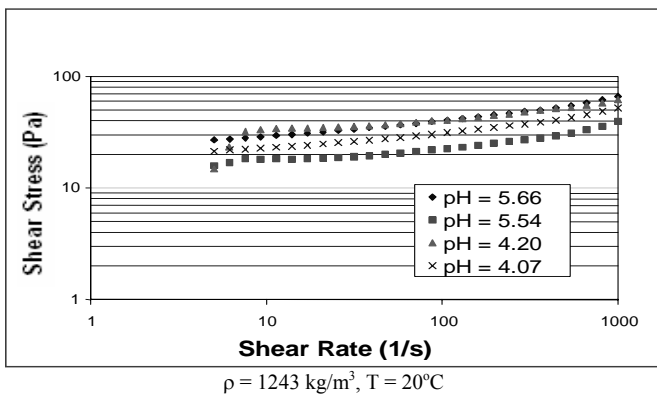


Figure 7. Variation of flow curves as a function of pH variation for  $C_v = 15\%$ .

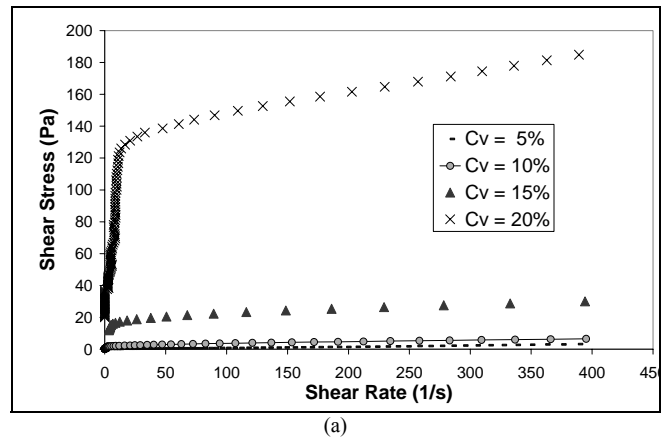
The results demonstrate that the variation influence in the number of free ions of the mixture in the shear stress  $\times$  shear rate curve is small, considered almost negligible for this study. However, the variation of this parameter proved to be more significant than the temperature effect on the shear stresses.

#### Analysis of Volume Concentration ( $C_v$ )

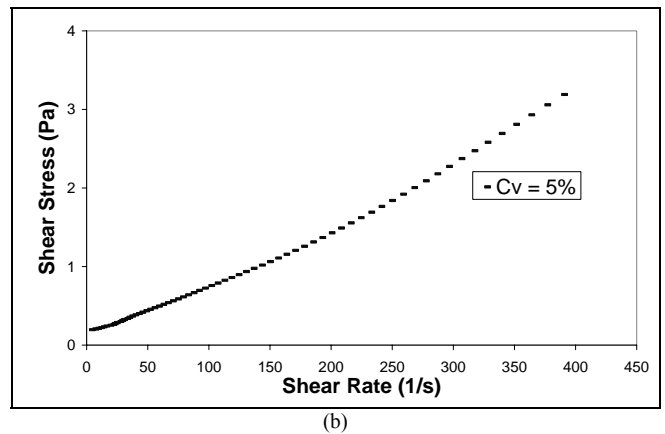
It is known that the viscosity of a hyperconcentrated fluid increases when the concentration of solids in the mixture is increased. This occurs because the greater the quantity of solid material, the greater is the internal frictional forces in the flowing fluid, and consequently, the shear stress that causes a certain shear rate will have to be greater.

Thus, it can be observed that the initial rigidity of the fluid increases with concentration, and therefore the minimum shear stress required to cause a shear rate (yield stress) is also greater.

In agreement with the literature, the  $C_v = 5\%$  concentration for this type of mixture still indicates predominant Newtonian properties, a proven and measured fact. These affirmations are evidenced in Fig. 8.



(a)

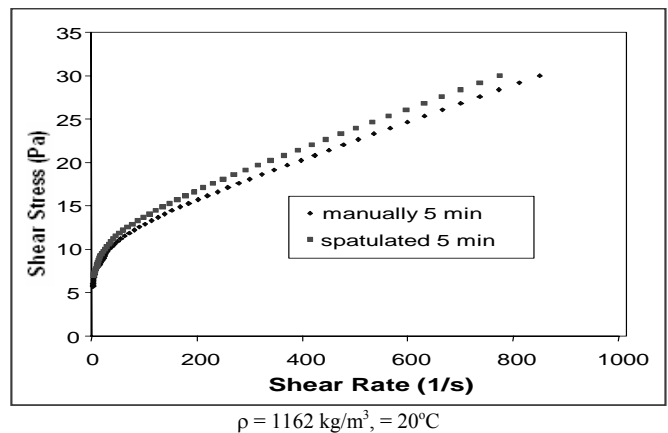


(b)

Figure 8. Variation of flow curves as a function of variation in volume concentration  $C_v$ , for  $T = 20^\circ\text{C}$ .

#### Analysis of the Mixture Preparation Influence

The preparation manner, whether by spatulating the sample, by mixing in the rheometer itself, or by manual agitation, is a parameter of which influence changes with time after preparation. The results presented in Figs. 9 and 10 are from two differently prepared samples, however, with the same volume concentration –  $C_v$ . The first mixture was agitated manually for 5 minutes in a closed recipient, while the second was gently spatulated for 5 minutes. Both were tested at the same temperature and it was noticed that the sample preparation manner practically did not influence the shear stress  $\times$  shear rate curve. The results show that the preparation manner did not influence the tests significantly.



$\rho = 1162 \text{ kg/m}^3, T = 20^\circ\text{C}$

Figure 9. Mixture preparation influence for  $C_v = 10\%$ .

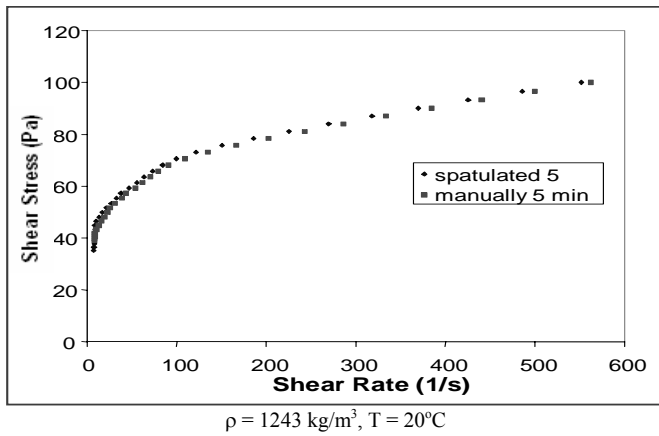


Figure 10. Mixture preparation influence for  $C_v = 15\%$ .

In fact, this affirmation is partially true because due to the long time spent saturating the clay particles, in samples that were tested at a time very close to the preparation (up to 48 hours), the manner of preparation considerably influences the mixture's rheological behavior. Meanwhile, for mixtures tested after a long resting time (over 48 hours), the preparation manner has little influence on the flow curve, as almost all the clay grains are already saturated.

Figure 11 shows the first and second tests of a sample with a volume concentration of 15% for different resting times (1, 2 and 35 days), after preparing the sample. One can see the resistance gain in the curves with time and with the sequence of tests.

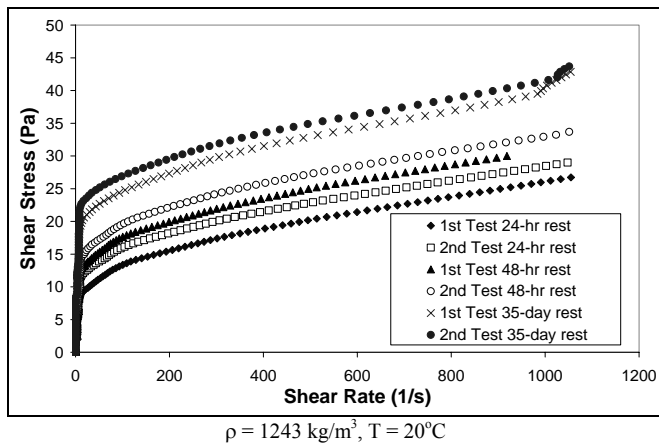


Figure 11. Shear stress x shear rate curves for the 1<sup>st</sup> and 2<sup>nd</sup> tests for different rest periods – sample with  $C_v = 15\%$ .

Considering that the sample tested with a 35-day rest is close to the maximum resistance attained by the sample, and also considering that the shear test can be regarded as a form of mixing, the following analysis can be obtained from Fig. 12:

- To reach resistances close to the resistance of the first test with a 35-day rest, the sample tested with 24 hours had to be tested 8 times and the sample with a 48 hour resting time required 5 tests;

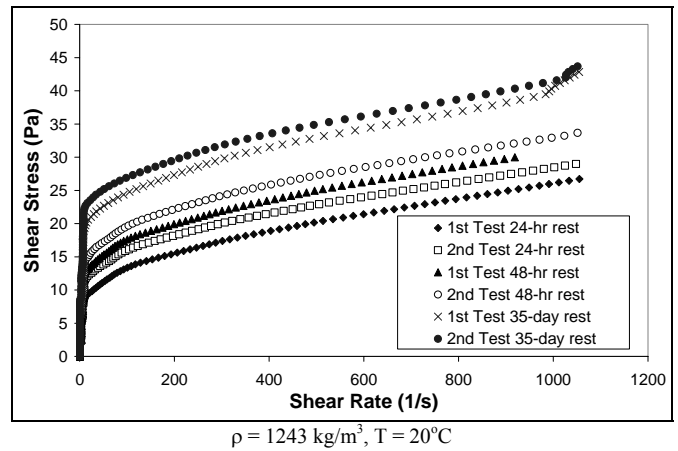


Figure 12. Comparison of shear stress x shear rate curves for a single sample with  $C_v = 15\%$  and different rest times.

- Analyzing the form of mixing in the tests, one concludes that the sample with 1-day resting time (24 hours) had a more vigorous mixing (therefore 8 times) than the sample that rested for 2 days (48 hours) in order to reach the resistance of the sample that rested for an extended period of time (35 days).

Thus, for relatively short resting times, the preparation manner should influence the mixture's flow curve.

#### Analysis of the Thixotropy Occurrence

Analysis of flow curve variation as a function of time (thixotropy) is an important aspect in rigorous rheological characterization of a hyperconcentrated mixture.

For these assays, 2 types of tests were conducted: in the first, the rheometer is programmed so as to impose increasing (loading) and decreasing shear rates (unloading) in various cycles of 120 s each, the cycles can be seen in Fig. 13a. For this assay (Fig. 13b), one can notice a larger shear rate of the sequent cycles in relation to the maximum point attained by the first cycle, this indicates a larger shear rate for the subsequent cycles in relation to the first, which can occur due to the sample, after the first cycle is unloaded, hence not being able to fully recover the initial resistance before being loaded again, and thus attain a larger shear rate than in the previous cycle.

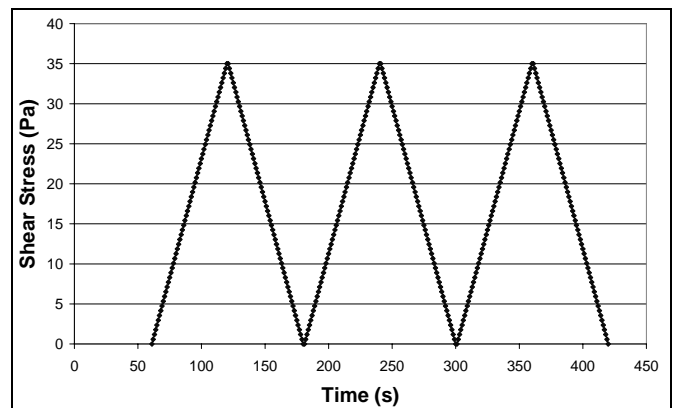


Figure 13a. Loading and unloading cycles.

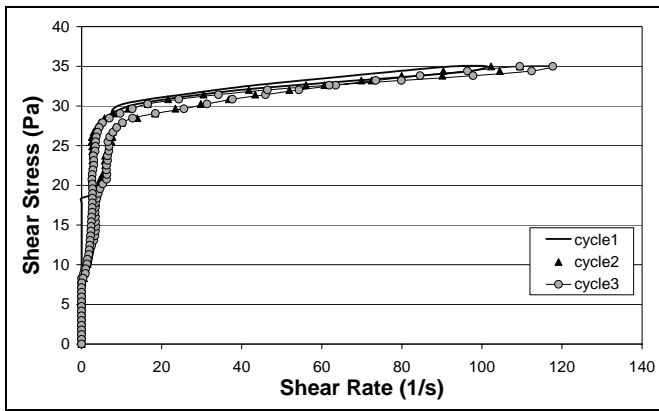


Figure 13b. Shear stress x shear rate curves for loading and unloading cycles for samples with  $C_v = 15\%$ .

In the second type of assay, constant loading track assays were conducted with resting periods. These tests analyze the sample's capacity to recover its original resistance as a function of rest and of the shear rate applied. To better understand tests of this nature, it is pertinent in this item to recall that the apparent viscosity of Non-Newtonian fluids is a function of the shear rates. For the Herschel-Bulkley rheological model adjusted to the tested samples, the apparent viscosity  $\mu_{ap}$  is given by:

$$\mu_{ap} = \frac{\tau}{\dot{\gamma}} = \frac{\tau_c}{\dot{\gamma}} + k\dot{\gamma}^{n-1} \quad (2)$$

While the "differential viscosity" (viscosity) is given by:

$$\frac{d\tau}{d\dot{\gamma}} = kn\dot{\gamma}^{n-1} \quad (3)$$

Thus, as can be seen in Fig. 14 (hypothetical figure), the "differential viscosities" ( $\mu = \mu(\dot{\gamma})$ ) represented by the tangents ( $\tan \alpha_1 > \tan \alpha_2$ ) and the apparent viscosities ( $\mu_{ap} = \mu_{ap}(\dot{\gamma})$ ) represented by the tangents ( $\tan \beta_1 > \tan \beta_2$ ) for smaller shear rates are greater than the viscosities for higher rates.

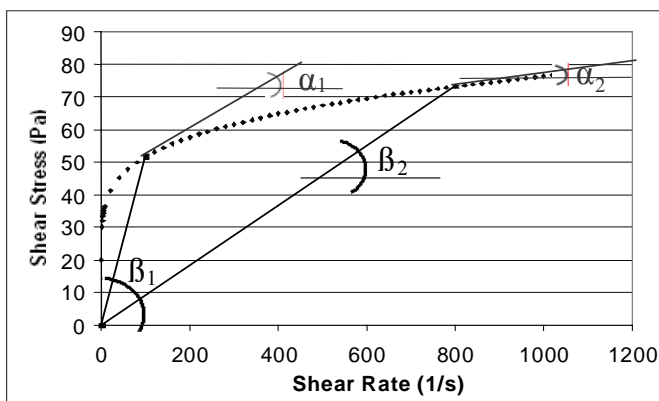
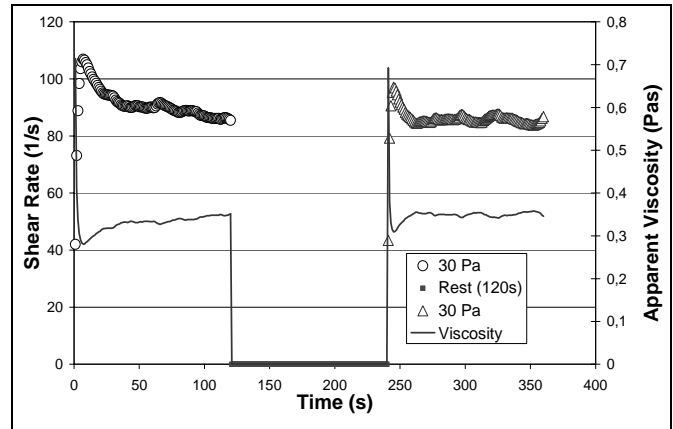


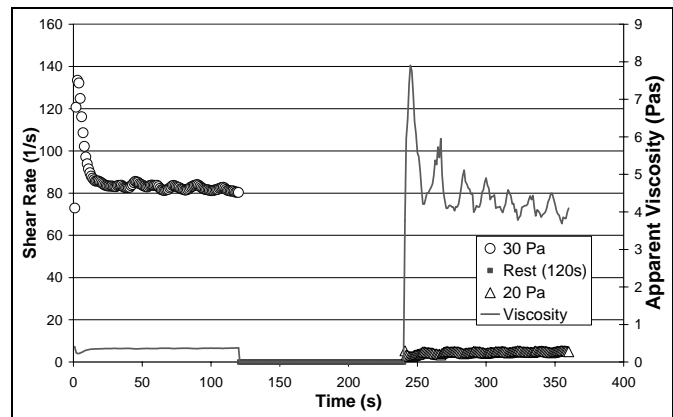
Figure 14. Viscosity variation as a function of the shear rate for a Herschel-Bulkley type hyperconcentrated fluid (actual viscosities  $\tan \alpha_1$  and  $\tan \alpha_2$ ; apparent viscosities  $\tan \beta_1$  and  $\tan \beta_2$ ).

For thixotropic tests, with constant loading tracks, the results can be seen in Figs. 15, 16a and 16b:



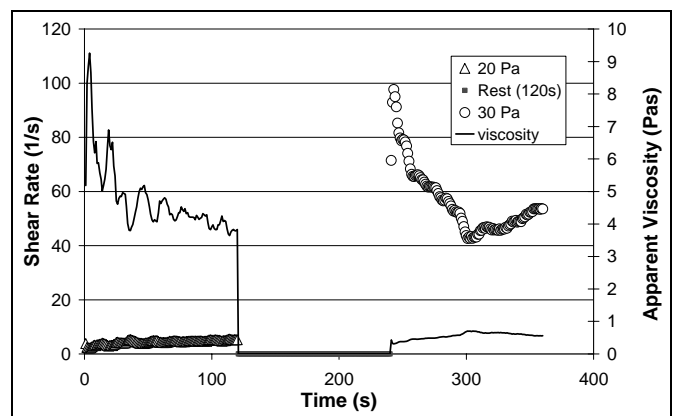
$C_v=15\%$ ,  $\rho=1243 \text{ kg/m}^3$ ,  $\text{pH}=5,66$ ,  $T=20^\circ\text{C}$ .

Figure 15. Shear rate x viscosity x time curves for sample with  $C_v = 15\%$  and constant loading of 120s followed by rest and constant loading again of 120 s.



$C_v=15\%$ ,  $\rho=1243 \text{ kg/m}^3$ ,  $\text{pH}=5,66$ ,  $T=20^\circ\text{C}$ .

Figure 16a. Shear rate x viscosity x time curves for sample with  $C_v = 15\%$  (1st constant loading track of 30 Pa and 2nd constant loading track of 20 Pa, with rest of 120 s).



$C_v=15\%$ ,  $\rho=1243 \text{ kg/m}^3$ ,  $\text{pH}=5,66$ ,  $T=20^\circ\text{C}$ .

Figure 16b. Shear rate x viscosity x time curves for sample with  $C_v = 15\%$  (1st constant loading track of 20 Pa and 2nd constant loading track of 30 Pa, with rest of 120 s).

Figure 15 illustrates that the mixture's apparent viscosity is constant at approximately 0.35 Pas in both loading tracks. It is also possible to notice that after resting the sample becomes viscous again and then loses it with the start of the second loading.

With relation to Figs. 16a and 16b, it is possible to notice that the Viscosity x Time Curves are inverted (in Fig. 16a, the first threshold is 0.30 Pas and for the second it is 4.50 Pas, while in Fig. 16b the first threshold is 4.50 Pas and the second is 0.50 Pas). In Fig. 16a, the first shear rate reduces the sample's viscosity, and with the rest period, the sample regains its viscosity and, as the second shear rate is lower than the first, the viscosity increases again. Moreover, when comparing the viscosity results of Figs. 16a and 16b inferred by Fig. 13b, we arrive at the same order of magnitude for stresses of 20 to 30 Pa.

However, Fig. 16b indicates that the first shear stress track produces a higher viscosity than the second track, however maintaining the same order of magnitude of the viscosities measured for each one of the shear stress tracks, shown in Fig. 16a. These results are coherent and express the reality shown in Fig. 14 and aforementioned.

Tests were also conducted with gradual loading and unloading tracks with the same loading and unloading time and with different times. The results are in Figs. 17a and 17b and in 18a and 18b.

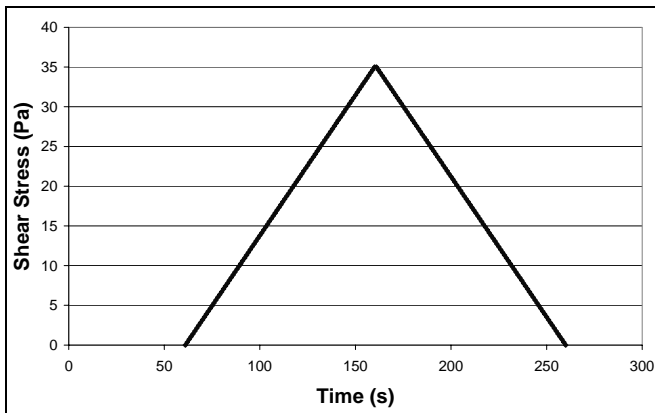


Figure 17a. Curves with the same gradual loading and unloading intervals (100 s of loading and 100 s of unloading) for sample with  $C_v = 15\%$ .

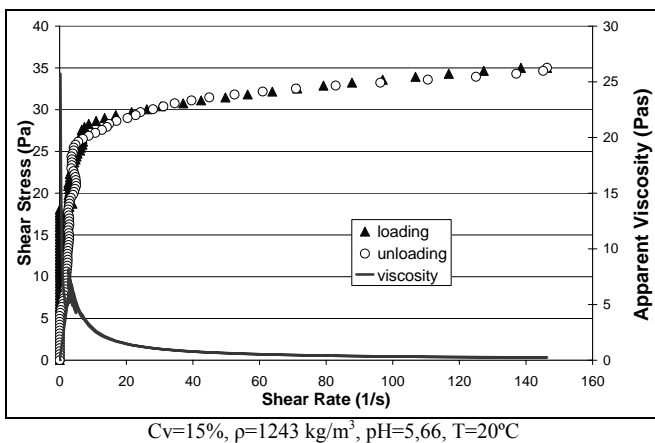


Figure 17b. Shear stress x shear rate curves for the loading and unloading configurations shown in Fig. 18a – sample with  $C_v = 15\%$ .

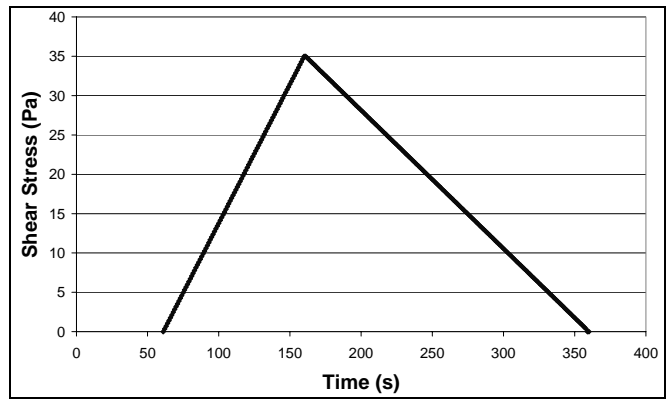


Figure 18a. Curves with unequal gradual loading and unloading intervals (90 s of loading and 210 s of unloading) for sample with  $C_v = 15\%$ .

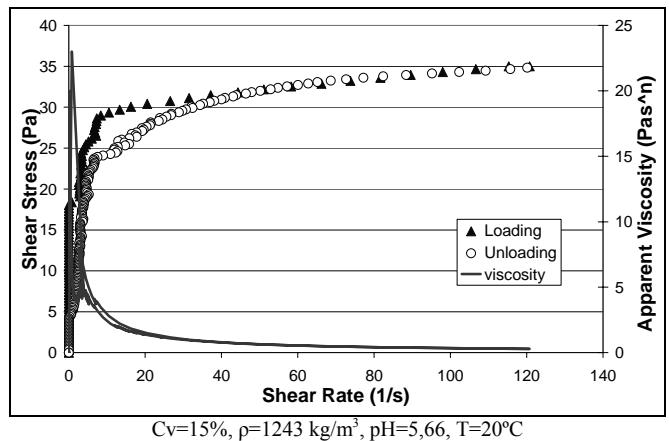


Figure 18b. Shear stress x shear rate curves for the loading and unloading configurations shown in Fig. 19a – sample with  $C_v = 15\%$ .

Comparing Figs. 17b and 18b, it can be observed that the loading and unloading curves at different times distance the corresponding Shear stress x Shear rate curves. This behavior indicates that the sample only responds equally for equal test times. With these results, one can conclude that the material has moderate thixotropy, since the material properties are recovered in short-term periods, in addition to showing the same characteristics for equal test times.

#### Analysis of the Tixotropy Occurrence Proposal of a Behavior Law for Controlled Tested Mixtures (Water+Kaolinitic Clay)

Next, it was regarded appropriate to show certain behavioral trends for some concentrations ( $C_v = 10\%$ ,  $C_v = 15\%$  and  $C_v = 20\%$ ) and a comparison of the Hershel-Bulkley model (with correlation to the experimental data  $R^2 > 98\%$ ) with the Binghamian and Newtonian model (Figs. 19, 20 and 21) was undertaken. The adjustment of the rheological models shown in Figs. 19, 20 and 21 and in Tab. 1 was obtained with the Rheo2000 software that comes with the R/S Rheometer used in this research.



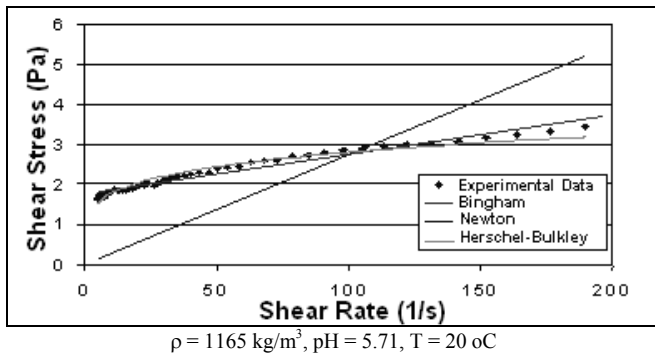


Figure 19. Rheological behavior of water+kaolinitic clay mixture with  $C_v = 10\%$ .

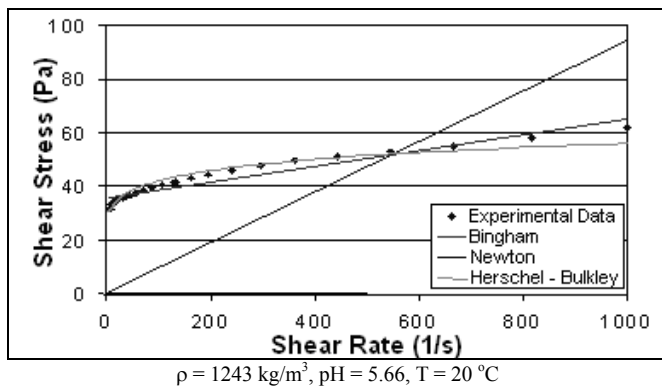


Figure 20. Rheological behavior of water+kaolinitic clay mixture with  $C_v = 15\%$ .

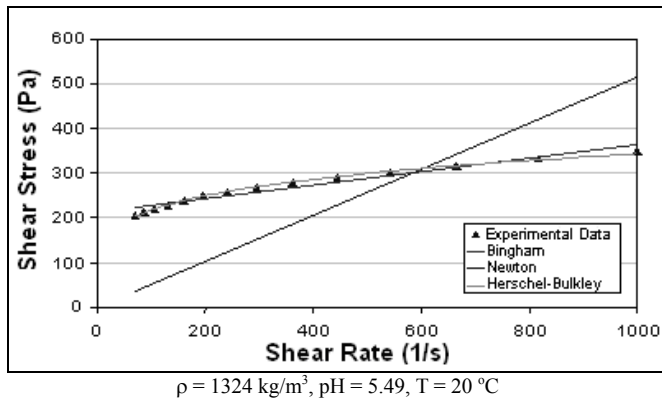


Figure 21. Rheological behavior of water+kaolinitic clay mixture with  $C_v = 20\%$ .

The Best fit found for the tested samples, as well as for  $C_v = 10\%$ ,  $C_v = 15\%$  and  $C_v = 20\%$  was by the Herschel-Bulkley. Although the Binghamian model might represent the material behavior for high deformation rates, it will overestimate the stress and especially the critical stress in situations of low deformation rates, thus the obvious option for the Herschel-Bulkley model.

Table 1. Parameters found in the adaptation of rheological models for water+kaolinitic clay mixtures.

$C_v$ (%)	Adjustment with Herschel - Bulkley				
	$\tau_c$ (Pa)	$k$ (Pas <sup>n</sup> )	$n$	$R^2$	
10	1.26	0.22	0.47	0.99	
15	29.45	1.30	0.46	0.98	
20	192.27	46.29	0.27	0.98	
$C_v$ (%)	Adjustment with Bingham			Adjustment with Newton ( $\tau_c=0$ e $n=1$ )	
	$\tau_c$ (Pa)	$\mu_B$ (Pas)	$R^2$	$\mu$ (Pas)	$R^2$
10	1.7529	0.010	0.960	0.027	-
15	35.818	0.029	0.929	0.094	-
20	211.66	0.152	0.950	0.515	-

### Global Quantitative Analysis of Rheological Parameters in Function of $C_v$ Variation

Bibliographical investigations (Wan, 1982; Huang and Garcia, 1998; Coussot, 1992) indicate that parameters of the proposed Herschel-Bulkley rheological model commonly follow, for kaolinitic clays, the following standard: values of  $\tau_c$  (yield stress) and  $k$  (consistency index) increase exponentially with volume concentration of the mixture's solids. In counterpart, the value of  $n$  (flow index) reduces with concentration, obeying a power function. These results were checked for volume concentration ranges of  $C_v < 10\%$  and  $C_v > 20\%$ . However, the tests conducted by this team varied in a broad range of  $5\% < C_v < 25\%$ .

Based on this information, one aims to check correlations pointed out between the rheological data of the tested mixtures and verify their agreement with the results from literature. To this effect, Figs. 22 to 27 show the data obtained in the experiments as well as the data already available in literature. From this new database, quantitative global behavior laws are defined for each rheological binomial.

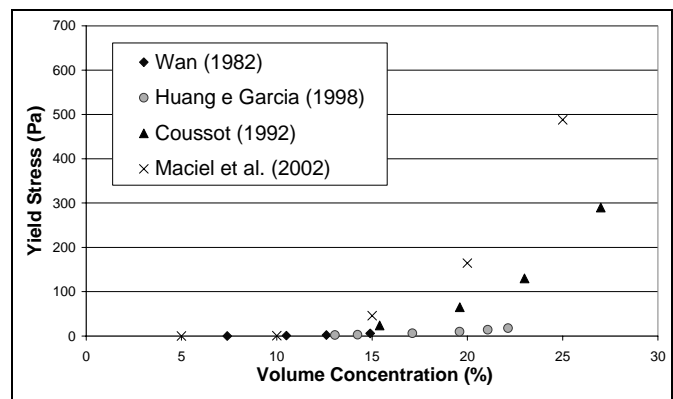


Figure 22. Progress of yield stress  $\tau_c$  in function of volume concentration  $C_v$ .

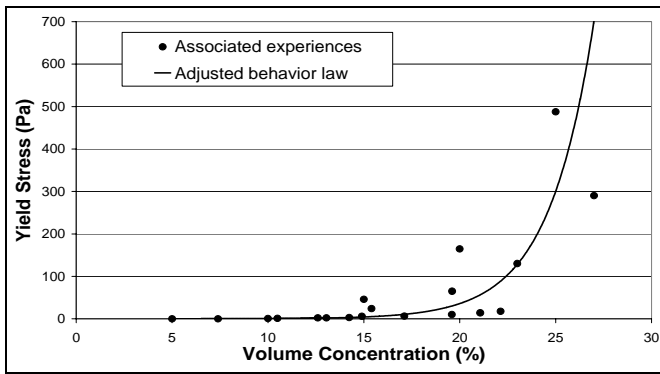


Figure 23. Behavior law of yield stress  $\tau_c$  in function of volume concentration  $C_v$  for all the data ( $\tau_c = 0,0073 e^{0,4248 C_v}$ ;  $R^2 = 0.8398$ ).

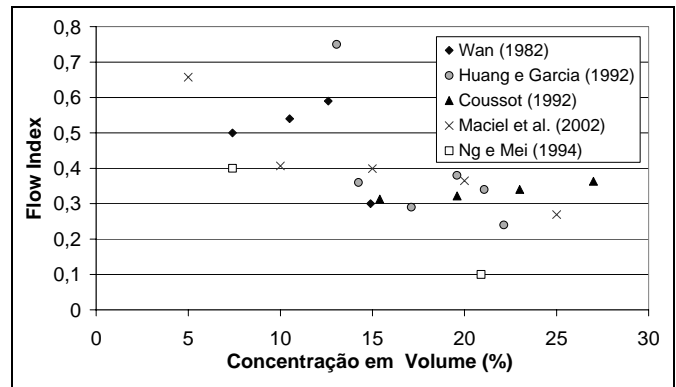


Figure 26. Progress of flow index  $n$  in function of volume concentration  $C_v$ .

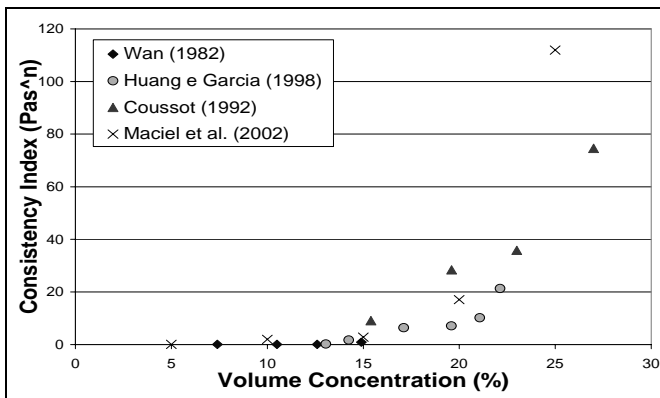


Figure 24. Progress of consistency index  $k$  ( $n$  varying between 0.1 and 0.8) in function of volume concentration  $C_v$ .

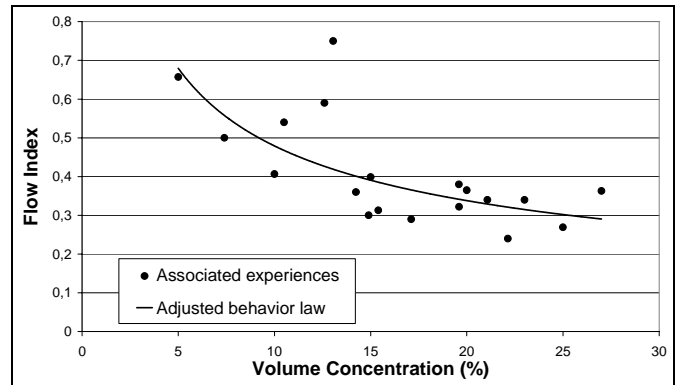


Figure 27. Behavior law of flow index  $n$  in function of volume concentration  $C_v$  for all the data ( $n = 1.529 C_v^{-0.5039}$ ,  $R^2 = 0.4967$ ).

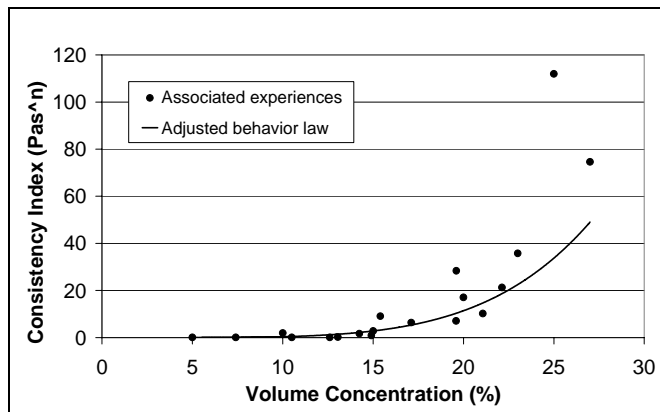


Figure 25. Behavior law of consistent index  $k$  in function of volume concentration  $C_v$  for all the data ( $k = 0,0073 e^{0,3665 C_v}$ ;  $R^2 = 0.8285$ ).

**Note:** To determine the behavior law of flow index  $n$ , the results of Ng and Mei (1994) were disregarded because they were obtained from non-kaolinitic material.

### Conclusions

From the analysis presented in the body of this work, we conclude the following:

- The mixtures tested in the rheometer (high accuracy) are well characterized with regard to the rheological progress curve;
- The kaolinitic clays tested proved to be, as expected, non-expansive, showing low thixotropic behavior and altering the basic rheological properties, such as yield stress ( $\tau_c$ ), consistency index ( $k$ ) and flow index ( $n$ ) as a function of volume concentration ( $C_v$ );
- The experimental results adjusted well to the Herschel-Bulkley rheological model, notably in determination of the yield stress and for shear rates lower than  $100 \text{ s}^{-1}$  (range observed “in the field” for mudflow phenomena). However, there was good adaptability of the Binghamian model to high shear rate.
- There was little influence of temperature and ionic potential within the conditions of the tests performed;
- In counterpart, the preparation manner of the samples proved to be a parameter to be explicitly controlled;
- The general behavior laws of the various rheological binomials were established and may, within the large

domain validity  $5\% < C_v < 25\%$ , be applied from an engineering point of view.

- In the opinion of the authors of this article, these results represent very useful and practical information for engineers and/or geophysicists involved with flowing sludge;
- In short, this article intends to rheologically characterize mixtures of water and clay comprising the so-called interstitial fluid of the actual mud mixtures found in nature, which as a rule, will provide additional elements, such as extended granulometries of sand, and when this is not the case, macroelements (coarse aggregate, stones, tufts). In fact, this is actually a dense particulate complex system. The work team at Unesp-Ilha Solteira has been involved in the study of calibration of friction laws in sloped channels, when such fluids flow on a free surface. Therefore, a platform of torrential lava with a slope variable equipped with sonic-level sensors and flow meters was built, which will enable determining and calibrating new laws of friction for channels, in addition to studying phenomena of instability (roll waves) frequently observed on the free surface of such muds.

## Acknowledgements

To FAPESP for financing the project (00/14853 – 5) and to CNPq and Fapesp for the Masters and Scientific Initiation scholarships.

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