Assessment of penetrometry technique for measuring the yield stress of muds and granular pastes

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Abstract: We discuss the possibility of using penetrometry technique for measuring the yield stress of concentrations made of grains immersed in a colloidal phase, such as concrete or muds. In that aim we used model materials made by suspending glass beads at different concentrations in a kaolin-water paste. We then show that a uniform shear stress develops along the object (plate or cylinder) beyond the entrance length. This shear stress plotted versus the object velocity exhibits a shape similar to the flow curve of the material determined from rheometry. For materials exhibiting the typical flow curve of a simple yield stress fluid, i.e. at bead concentrations smaller than 30%, the stress associated with an inflection point located at low velocities of this curve appears to correspond to the material yield stress. At larger concentrations of beads the suspensions have a more complex behaviour likely affected by its granular nature at a local scale and the possibility of migration or frictional effects, so that neither conventional rheometry nor penetrometry provide relevant data. We conclude by describing two practical penetrometry techniques for precisely measuring the yield stress of simple pastes.

1. Introduction

In industry as in nature, there is a wide range of materials that have a yield stress beyond which they flow like a liquid. The rising interest in this kind of material has led to the development of several methods to predict or measure their main rheological properties. Although problems occur in some cases, in general the most appropriate techniques rely on conventional rheometers where the material is studied in relatively well-controlled simple shear flows. In parallel, in industry various tests have been developed which aim at characterizing the mechanical properties of pasty materials, by measuring some critical strength of the material in a poorly controlled flow: squeeze test, slump test, consistometer, inclined plane flow, etc [1]. Although they do not involve viscometric flows which could readily be interpreted for determining the constitutive equation of the material these tests are particularly interesting as they make it possible to avoid various problems encountered with conventional rheometry: jamming of coarse particles in small gap, slippage or fracture with highly concentrated materials, difficulty of use in situ, cost of the apparatus, etc. As a consequence it has become challenging for rheologists to provide as straightforward as possible interpretations of data obtained from these tests.

Penetrometry is one of these techniques and one of the simplest ways to test the resistance to deformation or flow of a material. It consists in pushing a solid object, or leaving the object fall, through a bath of the material under study and measure the depth or velocity of penetration for a given force applied to the object or the force needed to reach a specific depth. This test is widely used in various industries. Depending on the field of application different measurement technologies, procedures (constant, variable or quasi-static penetration rate) and penetrometer geometries (cone, needle, plate, etc) have been used. A clarification of the link between the data from such tests and the effective rheological behavior of the materials would allow a more relevant interpretation and use of these tests.

Actually there has been only a few fundamental works aimed at understanding the link between the data obtained from this technique and the effective yield stress of the material. For long objects, i.e. with a length along the direction of penetration much larger than in the perpendicular directions, the usual approach consists in assuming that the origin of the force resisting motion is the viscous shear stress along the object surface in the direction of motion. Under these conditions one can estimate the yield stress from the critical force allowing displacement divided by the surface of the object along the motion direction and in contact with the fluid, possibly with a correction accounting for edge effects [2-4]. Another technique consists in displacing an object at a given velocity through the fluid and assuming that when the velocity tends to zero the drag force approaches the critical force associated with a shear stress equal to the yield stress along the object [5]. A last technique focuses on the withdrawing of the object initially immersed in the fluid. The force versus time response is recorded while the object is moved vertically at constant speed through the sample. It is assumed that the fluid yields at the end of the linear elastic deformation which can be distinguished from the force curve [6-7] Note that it was suggested that wall slip may occur along smooth surfaces displaced in a yield stress material [6, 8]. It thus appeared important to minimize this effect by using rough surfaces [4] or particular object shapes ("slotted plate") [7, 9].

Recently the force versus depth variations during the progressive penetration of a plate or a cylinder in a bath of simple yield stress fluids with negligible thixotropic character, namely Carbopol gels, concentrated emulsions and foams, was studied [10]. Three regimes could be distinguished: elastic deformation, penetration (partially immersed object), and displacement through the fluid (fully immersed object). A detailed analysis of the force as a function of the depth curves made it possible to show that in the partially immersed regime the force is the sum of the critical force before penetration and a term associated with a uniform shear stress along the main plate surface, which is independent on the object geometry (plate dimensions and cylinder radius). This understanding can be used to precisely determine the yield stress as the critical shear stress along the plate at vanishing velocities. For the simple model materials used an excellent agreement was found between the measured yield stress values from this technique and from rheometry.

These results have been obtained with somewhat ideal fluids. Here we wish to explore the possibility to use these techniques for measuring the yield stress of more complex materials such as cement pastes, fresh concrete, natural muds, etc. In order to cope with this question one approach could consist in testing a wide series of such materials. However, since we will never be able to test all types of materials it is likely that such an approach would not provide a general answer and anyway would not allow us to understand the origin of possible problems. We finally thought more useful to focus on model materials with a microstructure reproducing some important trends of these materials. In that aim it is necessary to review the physical characteristics we wish to reproduce.

First of all we are dealing with yield stress materials. In addition some of them are thixotropic. In that case it becomes difficult to define a single yield stress since the apparent yielding occurs at a stress level increasing with the time of rest or decreases with the preshear intensity. So we will leave apart this aspect. From a microstructural point of view both muddy and cementitious materials are made of grains of size ranging from a few tenths of microns to a few centimetres and suspended in a mixture of colloidal particles in water (plus additives in the case of concrete). In order to reproduce this multi-scale granular structure we used a matrix made of clay-water suspension in which the particles, although they are in the colloidal size, develop relatively low colloidal interactions. In this matrix we added glass beads at different concentrations. With such mixtures we finally have model systems of a wide range of industrial or natural suspensions, including a larger number of cementitious or muddy materials.

We start by presenting the materials, the equipments and the procedures. We review the rheological behavior of our suspensions in steady state. Then we analyze the different steps (regimes) of penetration of a plate through such materials. We show that a thorough analysis of the data makes it possible to measure directly the local value of the shear stress along a plate or a cylinder. Finally we compare the yield stress values obtained either by conventional rheometry or by penetrometry.

2. Materials and methods

2.1. Materials

The materials were prepared by mixing together different amounts of kaolin (*Speswhite* China Clay), beads (diameter in the range 200-400 μm) and water. For each mixture we took fixed relative amounts of kaolin and water and varied the amount of beads in order to obtain the desired concentration (see the respective values in Table 1). The final mixture may be seen as a fixed kaolin-water matrix in which different volume fractions of beads are suspended. Such material type has already been used as model fluids for laboratory studies of mudflow hydraulics [11-12]. The impact of the physico-chemical properties of this material on its mechanical behaviour has been studied for example in [13].

For a given mixture, the required amounts of kaolin, beads and water were put together in a beaker and the mixture was thoroughly mixed to make it homogeneous. A strong mixing was necessary to break the lumps sometimes formed by glass beads and kaolin in the mixture. The samples were stored in closed containers in order to avoid evaporation. After some time of storage sedimentation effect appears (some pure water appears at the free surface). Therefore, before any use, the samples were mixed again thoroughly.

Water	Kaolin	Glass beads
77.5	22.5	0
69.75	20.25	10
62	18	20
54.25	15.75	30
46.5	13.5	40
38.75	11.25	50
31	9	60
20.67	6	65

Table 1: Volume fraction (%) of each component type for the different mixtures.

2.2. Rheometry

Rheological tests were performed with a Bohlin rheometer (controlled shear stress) equipped with a vane geometry (inner diameter: 25mm; outer diameter: 36mm). Stress sweep tests were imposed, wherein the shear rate was increased logarithmically in time at a constant rate and then decreased logarithmically in time over a total time of one minute. The stress was followed as a function of the shear rate. Note that no preshear was applied on the material.

This is justified by the fact that when a shear rate is maintained for a time larger than several tenth of seconds the stress significantly and irreversibly decreases likely due to some artefacts such as an evolution of the free surface of the sample, shear localization or segregation effects. Despite various tests with this material type it did not appear possible to find a procedure allowing to overcome this effect.

2.3 Penetration test

2.3.1 Equipment

The instrument used for penetration test is composed of a vertical plate (or cylinder) linked to the plate of a dual-column testing system (Instron model 3365) which controls the vertical position with a resolution of 0.118 μm. The apparatus is equipped with a 10N static load gauge able to measure the force within $\pm 10^{-6}$ of the maximum value. The vertical plate is moved downwards at a constant speed so as to progressively penetrate the fluid bath and the resulting force applied onto it is measured. The speed can be varied from 0.001mm/s to 17 mm/s.

For all tests we covered the main solid surfaces of the plate with a layer of glass beads of diameter 1 mm in order to avoid wall slip effects. Thus the effective roughness was of the order of a millimeter, a dimension much larger than the elements of the materials used in this study.

Most tests were carried out in a 5 l cylindrical container of radius 9 cm. We have shown in our recent work [10] that the container size does not have any impact on the measurements as long as the distance between the plate and the container sides is larger than the liquid layer thickness along the moving plate, which is typically of the order of one centimeter. Moreover our technique for estimating the shear stress was shown to be independent of the plate geometry (see below). Here, for the plate geometry we used a plate with the following characteristics: $E = 0.6\text{cm}, H = 15\text{cm}, L = 7\text{cm}$, with E the thickness, L the width and H the height, and for the cylinders we used diameters of 0.84 cm and 1.56 cm.

2.3.2 Procedure

The material was poured in the container and left at rest for some time in order to ensure residual stress relaxation. The plate was first coated with material then the material in excess of the roughness was removed (cleaning operation) before immersing the plate for the first test; afterwards it was simply cleaned in the same way between two successive tests. All measurements were performed at room temperature (23°C).

Before immersing the plate the force was reset to zero. Then the plate was lowered at a constant speed (V). The force (F) as a function of the apparent depth of immersion, i.e. the distance h of downwards displacement of the plate from the initial contact with the sample free surface, was recorded from the first contact of the plate with the fluid until its complete immersion.

3. Experimental results and discussion

3.1 Rheological behavior

The flow curves obtained from the sweep tests exhibit a part with a steep slope at low shear rate in the increasing stress portion (see Figure 1). **In this part the material is in general still**

in its solid regime [1] because the total deformation imposed from the beginning of the test is smaller than the critical deformation associated with the solid-liquid transition. In this (solid) regime the shear stress is roughly proportional to the total deformation, which increases with time at a rate which depends on the way the imposed shear rate is increased. As a consequence this part of the curve does not reflect the behavior of the material in its liquid regime and its exact position in the rheogram depends on the rate of increase of the shear rate.

A maximum in the curve is then reached for some critical shear stress then we have some decrease followed by an increase of the stress towards higher shear rates (see Figure 1). We generally consider that for this local maximum (overshoot) of the stress the liquid regime has been reached. For a decreasing shear rate the curve starts to follow the increasing curve at high shear rates but departs from the increasing curve around the overshoot region (see Figure 1). The material is still in its liquid regime but here has been some evolution of the structure leading to some decrease of the apparent viscosity.

With a simple yield stress fluid in the decreasing curve we have a progressive decrease of the slope followed by a plateau towards low shear rates. With our suspensions we have the same shape beyond some critical shear rate, with a tendency of the curve to give a plateau (at a level associated with the dotted line in Figure 1), but instead of effectively tending towards a curve of slope equal to zero at very low shear rates, at the end of the decreasing part there is a new significant decrease of the stress towards lower shear rates (see inset of Figure 1). Finally we have an effect that we will call a *semi-plateau*. We attribute this feature to the same kind of artefact as those listed above (cf. Section 2.2) and concluded that the effective (dynamic) yield stress of the material can be estimated from the level of the stress plateau before this final decrease, say generally at shear rates around 5.10-2 s-1. More precisely we estimated the yield stress by fitting a Herschel-Bulkley model ($\tau > \tau_c \Rightarrow \tau = \tau_c + k\dot{\gamma}^n$, in which τ_c is the yield stress, and k and n are other material parameters) to the decreasing portion stress located above the dotted line.

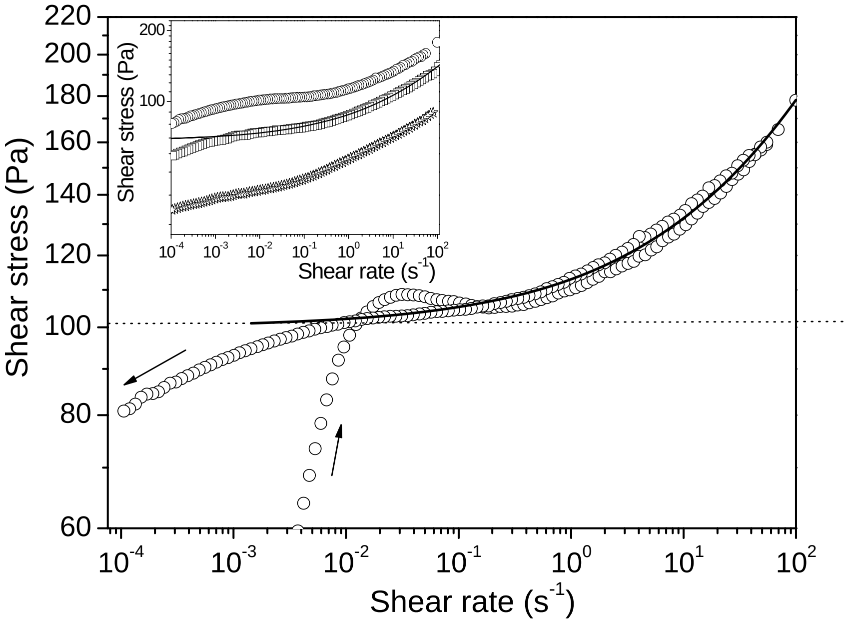

Figure 1: Typical flow curve obtained from shear rate sweep (increasing then decreasing) for the kaolin suspension + beads (concentration: 30%). The continuous lines (in the main figure and in the inset) are a Herschel-Bulkley model fitted to the data. The horizontal dotted line is situated at the level of the yield stress found from this fitting. Inset: Decreasing curves obtained at different concentrations of beads: (from bottom to top) 0%, 20%, 30%.

For low bead concentration, say smaller than 30%, the decreasing curve follows almost exactly the part of the increasing curve beyond the semi-plateau. In that case it is natural to consider that a steady-state flow in the liquid regime has been reached and we can use the decreasing curve as the effective flow curve of the material. For larger bead concentration there is some significant hysteresis in the stress sweep (an effect that we start to see at 30% (see Figure 1)), the increasing curve reaches a level higher than the semi-stress plateau during the decreasing part (see Figure 2). Such an effect is usually observed with thixotropic suspensions [1]. However, here the beads do not develop any colloidal interaction at the origin of usual thixotropy. As a consequence such an effect is likely due to some kind of jamming or interactions between the elements: the kaolin particles plus the beads might form some kind of network of grains in contact which would initially have to break before flowing, thus leading to a stress overshoot. **This type of interaction has been observed in suspensions on non-colloidal particles [14].** We will not try to analyze further this difficult problem as it is out of the scope of this work. Finally we can distinguish two types of yield stress: that associated with the semi-stress plateau (exactly just before the last decreasing part) in the decreasing curve (dynamic yield stress) and that associated with the stress overshoot in the increasing curve (static yield stress).

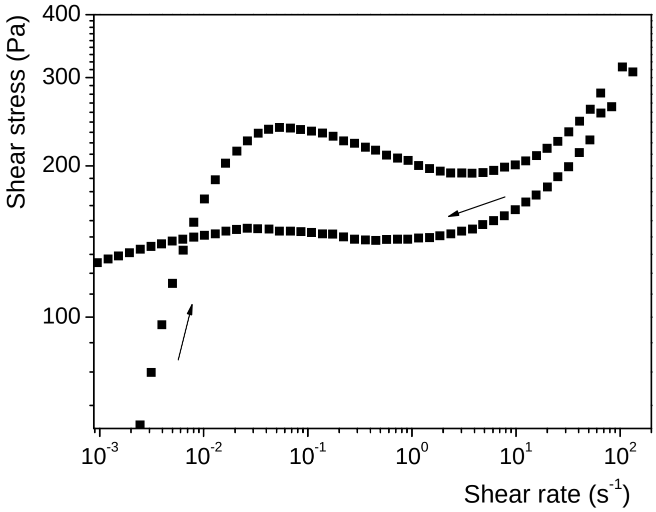

Figure 2: Flow curve obtained from shear rate sweep (increasing then decreasing) for the kaolin suspension + beads at a concentration of 40%.

In Figure 3 we compare the yield stress values obtained with different concentrations of beads. There is obviously a good agreement between the two types of yield stresses at bead concentrations equal or smaller than 30%, due to the absence of significant overshoot in the sweep test in that case. On the contrary we start to get a significant discrepancy at higher bead concentrations but there is no clear trend appearing in that case between the two types of values.

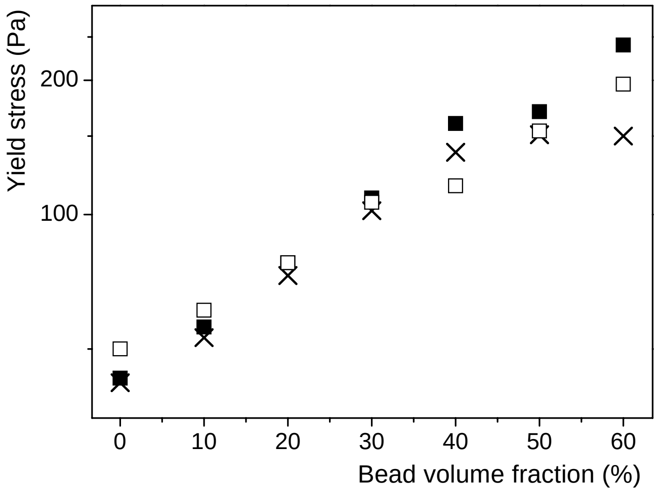

Figure 3: Yield stress of kaolin suspension + beads as a function of bead concentration according to different measurement technique: (crosses) plateau in decreasing flow curve (fitted with a Herschel-Bulkley model), (filled squares) overshoot in increasing flow curve (see text), (open squares) plateau in flow curve obtained from penetration tests (see text).

3.2 Penetration tests

3.2.1 Local stress determination

A typical force versus depth curve is shown in Figure 4. Here the recorded depth h is the distance between the current plate tip and the initial (horizontal) free surface of the fluid. We can distinguish two regimes: (I) From the first contact to the effective penetration; (II) Partially immersed plate. Since in regimes I and II the free surface of the fluid deforms before and after penetration the flow may be complex around the tip of the plate and we cannot readily interpret the force versus height curves. The current force may indeed include both some component related to the elastic deformation of the sample and some viscous force associated with the flow of the material in its liquid regime around the plate. A further insight is provided by the slope of this curve, i.e. $\mathrm{d}F/\mathrm{d}h$, which expresses the force increment needed to move an increment farther the plate against or through the material.

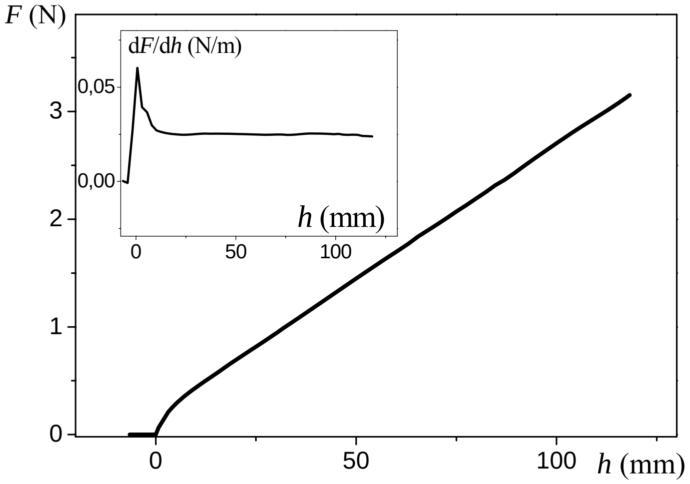

Figure 4: Penetration of a plate through a kaolin suspension (Mixture at 30% beads, plate velocity: 12mms^{-1}): typical aspect of the force vs depth of penetration. The inset shows the corresponding derivative of the force with the depth.

A typical curve of this derivative is shown in the inset of Figure 4: $\mathrm{d}F/\mathrm{d}h$ starts by rapidly increasing, an effect which might be due to the fact that, when it has not yet penetrated the fluid, the plate motion induces larger and larger deformations in the material which is still in its solid (elastic) regime. Then, when the plate has started to penetrate, it should move through the material with a liquid region around it. It seems reasonable to consider that the transition between these two regimes, namely the beginning of penetration, occurs around the peak of the $\mathrm{d}F/\mathrm{d}h$ curve. After the beginning of the plate penetration $\mathrm{d}F/\mathrm{d}h$ rapidly decreases and finally reaches a constant value (see inset of Figure 4) (note that we have again no particular explanation for the decrease before reaching this level). During the latter stage there is no further storage or loss of elasticity in the upper regions of the fluid, as proved by our observations of the free surface of the sample which is no more deformed. As a consequence the linear increase of the force suggests that at each step, say for a small increment of immersed length, the resulting additional amount of force is due to the flow in the liquid regime of the material along this increment of length just below the free surface, while the rest of the liquid flow and elastic deformation around the rest of the plate (below this short distance) are unchanged. The force variation in this regime can thus be used to compute the stationary stress associated with a flow in the liquid regime along the plate. Note that the linear increase of the force occurs only beyond some distance of penetration, which may be considered as the entrance length.

We can express the recorded force as the sum of different forces exerted by the material on the plate during its displacement. Those terms are the fluid resistance to deformation (or flow) along the plate, F_v, the buoyancy on the plate, B, and the capillary force, acting only when the plate is partially immersed (the capillary force on the wire being negligible in that case). The expression for the buoyancy is $B = \rho g S h$, in which ρ is the fluid density, g the gravity, and S the section area of the plate. Due to the roughness of the plate S is not precisely known. In order to determine its value we carried out independent force measurements with a plate well immersed in a Newtonian liquid at rest. In that case F_v and F_w are equal to zero and capillary effects are negligible (for sufficiently large h) so that $F = B$.

We have seen that by considering the derivative of F with respect to h we a priori get rid of the force terms due to surface tension and elastic deformation in the stationary regime. In that case the force increment is the sum of a viscous term plus the buoyancy force. We deduce that in this regime, for a given velocity, the shear stress associated with the liquid regime along the plate is constant and uniform and may be found from:

$$\tau = \frac{1}{P}\left[\frac{\mathrm{d}F}{\mathrm{d}h} - \rho g S\right] \tag{1}$$

in which here P is the periphery of the plate $P = 2(L+E)$.

It is worth emphasizing that according to the above approach *a unique value of the stress along the plate should be found whatever the plate geometry*. This is so because it was found from an analysis of the flow characteristics beyond the entrance length, where the flow is no more affected by the shape of the plate tip.

As a consequence we also applied this technique with a cylinder instead of a plate. The observed trends are qualitatively similar. For the analysis of the data we can use equation (1) with now $P = 2\pi R$ and S takes the value determined from separate tests as described before.

3.2.2 Flow curve (shear stress versus plate velocity)

The shear stress, as it is deduced from the procedure described in the previous Section, is plotted for different velocities and different bead concentrations in Figure 5. Note that in these tests we carried out measurements at velocity values taken at random in our range of test, i.e. we avoided imposing successively increasing or decreasing velocities. With such a procedure we intended to minimize the impact of some artefacts inducing effects varying monotonously with time or velocity. We also tested the reproducibility of the data obtained in such a way and it appeared quite good. Finally we estimate that the uncertainty on the flow curve data presented in Figure 5 is about 15%.

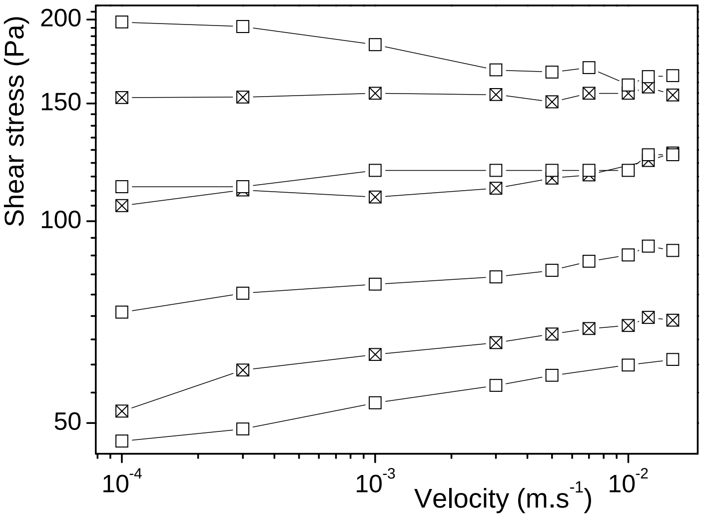

Figure 5: Penetration of a plate through kaolin suspension + beads: uniform shear stress along the plate as a function of the plate velocity for different bead concentrations: (from bottom to top and alternatively squares and cross squares) 0, 10, 20, 30, 40, 50, 60%.

We observe that the flow curves (force versus velocity) determined from plate penetration exhibit an unexpected trend as the concentration increases (see Figure 5): they do not have a single shape. At low or moderate concentration, say up to 30%, they have a shape similar to **the classical shape of the flow curve for a simple yield stress fluid, including a semi-plateau (see above) at low shear rate and a progressive increase of the stress with shear rate. In Figure 6 we have a zoomed representation of a typical example of these curves on which we can best distinguish this semi-plateau at low velocities. I**n that case, a further comparison of the plate penetration flow curve with the effective flow curve of the material shows that below or equal to 30%, they are almost strictly similar and may be superimposed by a simple horizontal translation of one of them (see Figure 6). This result means that all occurs as if the shear rate along the plate moving through the fluid was simply equal to the plate velocity divided by a characteristic length (λ) independent of the velocity. A similar result was obtained with simpler yield stress fluids (gels, emulsions, foams) [10] and internal measurements in transparent fluid [15] indeed showed that the liquid layer along the plate kept a constant thickness while the velocity profiles in this layer were similar when rescaled by the plate velocity. Here it is worth noticing that the characteristic length λ is similar for all kaolin suspensions at bead concentration below or equal to 30%.

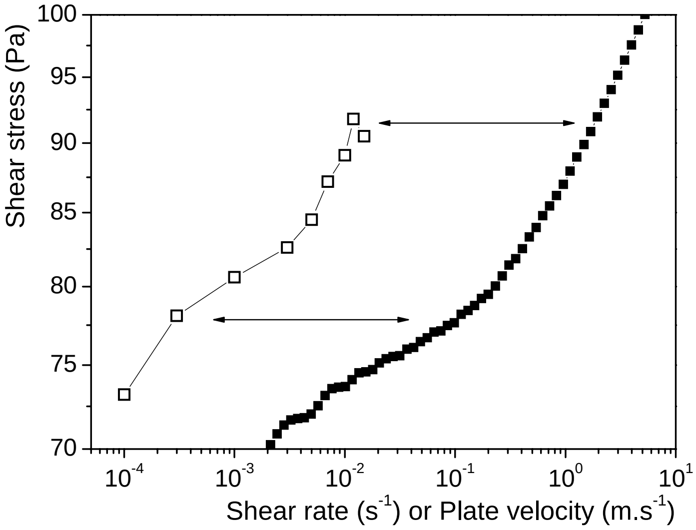

Figure 6: Flow curves obtained by two different techniques for a kaolin suspension + beads (20%): (filled squares) shear stress vs shear rate in a decreasing ramp of shear rate (rheometer), (open squares) uniform shear stress vs velocity in penetrometry.

At concentrations larger than 30% the flow curves obtained from plate penetration start to exhibit a shape differing from the usual simple yield stress fluid flow curve: as the bead concentration increases the curve becomes horizontal over a wider range of shear rates then starts to decrease when the shear rate increases at low values (see Figure 5). Although in the absence of internal measurements it is difficult to understand precisely the effects at the origin of such trends, these results nevertheless seem qualitatively consistent with our observations in conventional rheometry. Indeed precisely in the same range of concentrations, say at values larger than 30%, we observed some kind of instability at low shear rates, leading to a wide plateau (decreasing ramp) or a decreasing apparent flow curve (in the increasing ramp) (see Figure 2). It is likely that the effects (frictional network) suspected to be at the origin of the trends in conventional rheometry also develop in a more or less similar way during plate penetration.

We conclude that we can only get straightforward relevant measurements of the flow curve aspect at low or moderate bead concentrations, when migration or frictional effects are absent. Qualitatively similar results were obtained from experiments with cylinders of different sizes, i.e. features presented in Figure 5 and 6 were similar. As a consequence we do not present in detail these data but we will use the corresponding values of yield stress for the following discussion.

3.2.3 Yield stress measurement from penetrometry

Let us focus on measurements in the bead concentration range for which the rheometrical flow curve effectively corresponds to that of a simple yield stress fluid, i.e. at concentrations below or equal to 30%. We remark that the flow curve from plate penetration also exhibits a curious decrease of the stress at very low shear rates **(see Figure 6). As in conventional rheometry we assume that this is due to some artefact such as particle migration or frictional effects. We consider that the yield stress of the material may be estimated at the inflexion point located toward the low velocities of this curve, that we described**

above as the end of a semi-plateau similar to that observed for flow curves in conventional rheometry. We did the same with the different geometries (plate and cylinders) and first compared the obtained values for the plate with the different ways for determining the yield stress in conventional rheometry (see Figure 3). It clearly appears that our penetrometry technique makes it possible to get relevant data for the yield stress for our different suspensions at bead concentrations smaller than 30%: the yield stress deduced from penetration test with the plate provide values which are very close to those obtained from rheometry (see Figure 3). Then we can compare the values obtained from penetrometry with cylinders: it appears that they are very close to those obtained from measurements with plate (see Figure 7).

In Figure 7 we also added the yield stress values deduced from measurements on the kaolin suspension + beads (40%). The different values significantly fluctuate around some value which strongly differs from the value observed from conventional rheometry. This means that the technique is inappropriate for kaolin suspensions with high concentrations of beads.

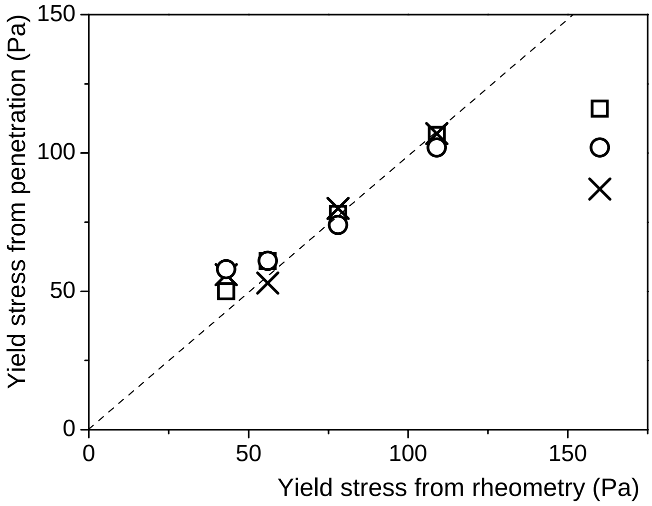

Figure 7: Yield stress of kaolin suspension + beads at different concentrations: (from left to right) 0, 10, 20, 30, 40%; and determined from penetrometry either with plate (squares), small cylinder (circles) or large cylinder (crosses), as a function of the yield stress value obtained from rheometry **(semi-stress plateau).** The dotted line illustrates what would be a perfect agreement.

We also attempted to measure the yield stress from relaxation tests in penetrometry. In this technique we impose a displacement of the plate at a given velocity, then suddenly stop the motion and follow the force versus time while the plate is fixed. It was shown for gels and emulsions that this technique provides values for the yield stress in very good agreement with the rheometrical values. However with kaolin suspensions the situation is more complex. The force as a function of time appears to continuously decrease over long time (see inset of Figure 8), i.e. it does not reach a clear plateau from which the yield stress can be directly deduced. Such an effect has likely the same origin as that of the stress decrease effect observed at low shear rates in rheometry or at low velocities for plate displacement. Moreover if now we arbitrarily consider that the yield stress is reached after a given time and compare the values obtained for different previous plate velocities we see that the apparent yield stress decreases with velocity (see Figure 8). It is likely that some migration effects again occur during such a test: the kaolin suspension does not remain perfectly homogeneous along the plate. Anyway we can conclude that these relaxation tests are inappropriate with such materials.

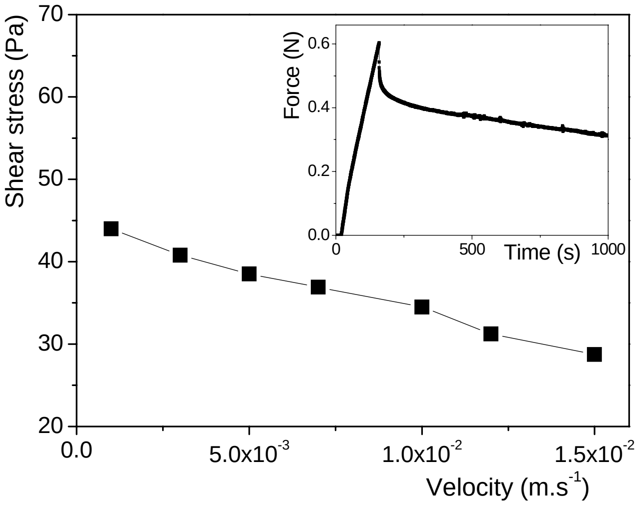

Figure 8: Residual shear stress measured after a given time of relaxation (500s) for a kaolin suspension (mixture at 20% beads) during plate relaxation test, as a function of the plate velocity before stoppage. The inset shows a typical force vs time curve during relaxation (plate velocity: 0.1 mm/s).

4. Conclusion and application

We have shown that for suspensions containing a moderate concentration of coarse grains the yield stress can be determined quite precisely from penetrometry. More generally, if we also take into account previous results on simpler fluids [10], we can now conclude that for any material which exhibits a behavior close to that of a simple yield stress fluid, i.e. without thixotropy or granular effects, this penetrometry technique is relevant for determining the material yield stress. **On the contrary, for materials exhibiting a more complex behavior, in particular when this behavior is affected by a significant concentration of coarse particles in suspension in a finer paste, the penetrometry technique does not provide a measure of the yield stress in a straightforward way, but this might also be the case for any other technique.**

Remark that the technique developed here relies on the observation of the force curve during the penetration through the fluid of a long cylindrical object along its main axis. In practice it may appear preferable not to have to compute the slope of the force versus depth curve. We can suggest simpler procedures which could even be used in the field or in situ.

Controlled velocity penetrometry

In that case one needs to have a specific apparatus making it possible to impose a given, as low as possible, velocity of penetration (say of the order of 0.1mm/s) and measure the force needed at two different times during penetration. The first time (computed from the beginning of penetration) must be such that the object has penetrated over a distance larger than say about ten times the characteristic width of the object. The second time can for example be twice the first one. Then the yield stress is deduced from the following equation, which directly derives from (1):

$$\tau = \frac{1}{P}\left[\frac{F_2 - F_1}{\mathrm{H}} - \rho g S\right] \qquad (2)$$

In which F_1 and F_2 are the two force values and H the length of penetration between the two times. In this approach the buoyancy stress ($\rho g S / P$) can be determined from a separate test with a bath of simple liquid of known density.

Controlled force penetrometry

If we apply a given force to a long object it will penetrate through the paste until the force component due to the stress applied along the object surface is equal to this force. At that time the object stops moving. In practice it may be difficult to appreciate the effective stoppage of the object since for a simple yield stress fluid this velocity in theory tends to zero progressively and a perfect stoppage is attained after an infinite time. In fact the maximum length of penetration is generally closely approached after a relatively short time. Anyway, with concentrated suspensions such a kaolin pastes we know that artefacts tend to occur at low velocities or for long tests. As a consequence it is critical not to try to wait for a strictly complete stoppage. For example the maximum time for measurement must be smaller than the time at which the velocity is of the order of 0.1mm/s (since at smaller velocities artefacts were observed), which means that the object moves over less than 2mm in 20s.

A simple test consists in measuring the (approximate) depth of complete penetration for two different forces (see Figure 9). The additional force between the two tests is strictly used for balancing the additional buoyancy force and the shearing the material at a stress almost equal to the yield stress along the object over the additional length of penetration (*H*). It is important to have an initial distance of penetration (for the first force) of the order or larger than ten times the characteristic width of the object, and an additional distance (for the second force) of the same order. In essence such a test is similar to that of Uhlherr et al. [4] in that it uses a controlled force and measure the stoppage conditions. The fundamental difference is that, with the help of measurements for two force levels and a substraction, we remove the force contribution due to the (unknown but constant) deformation of the material at the front of the object, and that due to deformation of the free surface of the sample. Finally one can simply estimate the yield stress from equation (2). We know from our experimental data that such an approach is relevant for estimating the yield stress of a simple yield stress fluid material.

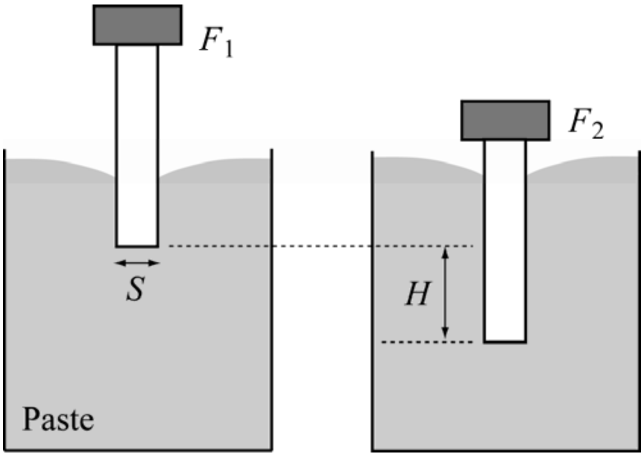

Figure 9: Scheme of principle of the simple penetrometry test proposed (see text) under controlled force for measuring the yield stress. One measures the displacement (*H*) of a long object, resulting from the application of a force F_2 after having imposed a force F_1.

Note that in practice a cone is often used instead of a long object such as a plate or a cylinder, likely because this is a more stable geometry. However, with such a geometry the above approach cannot be used. In fact we can even suspect that the entrance length is never overcome with such a geometry. We are never in a well-developed liquid regime around the object since the deformation and flow around the object tip continuously evolves as the object penetrates farther. Such a geometry can be interesting to make comparative measurements but cannot be used to get a relevant estimate of the yield stress.

7. References

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