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Practical determination of the rheological behavior of pasty biosolids

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Abstract

In this paper, we demonstrate that the rheological behavior of pasty sewage sludges, regardless of origin, treatment or composition, follows a Herschel-Bulkley model. The yield stress and solid volume fraction are found to be the only two distinctive rheological characteristics of these materials. By scaling the shear rate and the shear stress with two parameters depending only on the yield stress and the solid fraction, the flow curves of 48 pasty sludges all fall along a unique dimensionless master curve. This result may be used in practice to determine, from simple, independent measurements, the rheological behavior of any pasty sludge: the yield stress can be measured with the help of the 'slump test' and the solid concentration determined from the organic and mineral matter contents. The results obtained with this technique are in very good agreement with those obtained by direct rheometry.

Keywords: Flow curve; Pasty sewage sludge; Slump test; Solid volume fraction

1. Introduction

In order to improve wastewater treatment, rheological measurements provide useful information to quantify mechanical sludge properties, such as thickening ability (Frost and Owens, 1982). Nevertheless, rheometric tests often need sophisticated apparatus and require careful experimental procedures, which are not suitable for in situ measurements inside a treatment plant. This paper therefore presents a simple method for determining the rheological behavior of pasty sewage sludge without a rheometer.

Sewage sludge is often seen as a complex mixture and its rheological behavior considered to be highly dependent on the implemented treatments (Lotito et al., 1997; Battistoni, 1997). As a consequence, no general characteristics have been observed. A review of published results shows that these materials appeared to be non-Newtonian, possibly thixotropic or yielding fluids (Campbell and Crescuolo, 1982; Colin et al., 1976). On the basis of repetitive experimental procedures and statistical analysis, the steady state behavior was represented with the help of either a pseudo-plastic model (Hatfield, 1938; Behn, 1962; Valioulis, 1980; Battistoni, 1997), a Bingham model (Babbit, 1939; Geinopolos and Katz, 1964; Frost and Owens, 1982; Spinosa et al., 1989), or a Herschel-Bulkley model (Johnson, 1961; Mulbarger et al., 1981; Monteiro, 1997). These models may all be represented by the following model (Herschel-Bulkley):

$$\tau = \tau_c + K\dot{\gamma}^n \qquad 0 \le \tau_c \tag{1}$$

where τ and $\dot{\gamma}$ represent, respectively, the shear stress [Pa] and the shear rate [s^{-1}], and K and n are two material parameters. The different models are recovered by the following values of parameters: pseudo-plastic ($\tau_c = 0$) and Bingham plastic ($n = 1$).

To explain the different behaviors, the relationship between rheological parameters and sludge composition has been examined and in all the cases, the total solid content appeared to be the main factor governing the behavior (Battistoni, 1997; Battistoni et al., 1993, 2000; Slatter, 1997). Nevertheless, according to Lotito et al. (1997), a single factor did not appear sufficient to explain the variations of at least two independent parameters (yield stress, consistency index (and power-law index)). Many authors have focused on tests often used in water treatment, such as capillarity suction time or hydraulic

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retention time (Battistoni et al., 2000), but no physical interpretation was made. The role of the organic composition was evidenced by the work of Monteiro (1997), who observed the qualitative changes induced by controlled anaerobic digestion. He showed a fluidification of the material, which could be attributed to a variation of surface charge, depending on the main compounds of the organic phase (Forster, 1982, 1983, 2001).

Sludges of almost any composition may be subject to storage, landfilling, or agricultural spreading, as long as the regulations are respected. Then, to evaluate mechanical properties, relevant rheological characteristics must be easily measured and related to general parameters, common to any sludge. Following this idea, we here consider sludge in its simplest definition: the residue of wastewater treatment, composed of water, organic and mineral matter. On the basis of rheometrical tests we first underline the existence of general, qualitative characteristics of the behavior of pasty sewage sludges. Then we show that solid concentration is not a sufficient parameter to predict the complete behavior but it can be related to viscous dissipations. Finally, we propose a simple method to estimate the parameters of the flow curve of any given pasty sludge without using a rheometer.

2. General behavior of pasty sewage sludge

In this section we intentionally do not consider the detailed composition of the sludges. Samples used here were obtained randomly after the dewatering step in the storage area at the end of the treatment in various wastewater treatment plants located in Allier and Nièvre, two French rural countries, and in the US state of Delaware. Materials were stored at room temperature.

Rheometric tests were performed with a controlled stress rheometer, Paar Physica MC1 +, equipped with either parallel plates or large gap coaxial cylinders geometries, both with rough surfaces to avoid wall slip (Tabuteau et al., 2004), which significantly affects the results at low shear rates. Materials were systematically presheared at 300 rpm during 2 min then stayed at rest during 20 min. We first carried out creep tests by imposing successively increasing stresses, relaxing during 10 s between two levels, during a fixed time, then the stress was progressively decreased by similar steps. The total rotation angle, φ, was recorded at the end of each step. We used this rotation angle instead of the deformation because we suspect that the latter is not homogeneous in the gap (Baudez and Coussot, 2004). The data reported concern the total rotation angle after each step. For the flow curves we used the total rotation reached after each step during the decreasing ramp of stress, which a priori ensures that steady state flow conditions were reached.

In all the cases, for stresses smaller than a first critical value, τ_1, we obtain a linear relationship between φ and τ

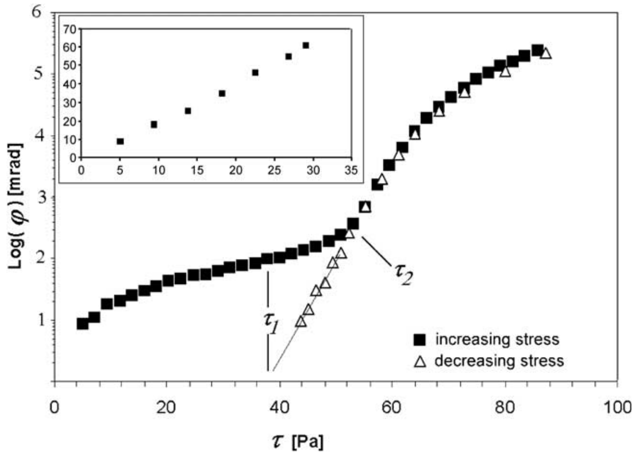

Fig. 1. Total deformation after 60 s at each step for successively increasing shear stresses (relaxing for 10 sec between steps) and for successively decreasing stresses without relaxation between steps (here, $\tau_1 = 36$ Pa and $\tau_2 = 52$ Pa, approx.). The inset graph enlarges the total deformation below τ_1. With such a representation, with the same axis, the linear relationship is clearer.

for the increasing ramp while no deformation is recorded for the decreasing ramp (Fig. 1). For stresses larger than a second critical value, τ_2, the two (increasing-decreasing) curves are superimposed which probably means that viscous dissipations prevail (Baudez and Coussot, 2001). The behavior of pasty sewage sludges can be summarized as follow (Baudez, 2001):

$$\begin{array}{ll} \tau < \tau_1 & \text{linear viscoelastic} \\ \tau_1 < \tau < \tau_2 & \text{non-linear viscoelastic} \\ \tau_2 < \tau & \text{purely viscous} \end{array} \quad (2)$$

The steady state flow characteristics can be inferred from the decreasing stress-rotation angle curve. The rotation velocity is deduced for each stress value by calculating the slope of the curve $\varphi = f(t)$ at the end of each step. The flow curve can then be determined taking into account the heterogeneous shear with the large gap coaxial cylinders (Piau, 1979). Typical results obtained in steady state are presented in Fig. 2. According to the considered stress interval, the experimental points are best fitted by a Herschel-Bulkley model in the range $\tau \geq \tau_1$ but if we consider only the range $\tau \geq \tau_2$ an Ostwald model is sufficient.

Thus, with the same material, the mathematical model representing the rheological behavior depends on the range of experimental data considered. Various models may be used in practice but only in the range of shear rates or stress in which they have been determined.

In the following, we will systematically use the Herschel-Bulkley model to represent the experimental behavior, by considering that the yield stress is $\tau_c = \tau_1$. The value of τ_1 is determined from creep tests as above described while the two other parameters (K and n) are determined by fitting the model to the decreasing stress flow

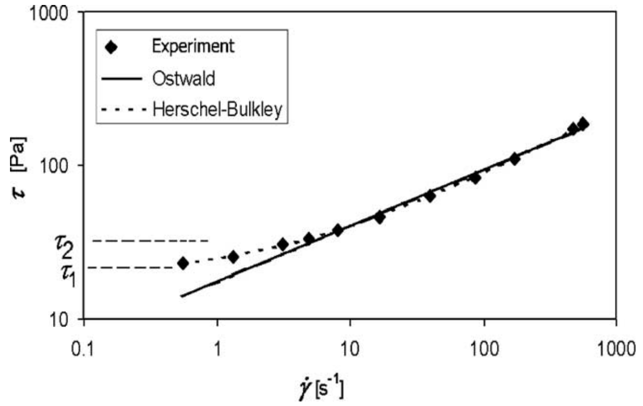

Fig. 2. Typical flow curve of a pasty sewage sludge. Data correspond to steady state values under controlled stresses. Two models have been fitted to the data: a Herschel–Bulkley model (here with $\tau_1 = 17$ Pa; $K = 7.6$ Pa s^n; $n = 0.49$), and a Ostwald model (here with $K = 17.3$ Pa s^n; $n = 0.37$).

curve. Sludges without yield stress ($\dot{\gamma} = 0$; $\tau = 0$) will not be analyzed.

3. Influence of water content

Pasty sewage sludge is a material with a large amount of water, even after the dewatering step (higher than 80%). It is thus appropriate, as established in much previous research, to analyze its behavior in relationship with the solid, volume concentration. This was determined by drying the sludge at 60 °C during 72 h (ASAE Standards, 1999). The respective amounts of organic and mineral matter were distinguished by burning the residue at 550 °C during 12 h. Drying the sludge following this procedure is more convenient than at 105 °C during 24 h in order to avoid volatilization of volatile fatty acids (Derikx et al., 1994; Baudez, 2001). Moreover, at 60 °C, bound water is not removed (Vesilind, 1994) and so can be considered as a solid phase compound. The solid, volume fraction was then calculated from the following equation:

$$\Phi_v = \frac{\%\mathrm{MO}}{d_{\mathrm{MO}}} + \frac{\%\mathrm{MM}}{d_{\mathrm{MM}}} \quad (3)$$

where %MO and %MM, respectively, represent the organic and mineral contents in mass. The specific masses of organic (d_{MO}) and mineral (d_{MM}) matter were, respectively, assumed to be equal to 1.0 and 2.6 (Desjardins and Roy, 1993).

Fig. 3 illustrates the absence of direct link between steady state behavior and solid volume fraction: the most concentrated sludge is not necessarily the most viscous. Nevertheless, one cannot ignore the role of water in sludge rheology: before and after the dewatering step, the sludge has not the same consistence, and its behavior has changed. As long as its solid composition does not change, the water content of a sewage sludge influences its rheological behavior.

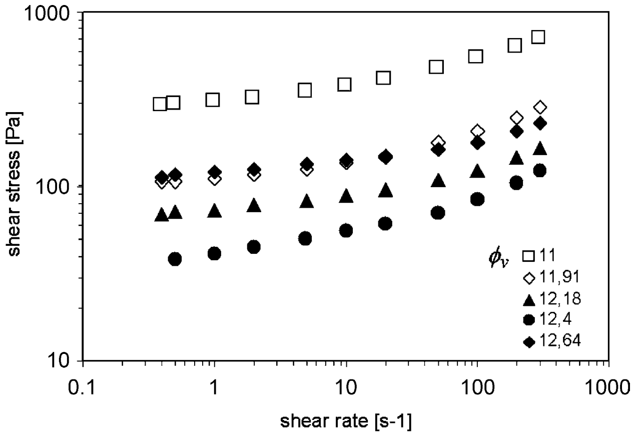

Fig. 3. Flow curves of pasty sewage sludges, from various plants, with different solid concentrations. The range of concentrations appears more limited than would be the case in terms of mass concentration; regardless, the relationship to rheological properties is clear.

In order to check this point we carried out tests with a material prepared with water and given fixed solid components obtained after freeze-drying. It is well known that freeze-drying alters physical characteristics, but only in a quantitative sense: qualitative rheological characteristics remained the same (Baudez, 2001). In this section, we only compared samples made with freeze-dried matter. No comparison was made with original sludge. Different samples were then prepared at different water contents, and left at rest during 5 h. Creep tests were performed for each sample to determine the relationship between mechanical characteristics and water content, all things being equal.

With such materials we found a simple, exponential relationship between the rheological parameters and the concentration of the sludge. This is especially clear for the second critical stress, which represents the limit between solid-like and liquid-like behaviors (Fig. 4). Similar results were found by Slatter (1997), with liquid sludge.

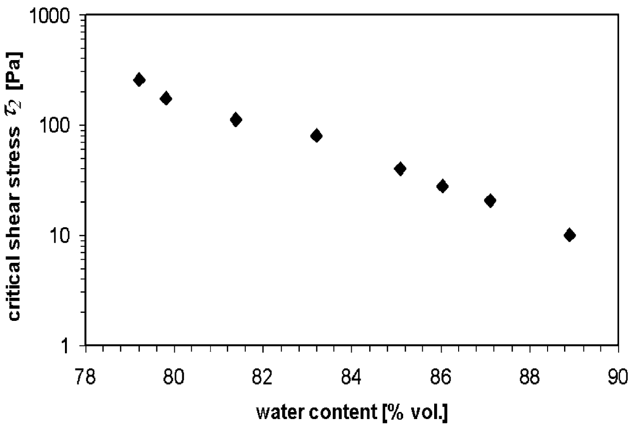

Fig. 4. Second critical stress as a function of the water content for a given sludge initially freeze dried.

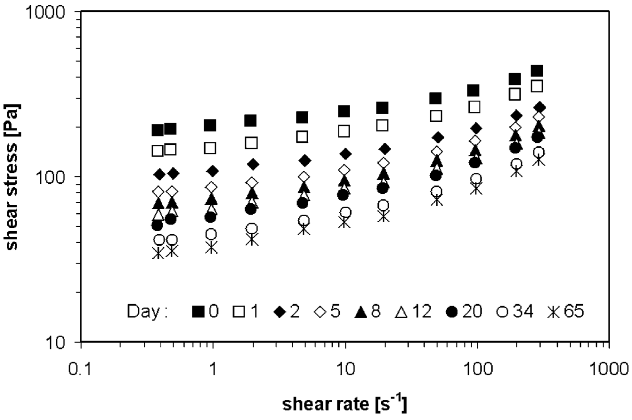

Fig. 5. Flow curves of a given sludge after different times of storage, with the same water content.

As shown by Monteiro (1997), the nature of the solid compounds is also of great importance. Baudez (2001) showed that during natural fermentation occurring during storage, the fluid behavior of a sludge sample became more predominant while its water content, as measured by 72 h drying at 60 °C, remained unchanged. Typical flow curves at different aging times are shown in Fig. 5 where we can see that the stress level of the curves decreases with the time of aging, whereas the overall aspect of the curve does not change.

To sum up we showed that pasty sewage sludges, whichever their composition, have a non-Newtonian behavior, always characterized by two critical stresses, τ_1 and τ_2, best fitted by a Herschel–Bulkley model at steady state. When solid interactions are more or less fixed the water content influences the behavior directly but, in general, solid interactions have a stronger influence than concentration, as may be seen in particular from the sludge fluidification during fermentation, while the water content remains unchanged (Baudez and Coussot, 2001).

4. Similarities in the steady state behavior

In order to compare more precisely the shape of the flow curves it is natural to scale the shear stress by the yield stress, such that all curves go through the same point ($\dot{\gamma} = 0$; $\tau_c = 1$). By this means we probably smooth the solid interactions since τ_1 represents the strength of particle interactions 'at rest'. In parallel it is necessary to scale the shear rate by a factor also related to viscous dissipation. This may be done by assuming that for high shear rates the energy dissipation resulting from interactions between solid particles becomes negligible compared to hydrodynamic dissipation. Under these conditions, like Coussot (1995); Coussot and Ancey (1999) for mineral

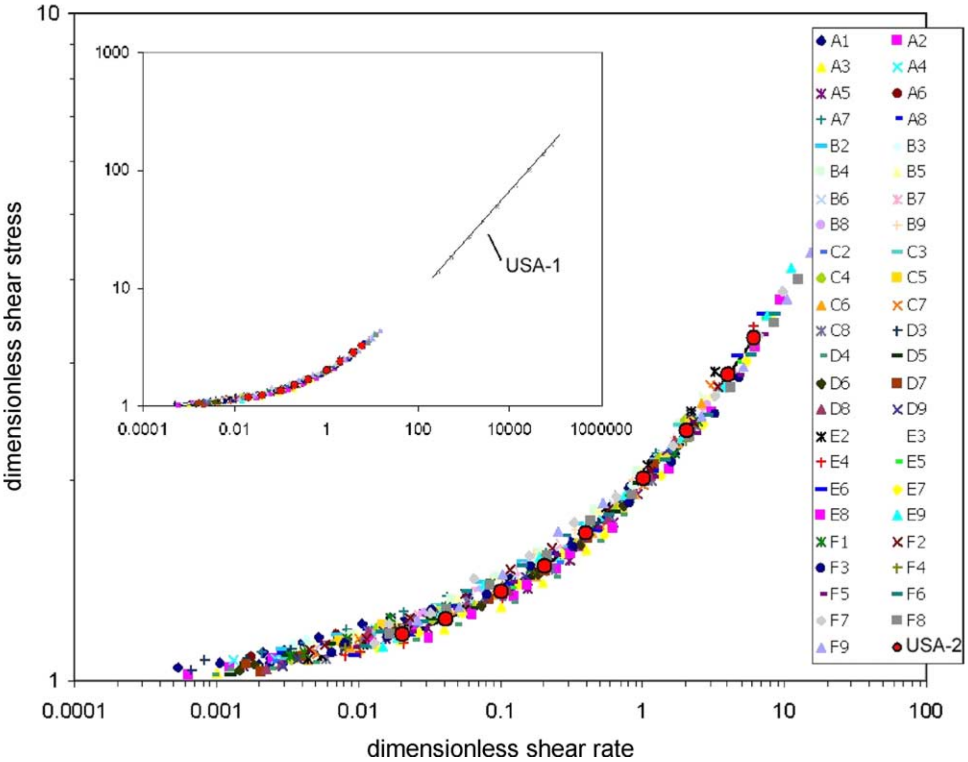

Fig. 6. Dimensionless flow curves for 48 sludges of various ages picked in different WWTP. Letters in the legend represent a plant, and numbers a sample picked in the considered plant. The two sludge samples named USA-1 and USA-2 were provided by Steven Dentel of the University of Delaware. USA-2 is a synthetic sludge (TS = 6.8%) made with latex and USA-1 is a digested sludge (TS = 2.3%), with no yield stress, best fitted by an Ostwald model. A master curve in the form of (6) has been be fitted to the data sets. The straight line represents the supposed asymptotic curve $Y = X^n$.

suspensions, it is natural to use a dimensionless number, Γ, defined as:

$$\Gamma = \frac{\mu \dot{\gamma}}{\tau_1} \quad (4)$$

where μ is the viscosity of the equivalent suspension of force-free particles in water. Within our framework it is sufficient to assume its value as 1 Pa s. Then the Herschel–Bulkley model can be written as:

$$\frac{\tau}{\tau_1} = 1 + \frac{K}{\tau_1^{1-n}} \cdot \left(\frac{\dot{\gamma}}{\tau_1}\right)^n = 1 + \lambda \cdot \Gamma^{\ n} = 1 + (\sqrt[n]{\lambda} \cdot \Gamma)^n \quad (5)$$

with $\lambda = K\tau_1^{n-1}$

In practice, λ can be determined from the flow curve by reducing the Herschel-Bulkley parameters by the first critical stress, τ_1.

The flow curves of all sludge samples may now be plotted in the dimensionless diagram ($\lambda^{1/n}\dot{\gamma}/\tau_1$; τ/τ_1). It appears that all the curves of our 48 samples (including a synthetic sludge made with latex), fall along a single, dimensionless, Herschel–Bulkley master curve (Fig. 6), which can be fitted by the following very simple equation:

$$Y = 1 + X^n; \ n \approx 0.45 \quad (6)$$

Note that, for high Γ values, Eq. (6) tends towards an Ostwald model, which seems consistent with the results of Fig. 2.

Since n can as a first approximation be considered as constant for all sewage sludges (at least in the range of shear rates in which we tested them), only two parameters remain to be determined: τ_1 and λ. The former may be considered indicative of the solid interactions 'at rest' while the latter essentially reflects the hydrodynamic interactions, which obviously in particular depend on the solid fraction but mainly through the volume the particles occupy and not significantly through their interactions. At this stage it becomes clear that a practical method, different from usual rheometry and making it possible to determine these two parameters would be very interesting for engineers and technicians. For the yield stress we present in the following a simple test, adapted from that used for fresh concrete, which can be carried out in situ. The determination of λ is more difficult since it is not related to a simple mechanical property but we propose an approximate approach based on concentration measurement.

5. Practical determination of the yield stress with the slump test

Different techniques, such as vane technique (Nguyen and Boger, 1983), inclined plane (Coussot and Boyer, 1995), slump test (Pashias et al., 1996), and Kasumeter (Spinosa and Lotito, 2003), exist for measuring the yield stress of fluids. The slump test appears to be the easiest tool to perform measurements inside the treatment plant because it only needs a piece of cylindrical rigid plastic pipe. This technique, which consists of suddenly leaving a volume of material flowing over a horizontal surface, is currently used in the concrete industry and in other fields (alumina industry for example) as a practical means to obtain a characteristic (the 'slump', i.e. the difference between the initial and the final heights) of the mechanical behavior of the material. Unfortunately the corresponding flow is not as controlled as inclined plane flows (Coussot and Boyer, 1995) from which it is in principle possible to extract the exact value of the yield stress. However, the slump test has the great advantage of requiring only a small volume of material. This explains why different authors have recently attempted to provide a stronger theoretical basis for the analysis of this test. After the works of Murata (1984); Chandler (1986); Pashias et al. (1996) were the first to propose a simple analytical approach making it possible to estimate the fluid yield stress from its slump. A comparison of these theoretical predictions with experimental results was rather successful, even if there remained a significant uncertainty on the exact determination of the yield stress. A comprehensive comparison between rheometric measurements and slump test results has appeared elsewhere (Baudez et al., 2002).

Since in general pasty sewage sludge have higher yield stresses than fresh concrete, we proposed a modification of the slump test (Baudez et al., 2002), i.e. an additional mass is placed at the top of the sample, which assures that small sample volumes can still be used (Fig. 7).

Following the theoretical analysis of Pashias et al. (1996) modified by Baudez et al. (2002), the yield stress τ_1 can be deduced form the slump height s with the following equation:

$$s = H - (h_0 + h_1) = H + z_0 - \frac{2\tau_1}{\rho g}\left(1 + \ln\left(\frac{\rho g (H + z_0)}{2\tau_1}\right)\right),$$

$$z_0 = \frac{m_0}{\rho \pi R^2} \quad (7)$$

where z_0 represents the 'virtual' increase of the sludge cylinder due to the added mass. H is the initial height of the cylinder and ρ represents the density. When the slump has

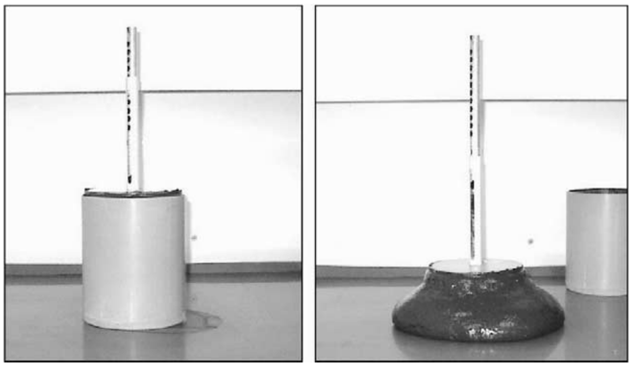

Fig. 7. The modified slump test with the additional hat: aspect of the cylinder and the material inside before (left) and just after (right) the test.

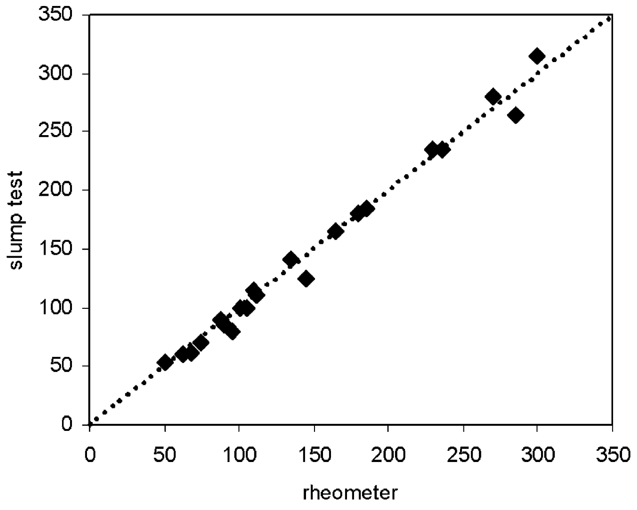

Fig. 8. Critical stresses obtained from rheometry and slump test with different sewage sludges.

been measured, knowing the mass of the hat, the yield stress can be determined from this equation, for example graphically or with a spreadsheet.

The results (in terms of yield stress) obtained from such slump tests and usual rheometrical tests (as above described) were compared with various samples (Fig. 8). An excellent agreement between these two techniques is found, which means that the yield stress clearly can be estimated in situ.

6. Practical determination of the parameter λ

It is instructive first to consider the rheological behavior of a given sludge during aging. As was already shown by Baudez and Coussot (2001), all the flow curves of the same sludge at different ages may fall along a single master curve when the stress and the shear rate are scaled by the yield stress only (cf. Fig. 9). This tends to confirm our suggestion that as long as the water content is fixed, only the yield stress

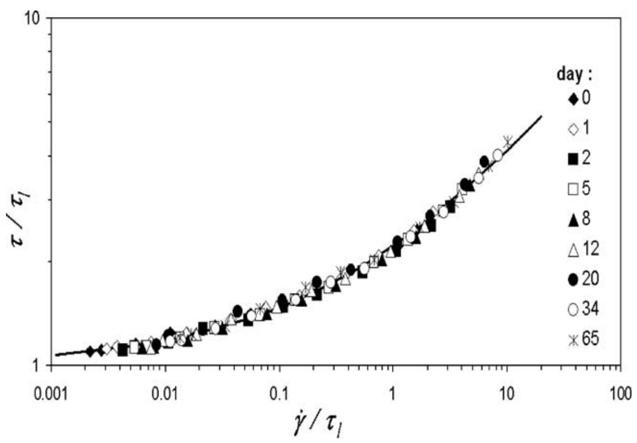

Fig. 9. Dimensionless flow curves (the stress and the shear rate have been scaled by the yield stress) for the sludge of Fig. 4.

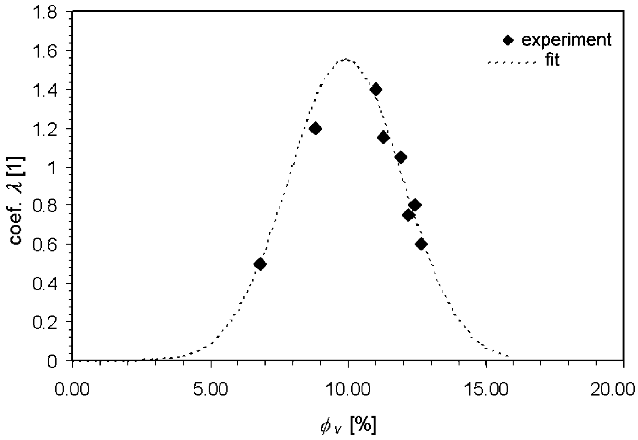

Fig. 10. λ as a function of Φ_v for eight different sludges, including the synthetic sludge. The dotted line corresponds to the fitted model with $p = 1.56, q = 0.12$, and $\Phi_0 = 9.9$; $R^2 = 0.97$.

plays a role in the behavior. The different aspects of the flow curves of different sludges simply result from the variation of λ, that we will suppose to be mainly related to the water content.

Under these conditions we can analyze the relationship between λ and the solid concentration. In this aim we determined from rheological tests the dimensionless flow curve (under aging) of various samples obtained in many wastewater treatment plants with different water contents. For each sample the solid concentration Φ_v was computed according to Eq. (3) from drying tests. The results are presented in Fig. 10. Some general trends appear. The points are aligned along a single curve that may be represented by the following model valid for solid concentration, *in volume*, between 5% and 15%:

$$\lambda = p \exp(-q\cdot(\Phi_v - \Phi_0)^2) \quad (8)$$

where p, q and Φ_0 are material parameters which have no physical significance yet. To sum up, according to Eqs. (5) and (8), the rheological behavior in steady state of a given pasty sludge can represented by:

$$\frac{\tau}{\tau_1} = 1 + (p \exp(-q\cdot(\Phi_v - \Phi_0)^2))\Gamma^{0.45} \quad (9)$$

It is worth noting that this empirical equation relies on the supposed simple relation between the solid concentration and λ which effectively appears in Fig. 10 but from a certain set of data, including a synthetic sludge. This equation is surprisingly successful considering the variable properties of wastewater sludges, and given the number of sludges represented, is suggested to be generally applicable.

To check the validity and uncertainty of our approach we compare in Fig. 11 the flow curve as it is obtained from usual rheometry and from our practical method. The results are in very good agreement. Thus the practical method, consisting of separately measuring the yield stress via slump test and the dimensionless 'consistency' index by determining the solid volume fraction, appears to be able

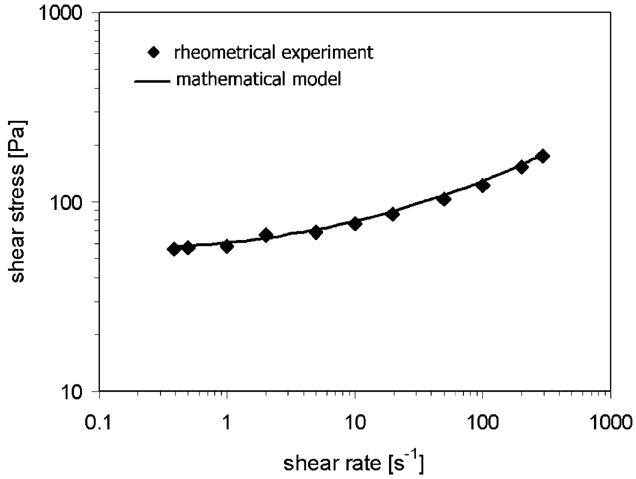

Fig. 11. Comparison of flow curves as determined from our practical technique (independent measurements of solid fraction and yield stress from slump test) and from conventional rheometrical tests. The characteristics of the sludge were: $H_2O = 85.7\%, MO = 9.9\%, MM = 4.4\%$. The slump test gave 52 Pa for the yield stress and the Herschel–Bulkley model measured with the rheometer was $\tau = 51.7 + 8\dot{\gamma}^{0.48}$.

to predict the behavior of pasty sewage sludge with a very good precision in the range of solid concentration [5–15%].

This practical determination of the flow curve parameters may not be recapitulated:

- the yield stress is estimated from slump test with a spreadsheet;
- the organic and mineral matter contents are determined by drying the sludge at 60 °C for 72 h (so the bound water is not removed) and the ratio between organic and mineral is obtained by burning the residue at 550 °C during 12 h;
- the flow curve of the sludge is then given by Eq. (9).

7. Conclusion

Pasty sewage sludges exhibit a non-Newtonian behavior characterized by three domains: a linear viscoelastic part below a first critical shear stress, then an intermediate regime and beyond a second critical stress, a purely viscous part. All the rheological parameters are linked with both concentration and solid interactions but interactions between the elements of the solid structure keep some similarity when the concentration remain unchanged. By scaling the shear stress and the shear rate, a simple dimensionless model can be fitted to the data representing the steady state behavior of any pasty sewage sludge. It then appears that basic measurements of two characteristics, the yield stress and the solid concentration, are sufficient to obtain the flow curve of materials with a solid concentration between 5 and 15%.

In practice this result is quite useful for WWTP workers who need to rapidly estimate some relevant, mechanical characteristics of their material without sophisticated equipment, especially for storage, landfill or spreading.

At this stage this method might provide a good estimate of the rheological behavior of many sludges. The model was also found applicable to a synthetic sludge prepared at the University of Delaware, suggesting that the relationship between the solid content and the consistency index could be more general than just describing pasty sewage sludges. However there remains some uncertainty on the validity of our empirical model for the consistency index as a function of the solid concentration. Future work should clarify this point of possibly establish a more physically sounded relationship between this parameter and some specific physico-chemical characteristics of the sludges.

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