Research Article

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Experimental difficulties often encountered with sludge rheological properties determination and advices to perform reliable measurements

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Abstract: Rheological parameters being of great importance for sludge process management, they are increasingly studied. However, experimental procedures may strongly impact their determination. Sample volume, measuring device depth and roughness but also mechanical history, have to be well-controlled to ensure reproducible results. Indeed, even if shear history can be erased with a sufficient preshear for diluted sludge, this paper clearly established that no steady state can be achieved for concentrated sludge. The longer the shear history, the lower the rheological characteristics: Reproducible results are hardly obtained.

More importantly, slippage appeared to occur even with surfaces of moderate roughness and the phenomenon is all the more important that the dry matter is high.

From all these observations, an experimental procedure based on the control of preshear and rest periods has been defined. Advices and precautions to observe are given in order to ensure reproducible and obtain unaltered results.

Keywords: Sludge, Rheology, Slippage, Shear history, Experimental procedure

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1 Introduction

According to the UNO [1], 55% of the world population lived in urban area in 2014 and this fraction will increase

up to 66% in 2050. Urban areas management will become one of the biggest challenges of the 21st century. As a direct consequence, wastewater management appears of crucial interest to protect public health and to ensure safe water supply. Sustainable urban areas thus imply high-performance wastewater treatment plants allowing to not only produce clean water but also to efficiently manage and recycle the residual sludge. High-performance systems require process control, especially flowing properties measurements for pumping devices [2–4], mixers and aeration systems [3, 5]. This implies an accurate estimation of sludge rheological properties.

However, sludge undergoes continuous physical, chemical and biological changes from the inlet to the outlet of the plant, depending on the applied treatments. It starts with very diluted and untreated raw material and ends with stabilized and dewatered pasty material. From a rheological point of view, the diluted sludge may be considered Newtonian [6] but as soon as the solid concentration increases, the non-Newtonian characteristics become predominant [7–10]. Sludge can no longer be summarized by a single viscosity but by a viscosity function which usually decreases as the shear intensity increases [11].

However, the thicker the sludge does not necessarily mean the higher the solid content [12, 13]. Overall, the variable nature of sludge has a strong influence on its rheological characteristics [11].

Indeed, rheological properties may differ according to the implemented treatments [14, 15]. For example, anaerobic digestion [16–18] or fermentation [11] induce a strong decrease of rheological characteristics.

Sludge origin and composition quantitatively impact the rheological behavior but they are not sufficient to explain the wide range of models found in the literature which are mainly empirical and based on data fitting. Measured rheological properties may also differ according to the experimental procedure used [8, 19]: As often described in the literature for non-Newtonian materials [20, 21], accurate flow curve determination implies the adoption of careful and well-controlled experimental pro-

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cedures to ensure reproducible results. These include appropriate geometry and experimental tests adapted to material characteristics ensuring data are fully representative of bulk properties. But this information is not systematically mentioned in the literature.

In the literature, sludge is always presented as a non-Newtonian shear-thinning material [10, 22–24], possibly with a yield stress. [11, 25, 26], highlight both thixotropic colloidal suspension characteristics at high shear rate [27] and polymeric behavior at low shear strain [24].

Sludge flow curve determination is classically determined by applying a stress sweep (respectively a shear rate sweep) [28, 29], to measure the corresponding shear rate (respectively shear stress) and to plot the shear stress versus the shear rate on a diagram. However, as sludge is known to be thixotropic [27, 30], results are also impacted by mechanical history. To limit this impact, a preshear phase often followed by a rest period is usually considered to erase mechanical history [31].

When existing and defined, preshear duration and intensity differ from one paper to another [25, 32–34], possibly contributing to the results disparity, as preshear intensity and duration impact final results [19, 30, 35].

To summarize, contrary to what one might think, reliable rheological measurements are not easy to perform and impacting factors have to be carefully considered. The way they are considered – or not – may affect final results. This paper intends to give some tips and precautions to take into account to ensure sludge rheological measurements are correctly performed.

2 Basis of experimental rheology

Rheology deals with the deformation and flow of condensed matter under the influence of externally acting forces [36]. Usually, the focus is solely made on the (simple shear) viscosity function, *i.e.* the relationship between the shear stress and the shear rate. This approach sounds reasonable when dealing with diluted materials for which lubrication theory apply [37] but it appears to be no longer valid as the solid concentration increases and elastic effects appear [38], which is mostly always the case with wastewater sludge for which yield stress and elasticity are noticeable at around 2% solids [34, 39, 40].

In viscometric flows, assuming that the temporal evolution of the material behavior is negligible, experiments often consist to establish the shear stress state through the

viscosity function (1):

$$\eta(\dot{\gamma}) = \frac{\tau}{\dot{\gamma}} \quad (1)$$

Shear rate ($\dot{\gamma}$) and shear stress (τ) are deduced from torque and displacement measurements using specific equations depending on the geometry. The standard geometries usually used are the concentric cylinders (also called the Couette geometry), the parallel plates and the cone-plate geometry. Among these geometries, the Couette one is the most frequently used [9, 41].

Equation (1) can be illustrated through Couette geometry.

A Couette geometry consists in concentric cup and bob (Figure 1), one rotating while the other remains immobile. Usually, the rotating device is the bob.

Figure 1: Schematic Couette geometry. M and ϕ are respectively the torque and the rotational speed of the bob.

In such geometry, the shear stress and shear rate distributions are determined from the following equations [42]:

$$\dot{\gamma} = -r \frac{\partial \phi}{\partial r} \quad (2)$$

$$\tau_r = \frac{M}{2\pi r^2 h} \quad (3)$$

where R_1 , R_2 and h are respectively the inner and outer radius and the height of the cylinders, r is a radius between R_1 and R_2 . These equations highlight that the shear stress distribution is known and varies with $\frac{1}{r^2}$ while the shear rate distribution is not known and needs the knowledge of the velocity profile.

For an unknown fluid and by assuming that the temporal evolution of the fluid behavior is negligible, the behavior law is of the form:

$$\dot{\gamma} = f(\tau) \quad (4)$$

From (2), it can be deduced that:

$$\begin{aligned} \dot{\gamma} = -r \frac{\partial \dot{\phi}}{\partial r} &\iff \dot{\phi} = \int_{R_1}^{R_2} \frac{\dot{\gamma}(r)}{r} dr \iff \dot{\phi} \\ &= -\frac{1}{2} \int_{\tau_1}^{\tau_2} \frac{f(\tau)}{\tau} d\tau \end{aligned} \quad (5)$$

The solution of (5) first implies to know the form of the equation fitting the rheological behavior of the material *i.e.* the functional relationship f between shear rate and shear stress must be known [43]. Second, it also implies the gap is fully sheared. Indeed, in the case of yield stress fluid for which an unsheared (dead) zone may exist, Equations (5) comes to:

$$\dot{\phi} = \int_{R_1}^{R_c} \frac{\dot{\gamma}(r)}{r} dr \quad (6)$$

where R_c represents the critical radius between the sheared and unsheared region.

Moreover, with a Couette geometry, the measured torque includes not only the contribution of the shearing of the vertical surfaces but also the shearing contribution of the top and bottom surfaces that have to be assessed [44].

3 Materials and methods

Dewatered sludge was sampled from the wastewater treatment plant then stored for several weeks before experiments to ensure the material is well stabilized, with negligible biological evolution. As a consequence, it can be assumed that sludge will have constant characteristics over one week, and used to check the reproducibility of the results. Moreover, after several weeks of storage, sludge turns into kind of homogeneous gel-like material (without visible flocs): This gel-like structure allows the use of a small gap and eases the calculation of shear rate and shear stress.

Dry matter was determined by drying the sample 24h at 105°C. Initial dry matter was 18% and diluted samples were prepared from the original sludge by adding deionized water (see average dry matters in Table 1). Diluted samples were then stored for additional 24hours for complete hydration.

Table 1: Dry matter percentage (by weight) of the diluted samples.

Diluted samples	Dry matter (%)	
	Average	Error
	4.16	0.017
	5.75	0.023
	8.35	0.001
	9.18	0.021
	10.05	0.004
	11.04	0.019
	11.68	0.025

A well-known non-thixotropic yield stress hair gel (see description in Table 2) model material [45, 46] was also used to check whether the results discrepancies come from the material or from the rheometer and the experimental procedure used.

Before each rheological measurement, temperature was set-up at 20°C ($\pm 0.1^\circ\text{C}$) and as soon as the temperature target was reached, a rest period of 5 minutes was applied to ensure temperature homogeneity within the samples.

A Malvern strain-controlled *Kinexus Pro* rheometer equipped with a cup and bob geometry (inner cylinder: $\varnothing$ 34mm, outer cylinder: $\varnothing$ 36.2mm, length: 70mm) was used. The bob had 3 different roughness: Smooth, sandblasted (60 μm of roughness) and serrated (150 μm of roughness). For both the bob roughness, the cup roughness is 150 μm . The experimental procedure consisted in the application of an increasing shear rate ramp from 0.1s⁻¹ to 500s⁻¹ in 5mn. Despite its weaknesses, among which the negative impact of inertia [19], the increasing shear rate ramp has been specially chosen to highlight how the final results may be connected to the experimental procedure. Steady state behavior has also been determined by applying constant shear rate steps of increasing intensities, until a steady shear stress has been reached.

Measurements were done in triplicate to evaluate the reproducibility.

Impact of sample volume and mobile depth on the flow curve have been first evaluated with the hair gel to ensure changes only come from experimental setup. Table 2 summarizes the experimental setups.

Figure 2: Flow curves of the gel according to the mobile depth (a) or the sample volume (b). The gel represents 4.56% of the total mass of the sample.

Then, for a given sample volume and bob depth, the impact of ramp duration has been observed by varying the duration from 2 minutes to 20 minutes.

For specific experimental conditions, the impact of mechanical history (preshear intensity ranging from 200 to 2000s⁻¹ and preshear duration from 5 to 30 minutes) on the final flow curve has been evaluated on diluted and concentrated sludge.

Finally, for three different mechanical histories, the structural evolution during rest has been analyzed by applying a 0.1% oscillatory shear strain with 1Hz of frequency, and by recording the elastic modulus over time.

4 Results and discussion

4.1 Impact of experimental setup and surface roughness

Even with a model material, experimental data are impacted by mobile depth or sample volume (Figure 2): The higher the mobile depth, the higher the shear stress level. A similar trend is observed with the sample volume at a given mobile depth. For a given shear rate, the shear stress level increases linearly with the sample volume or the mo-

Table 2: Experimental setups for the hair gel. [Full name of the gel (in French): Vivelles DOP Gel coiffant fixation extra-forte force 7].

Setups	Mobile depth (mm)	Sample volume (ml)	Ramp duration (min)
(1)	11	19.98	2
	10		
	9		
	8		
	7		
	6		
	5		
	4		
	3		
(2)	2	16.04	2
	0.5		
	5		

Figure 3: Shear stress corrective factor as a function of the mobile depth (a) or of the sample volume (b).

mobile depth. However, by applying a corrective factor linearly correlated to the mobile depth (Figure 3a) or the sample volume (Figure 3b), all the flow curves are superimposed (Figure 4). This corrective factor is obtained by reducing all stress levels to that corresponding to the mobile depth of 0.5 mm and the sample volume of 20 ml. Moreover, the corrective factor of both cases was found to be linearly linked to the normal force (Figure 5).

In the following, sample volume and mobile depth will be fixed at 20 ml and 5 mm. This depth corresponds to the Malvern specification for the considered geometry.

Roughness does not apparently impact the gel results (Figure 6a) even if some slight change can be noticed in the lower shear rate range (Figure 6b) suggesting the existence of slight slippage when smooth but also sandblasted surfaces are used.

Slippage on smooth surfaces has been largely well documented in the literature [45, 47, 48] but more seldom on rough surfaces. Indeed, non-slippage condition is assumed to apply when the roughness is sufficiently high [49] which is the case with our serrated tools. However Divoux, Tamarii [50] reported slippage with a gel despite a roughness of about 60 μm , which is approximately the sandblasted one. Thus, using non-smooth surfaces is not a sufficient condition to prevent slippage.

When varying the slope of the ramp, flow curves are superimposed in the high shear rate range regardless the ramp duration but transient effects take place in the low shear rate range (Figure 7). The departure from the steady state curve appears at a critical shear rate which is all the more high that the ramp duration is short (see insert in Figure 7). This could be caused by inertia [19] but the shear strain associated to this critical shear rate is constant (Figure 8) indicating that transient regime is dominated by solid-like properties [46].

4.2 Impact of material nature: additional difficulties when dealing with sludge

Low concentrated sludge is somewhere similar to the gels. Below a critical dry content (5.75% with our samples), no slippage is evidenced whichever the surface roughness (Figure 9). Mechanical history can be erased by applying a sufficient preshear in duration or intensity (Figure 10) and a steady state is reached.

However, for concentrated sludge, differences are noticeable. Slippage is systematically observed with the smooth surface and is all the more important that the solid concentration increases (Figure 11a). The same observation can be made with the sandblasted surface (Figure 11b) for which slippage occurs above a higher solid concentra-

Figure 4: Flow curves of the gel after shear stress correction. The corrected shear stress is obtained by dividing the shear stress level by the mobile depth corrective factor (a) or by the sample volume corrective factor (b).

Figure 5: Shear stress corrective factor as a function of the normal force from the mobile depth (a) or the sample volume (b) variations. The line fits in figures (a) and (b) are respectively: $1 + 0.064 \cdot NF$ and $1 + 0.21 \cdot NF$. NF is the normal force.

Figure 6: Gel flow curves according to the surface roughness. The fig. b) is the zoom done in the area bounded by the dots in figure a). The reference states of the samples are estimated following the elastic moduli when the material is on rest. For the serrated surface, the smooth surface and the sandblasted surface, the reference states are respectively 210.33 ± 0.22 Pa, 210.77 ± 0.33 Pa and 207.9 ± 2.86 Pa. The gel represents 4.56% of the total mass of the sample.

Figure 7: Flow curves of the gel according to the ramp duration. The inserted figure is the zoom of the area bounded by the dots. The gel represents 2.71% of the total mass of the sample.

tion (7% here). As for the gel, and consistently with previous studies [35], slippage magnitude is mainly noticeable in the low shear rate range and is more important with the smooth surface compared to the sandblasted one (the insert in Figure 12). No slippage is observed at high shear rates (Figure 12).

Figure 8: Critical strain of the gel according to the ramp duration. The gel represents 2.71% of the total mass of the sample.

More importantly, no steady state is reached with concentrated sludge (Figure 13). This result is highly questioning as it means the rheological behavior of sludge is strongly connected to its mechanical history. Indeed, shear rate range and duration both impact the flow curve: The lower the slope, the lower the stress level of the flow curve, mostly at high shear rate (Figure 14).

This result simply comes from the overall shearing time sludge was submitted to: The lower the slope, the

Figure 9: Flow curve of the secondary semi-diluted sludge according to the surface roughness.

Figure 10: Preshear stress evolution of the semi-diluted sludge according to preshear time. The preshear intensity is 1000 s^{-1} .

longer the sludge is sheared for a given shear rate. The overall shear strain is higher as the slope is lower: Because mechanical history induces a decrease of the viscosity, the corresponding shear stress is lower.

The above-mentioned results explain why reported results may vary from one operator to another with the same material. More importantly, they open up other problems related to sludge flowing properties such as wall slip conditions and the inevitable impact of the mechanical histories which appears to be fundamental characteristics to model the sludge flows along a treatment train.

Rheological measurement is not only the application of a shear rate ramp: Impacting factors have to be carefully considered. The way they are considered – or not – will modify the experimental results.

4.3 Experimental procedure definition for sludge rheological measurements

As showed earlier, sample volume and mobile depth both impact the quantification of rheological parameters. Because most of modern rheometers allow the control of the depth, this latest should not be a problem. However, the volume has to be carefully measured – and mentioned in the procedure – as it is an important parameter affecting the measurement.

The observed shift between results comes from the excess volume above the bob which induce an additional shear stress. This additional shear stress is directly correlated to the normal force, meaning that shear stress and normal stress are interdependent. To ensure appropriate measurements, bobs often present a mark indicating the level of immersion to be respected.

With non-thixotropic materials, respecting the user conditions defined by the supplier or at least always using the same mobile depth and sample volume will allow re-

Figure 11: Critical shear rate slippage between smooth and serrated surfaces (a) or sandblasted and serrated surfaces (b) according to the dry matter content. The critical shear rate is the lowest shear rate from which the flow curves of the corresponding roughness are superimposed.

Figure 12: Flow curves of a concentrated sludge according to the surface roughness. The inserted figure is the zoom of the area bounded by the dots.

Figure 13: Yield stress evolution of the concentrated sludge according to the preshear intensity. The duration is 5mn for a given preshear intensity. The yield stress is determined by fitting the flow curve with a Herschel-Bulkley model.

producible measurements. Unfortunately, neither the sample volume nor the mobile depth is indicated in sludge rheology literature. Even if it is expected these guidelines are followed, because results sometimes differ, the procedure should precise volume sample and mobile depth.

Moreover, it has been shown that slippage occurred even with rough surfaces and its impact is even all the more important that the solid content is high: The more concentrated the sludge, the more impacting the slippage, leading to a heterogeneous deformation [51] and to an underestimation of the yield stress, potentially leading to no apparent yield stress at all. Because slippage comes

Figure 14: Flow curves of the concentrated sludge according to the ramp duration. The inserted figure is the zoom of the principal figure between 4 s⁻¹ and 400 s⁻¹. The dry matter of the samples is 10.05%.

from the interactions between material and measuring tool surfaces [52], roughness must be sufficient to avoid slippage: A highly concentrated sludge requires a higher surface roughness. Thus, as previously highlighted, roughness value is an important parameter to be mentioned as well. From our knowledge, a 0.5 mm serrated surface appeared largely sufficient for biological sludge up to 12%.

When sludge displays a yield stress, which is the case when the solid content is higher than 2-3% [24, 27], the solid-like behavior is dominating as long as a critical shear strain has not been reached [53]. If a shear rate ramp is applied, the most frequent case in sludge rheology literature [17, 22, 54], low shear rates may correspond to an overall shear strain smaller than the critical one and sludge behaves like a solid in this range [46], leading to a perturbed flow curve with again an underestimated yield stress.

Moreover, because of sludge thixotropic properties, stress level is also impacted by shear history: The longer the shear rate ramp, the lower the stress level and so the lower the apparent viscosity and the yield stress. Sludge appears to be more fluid that the ramp is low.

To neglect as much as possible these time-effects, an energetic preshear, in intensity and duration, is often applied. However, it does not always allow the reset of shear history, especially when the sludge is paste-like (solid content higher than 5%): As for the slope of the ramp, the more intense the preshear, the lower the shear stress level.

Consequently, an arbitrary, even long, preshear cannot help at reaching a reference state.

To ensure measurements are performed with a material at the same reference state, both preshear intensity and duration and/or rest duration have to be adapted to reach a reference level designed by the operator. This can

Figure 15: Stop condition of the preshear based on a shear viscosity value of the sludge. The sludge dry matter represents 8.35%. The preshear intensity is 1000 s⁻¹ for both the three preshears. The sample is put in rest until reaching an elastic modulus equal to at around 60 Pa after each preshear (see Figure 15).

Figure 16: Stop condition of the restructuring period based on an elastic modulus value of the sludge. The preshear intensity is 1000 s⁻¹ for both the three preshears.

be done by including a “stop condition” in the experimental procedure: Preshear stops when a viscosity level has been reached (Figure 15) and/or rest stops when a restructuring level has been reached (Figure 16). In the latter case, when the elastic modulus reaches a ‘trigger’, the rest period ends and the next step starts.

This trigger allows a better reproducibility and a better comparison of the results. Indeed, adapting the preshear intensity and/or duration to reach a defined apparent viscosity representative of a given microstructure state ends to fully reproducible measurements (Figure 17).

Thus, preshear characteristics have to be considered as full part of the characterization procedure and must be well specified and representative of the process to be modelled.

Figure 17: Reproducibility of the sludge flow curve when a same reference state based on the elastic modulus value is chosen. The elastic modulus reference is reported in Figure 15.

5 Conclusion

Sludge rheology literature sometimes present disparities. The origins of these disparities have been investigated and it has been shown that two main external parameters, sample volume and mobile depth play a major role on results. Everything being equal, an increasing sample volume or mobile depth leads to an increasing level shear stress of the flow curve: These parameters impact the quantitative definition of rheological properties.

Besides, slippage inevitable occurs with concentrated sludge if the surface roughness is not sufficient and is the more important as the solid content is higher. Slippage may be high enough to apparently erase the yield stress, leading to inappropriate definition of the rheological behavior.

Finally, as internal origin, the time-dependent behavior and the mechanical history of the sludge were found to strongly impact the rheological measurements. Because of its thixotropic properties, the throughout stress level of the sludge flow curve decreases with an increasing duration of the ramp.

As far as the mechanical history is concerned, getting a steady state by preshearing the sample is only possible for diluted sludge. For concentrated sludge, the longer the preshear, the lower the rheological characteristics, without reaching any plateau.

Thus, to ensure reproducible measurements, the following experimental procedures have to be respected. First, the tool roughness must be sufficient to avoid slippage: A 0.5mm serrated surface appeared largely sufficient for biological sludge up to 12%. Then, to control the impact of the edge effects on the experimental data, the sample

volume and the mobile depth must be fixed. Besides, to go over the mechanical history, the reference state must be linked to a setpoint and not to a preshear duration or intensity. As long as the setpoint value is not reached, the reference state is not defined. The chosen reference state must be also reported in measurements methods. Finally, the slope of the shear rate ramp and the full procedure must be adapted to the process to be modelled. Indeed, sludge rheological measurements do not look at the determination of “definitive” rheological properties but more at the determination of “accurate” rheological properties regarding the process and the usually interconnected previous steps.

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