
Rheological characterization of sludge in divided granular-like and pasty states using a granular rheometer

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ABSTRACT

In this study, rheological properties of sludge are obtained for total solid (TS) content from 20 to 48 wt. %, using a granular rheometer (FT4 – Freeman Technology, UK). The physical consistency of sludge is first analysed using the soil mechanical approach based on Atterberg limits. Above the plastic limit (TS=43 wt. %), the sludge is in kind of divided granular-like state, while, below this limit, the sludge looks like a pasty material. Shear stresses/consolidations curves of both divided granular-like and pasty samples can be modelled by the Coulomb rupture criterion, highlighting a frictional behaviour. It is observed that the cohesion and the flowability index evolve inversely against TS content. As soon as TS exceeds 43 wt. %, a sharp decrease of the cohesion is highlighted which induces an increase of the flowability index. According to the standard classification of powder flowability, the divided granular-like sludge behaves like cohesive powders, while the behaviour of the pasty sludge is similar to that of very cohesive powder. The accuracy of measurements on pasty sludge is supported by an experimental comparison with a conventional rotating rheometer.

Keywords: Rheology, Sludge, Paste, Plastic phase, Divided granular-like, Solid, FT4 Granular rheometer, Cohesion, Friction, Flowability index

1. Introduction

Sludge management is an important issue for large municipal waste water treatment plants. From the available option, energy recovery from incineration, pyrolysis or gasification are promising routes. However, after mechanical dewatering, municipal sludge has a total solid (TS) content ranging from 18 to 35 wt. % TS [62]. These solids concentrations are too low for an efficient energy valorisation. Indeed, a minimum of 60 wt. % TS is expected for co-

incineration with municipal solid wastes while more than 85 wt. % is needed for pyrolysis and gasification [14,63,64]. As a consequence, drying is essential to increase the TS content prior to thermal valorisation [14,63,65–69]. However, during the drying process, the pasty sludge passes through a highly cohesive, plastic phase, where sludge tends to stick to the dryer walls and paddles, before turning into a free flowing when the granulation occurs. These textural modifications impact sludge hydrodynamics and negatively affect the dryer efficiency. The transition between plastic and divided granular-like states generally occurs at a TS mean value of 60 wt. % [1,2,4,5,9,15,16,19].

Despite the technology requirement, only few publications are devoted to the rheological characterization of sludge in pasty state (sludge having TS higher than 15 wt. %) mainly because of the difficulties to carry out relevant measurements [2,21–25]. Mouzaoui et al. succeeded to investigate the rheological behaviour of sludge up to the plastic limit around 43 wt. % TS (according to Atterberg's definition [40]), using a conventional rotating rheometer and without any physical pre-treatment [26,32,70]. They highlighted the existence of a frictional contribution, evidenced by the onset of dilatancy above 14 wt. % TS. Due to the increasing impact of the frictional contribution when the TS increases, the sludge moved from a plastic state to a wet divided one and conventional rotating rheometers are no longer suitable for the purpose of determining the rheological properties of sludge above the plastic limit.

The standard method to characterize granular materials flowing properties is the shear testing. Many shear testers have been developed to characterise the flowing properties of granular materials. Among them, the Jenike cell, the Schulze ring cell and the FT4 tester are extensively used [71–73].

The overall principle consists in applying a normal stress on the sample to create a consolidation (normal stress) and then to exert a tangential stress until the medium yields. For a given consolidation, the shear stress increases until a maximum which is defined as the rupture stress for a given consolidation. Running a shear test at different consolidations gives a curve called the “yield locus”. This curve characterizes the conditions under which granular material flows [74]. This curve characterises also the friction, the cohesion the flowability of the medium. To characterize the flowability of powders, Jenike proposed to use the flowability index ff_c [72,75]. The classification of powders flowability was proposed by Jenike and extended by [76] (Table 1).

Table 1. Classification of powder according to the ff_c value.

Flowability index	Flow behaviour	Examples	References
$ff_c < 1$	Not flowing	-	-
$1 < ff_c < 2$	Very cohesive	Nano-sized powder of carbon black	Leturia et al. [77]
		Flacked oat	Lopes Neto et al. [78]
$2 < ff_c < 4$	Cohesive	Coal with diameter $< 17.7 \mu\text{m}$	Liu et al. [79]
		Corn-starch	Lopes Neto et al. [78]
$4 < ff_c < 6$	Easy-flowing	Coal with $17.7 < \text{diameter} < 141.3 \mu\text{m}$	Liu et al. [79]
		Flacked corn	Lopes Neto et al. [78]
$ff_c > 10$	Free-flowing	Coal with diameter $< 141.3 \mu\text{m}$	Liu et al. [79]
		PVC powder	Leturia et al. [77]

The behaviour of granular materials varies strongly with water content. Under dry conditions, cohesive forces arise from inter-particle forces, mainly Van-der-Waals and electrostatic forces. Under wet conditions, the cohesive forces increase with the progressive increase of water content due to capillary bridges formed between particles [80–83]. The friction remains roughly constant with the increase of water content in the case of non-cohesive particles such as glass beads [80]. However, it strongly increases with the water content in the case of cohesive particles, such as food powders like semolina [81,84]. Indeed, rough particles are formed by agglomeration of native particles and nuclei. This roughness is responsible of the increase in the number of asperities and thus of contact points, which contributes to an increase of the friction coefficient. For a water content close to the plastic limit (around 52 % of dry basis water content of semolina), a certain proportion of the agglomerates already undergoes the agglomerate/paste transition. As a result, cohesion drops sharply. As a result, cohesion drops sharply. Moreover, the medium can no longer be described as a granular material (native particles, nuclei, and agglomerates), but like a continuous granular paste [81].

However, these results were defined on granular materials. Moreover, to the author's best knowledge no similar approach has been performed on pasty dewatered sludge. Thus, the aim of this study is to characterise the flowing properties of sludge above the plastic limit (also called divided granular-like sludge in the following) using a granular rheometer and to highlight the influence of the TS content of the flowability. In a second part, the use of the granular rheometer is extended below the plastic limit to characterise the transition between the pasty and divided granular-like states. Results obtained on pasty sludge are then compared to those obtained with a conventional rotating rheometer to support the accuracy of measurements.

2. Material and methods

2.1 Sludge origin and conditioning

The dewatered digested sludge was sampled at the waste water treatment plant (WWTP) of Albi city (France) after the step of centrifugation. Its initial TS is 20 wt. %. Samples at higher TS contents are prepared by centrifugation (at 30000 rpm or 108800 g) for 30 minutes to 24 hours at controlled ambient temperature. After 24 hours of centrifugation, the TS content doesn't increase any more. To obtain higher TS content, part of the highest concentrated sludge is let at room temperature for 4 to 8 hours for natural drying.

2.2 Sludge characteristics

The final TS content is determined by drying the wet sample at 105°C during 24 hours. The volatile solid (VS) content is determined after igniting the dry solids at 550 °C for 2 h [37].

After centrifugation, samples have TS contents ranging from 20 to 43 wt. % depending on the dewatering duration. After drying at ambient temperature during 4 and 8 hours, samples have TS contents of 45 and 48 wt. %, respectively. The VS content is equal to 63 % (of dry weight).

The plastic limit (TS_p) is determined by using standardised soil mechanics trials based on Atterberg's limits [39,40]. Experimentally, it corresponds to the concentration at which the sludge can no longer be rolled into threads of 3 mm in diameter and 10 cm in length without breaking into pieces. The transition from plastic to divided granular-like (or semi-solid) states occurs at $TS_p=43$ wt. % (Figure 1).

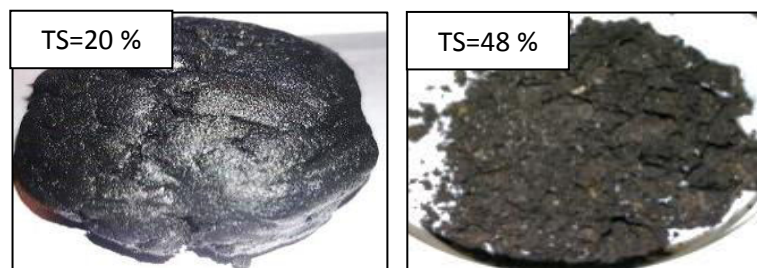


Figure 1. Pictures of sludge having a pasty state (left) and a divided granular-like state (right).

If necessary, the volume concentration ϕ could be calculated according to Equation 1:

$$\phi = \frac{1}{1 + \frac{\rho_s}{\rho_e} \cdot \frac{1 - TS}{TS}} \quad \text{Equation 1}$$

Where ρ_s and ρ_e are the densities of solid and the liquid fraction. Sludge can be assumed as a two phase (water and total solids) mixture, in which the liquid fraction corresponds to water

and the solid fraction to organic and mineral matters. The dry density ρ_s is measured at $\rho_s=1700 \text{ kg/ m}^3$ using a helium pycnometer (Micromeritics Accupyc II 1340).

2.3 Rheological measurements

Above the plastic limit ($TS > TS_p$), rheological measurements can only be performed with a granular rheometer. However, below the plastic limit ($TS < TS_p$) conventional rotating rheometers can be used too. Therefore, both devices are used to characterise the rheological behaviour of the pasty sludge ($20 \text{ wt. } \% < TS < TS_p$). The two equipment are briefly described in the following with the experimental protocols implemented.

2.3.1 Granular rheometer (divided granular-like and pasty sludge)

FT4 rotational rheometer (Freeman Technology, UK) is used to carry out the experimental tests in shear measurements. Experiments are all done at ambient temperature. This apparatus is able to apply a constant normal stress of consolidation on the sample over the range 1-100 kPa. It can rotate with a constant velocity (18 or 36°/min) thanks to a shearing torque over the range 2-900000 μNm .

Measurements are carried out in a cylindrical glass cell of 25 mm of diameter and 10 mm of height (figure 2-a). The cell is filled to the brim with sludge as shown in Figure 2-b.

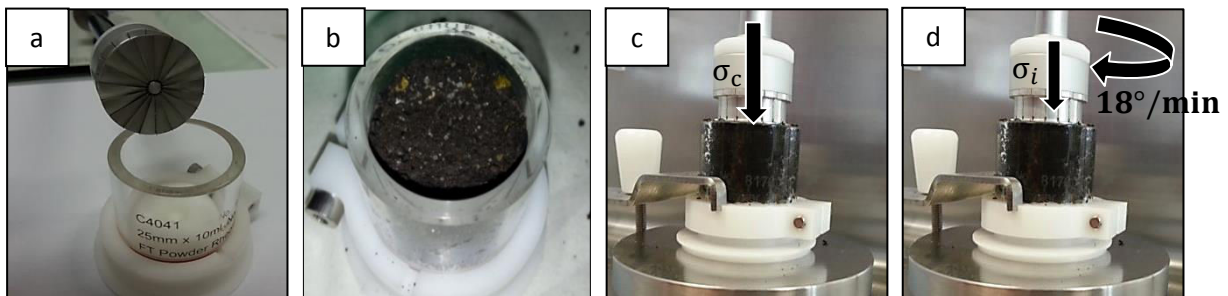


Figure 2. FT4 granular medium shear cell, sludge preparation and shearing procedure.

The sample is submitted to a pre-consolidation stress σ_c for 60 s (Figure 2-c). This step is needed to identically prepare all the samples and, therefore, to obtain reproducible results. The pre-consolidation is then removed and a consolidation, lower than the pre-consolidation σ_c , is applied. The consolidated sample is then subjected to a constant angular velocity (figure 2-d). Measurements at a speed of 36°/min were poorly reproducible. Thus, a velocity of 18°/min has been considered to perform all the experiments.

A schematic diagram of the shear cell measurements is presented in Figure 3. For a given consolidation (σ_i), the shear stress signal is recorded versus time. The shear stress τ (ratio of the torque to the sheared surface) increases until a maximum shear stress is observed. The peak corresponds to the rupture stress τ_{si} , which characterizes material failure and flow at the considered consolidation σ_i [74].

The tangential stress is then cancelled, the normal load cancelled and the sample is replaced to continue the measurements. This procedure is repeated with increasing consolidations (σ_i), all being smaller than the pre-consolidation (σ_c). The different pairs (τ_{si} , σ_i) constitute the so-called yield locus curve (Figure 3 right), for a given pre-consolidation σ_c .

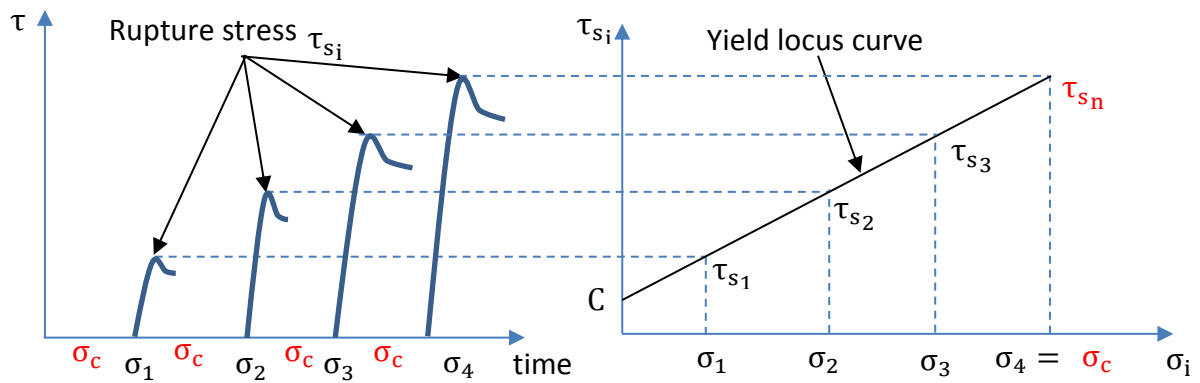


Figure 3. Descriptive diagram of the shearing test.

The same procedure is repeated for the different TS contents ranging between 48 and 20 wt. %.

To identify the optimal pre-consolidation (σ_c) and thus the range of the applied consolidations (σ_i) that will be used in this study for all the sludge samples, various values of consolidation (σ_c), ranging from 20 to 90 kPa, were selected. After the pre-consolidation σ_c , a consolidation equal to the value of σ_c was applied to the sample. The shearing test is performed (with a velocity of 18°/min) on the highest and lowest concentrations (48 wt. % and 20 wt. % TS).

The yield locus curve is modelled by the Coulomb rupture criterion, according to Equation 2:

$$\tau_{si} = \mu \sigma_i + C \quad \text{Equation 2}$$

Where μ is a static friction coefficient and C the cohesion which is defined as the shear resistance at zero normal stress [85–87].

The flowability index, defined by Equation 3, can be determined from the yield locus curve and Mohr-circles (Figure 4).

$$ff_c = \sigma'_1 / \sigma'_c$$

Equation 3

With σ'_1 the major consolidation and σ'_c the unconfined yield strength. The major consolidation, σ'_1 , is the maximum consolidation that can exerted on the medium. It is obtained by drawing a large Mohr circle which passes through the last point of the yield locus curve and is tangent to it. The unconfined yield strength is equal to the diameter of the Mohr-circle passing by the origin and tangent to the yield locus curve.

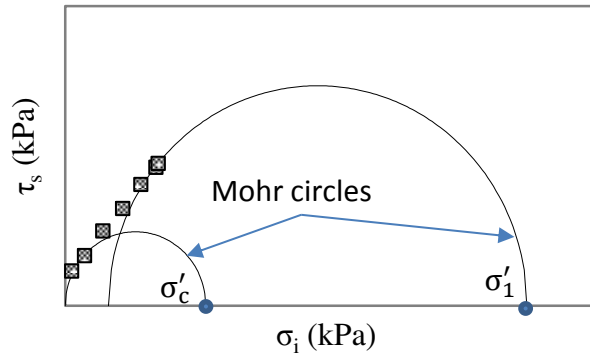


Figure 4. The yield locus curve and Mohr-circles.

σ'_1 and σ'_c are the major consolidation and the unconfined yield strength, respectively.

2.3.2 Conventional rotating rheometer (pasty sludge)

Rheological measurements are performed with a stress-controlled RS600 instrument (HAAKE) piloted by RheoWin software. This rheometer can apply a torque in the range of $M_{min}=0.1$ and $M_{max}=200000 \mu Nm$. The rheometer is equipped with a normal force sensor with a detection range of $F_n=0.02-30 N$. Serrated parallel plates geometry is used (with radius $R=10$ and $R=17.5 mm$) to avoid wall slip (Figure 5). Measurements are performed at room temperature. A constant gap of 2 mm is maintained.

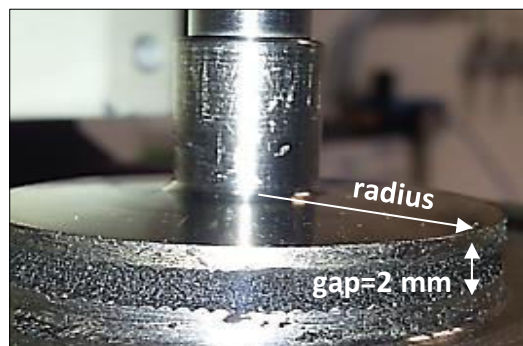


Figure 5. Pasty sludge (TS=20 wt. %) sheared between parallel plates.

A preliminary constant dynamic strain ($\gamma \sim 0.3 \%$) in the linear viscoelastic range is applied for at least 300 seconds to relax stresses generated by loading the sample between the parallel

plates [26]. Then, a stress sweep, corresponding to 0.1 to 1000 %, is applied at 1 Hz. The normal stress ($\sigma = F_n/\pi R^2$) is recorded in parallel with viscoelastic properties. To avoid fracturing, the stress sweep is performed according to the experimental procedure of surface correction developed by Mouzaoui et al. [26,32]. This procedure consists of applying a stress sweep in successive steps of constant dynamic stress of increasing intensity. Prior each constant stress step, a step at a reference state defined by a constant dynamic rotational angle ϕ_{ref} is applied. The latter corresponds to a constant dynamic reference strain $\gamma_{ref}=0.3$ % chosen in the linear viscoelastic plateau (LVE), where, by definition, stress and strain are proportional. By applying this reference state, edge effects (fractures) lead to a decrease of the corresponding stress and complex modulus (stress and strain ratio). As the complex modulus must be identical at the reference state before and after fractures, the surface really sheared (S) can be calculated, allowing the correction of the stress τ and the corresponding strain γ .

3. Results and discussion

3.1. Rheological behaviour of sludge using the FT4 granular rheometer

3.1.1. Determination of the optimal pre-consolidation σ_c and the corresponding σ_i

The shear stress signal is plotted in Figure 6 for the most and the less concentrated sludge (48 wt. % TS and 20 wt. % TS, respectively) at various values of σ_c .

For TS equal to 48 wt. % (namely above the plastic limit), the typical powder-like behaviour is recorded whatever the applied consolidation: the shear stress increases as a function of time until a maximum and then progressively decreases before tending toward a plateau [73,74,88].

For the less concentrated sludge (20 wt. % TS), instabilities in shear stress measurements take place at sufficiently high shear consolidation (from 40 kPa). The pasty sludge resists to the consolidation increase until it is not strong enough to support the applied load: the sludge escapes the measurement cell (Figure 6, right-insert). The same behaviour was observed for all the pasty samples ($TS < TS_p$), but the more concentrated, the more resistant the sludge is. Therefore, to pre-consolidate all the samples in the same way, the pre-consolidation is taken equal to $\sigma_c=30$ kPa in the following.

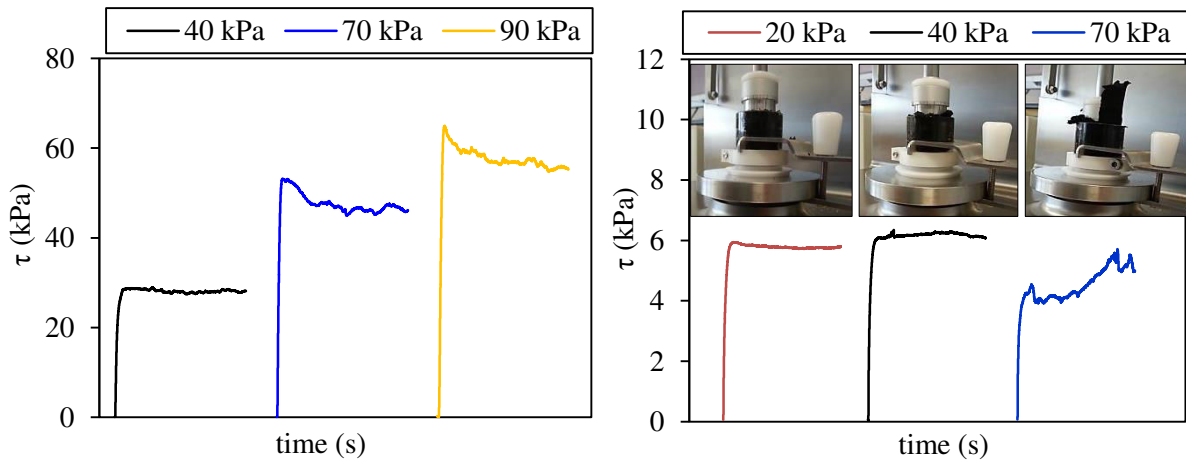


Figure 6. Shear stress versus time for different normal stresses (consolidation). Left: divided granular-like sludge (TS=48 wt. %); Right: pasty sludge (TS=20 wt. %).

3.1.2. Influence of TS content on the rupture stress, the friction coefficient and the cohesion

Figure 7 illustrates the rupture stress (τ_s) versus the normal stress (σ_i) for different TS contents in the range 20-48 wt. %. Curves exhibit a linear increase highlighting a frictional behaviour, like powders and granular materials. The variation of the shear stress-normal stress can be modelled following the Coulomb rupture criterion (Equation 2), whatever the TS content.

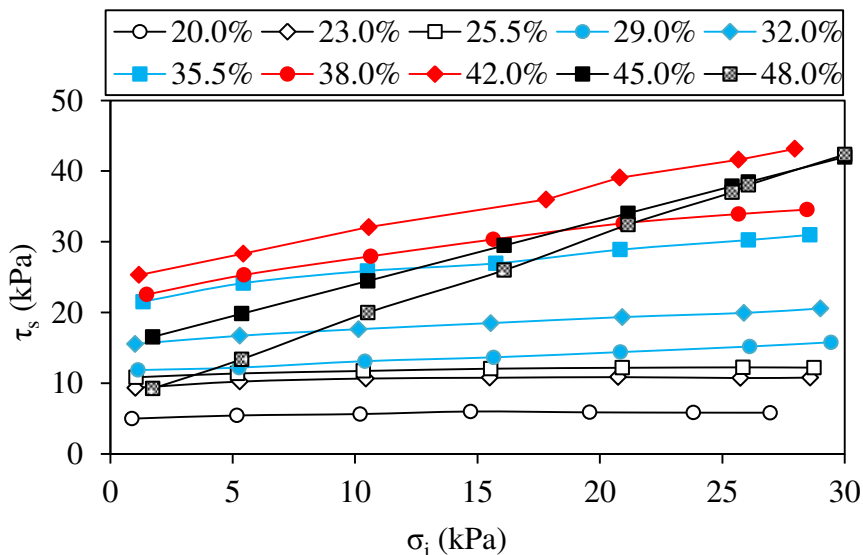


Figure 7. Rupture stress (τ_s) as a function of the consolidation (σ_i) for various TS contents between 20 and 48 wt. %.

Values of the friction coefficient and cohesion are reported in the Table 2 and Figure 8, together with the flowability index. The flowability index and the cohesion evolve inversely

as function of the TS content. The flowability index decreases while the cohesion increases, when the TS content of sludge increases from 25.5 to 43 wt. %. Then, the flowability index increases while the apparent cohesion decreases sharply when TS content increases beyond 43 until 48 wt. %. The friction coefficient increases with TS in the whole range of concentration.

Table 2. Values of the friction coefficient cohesion the flowability index as function of TS contents.

TS (wt. %)	20.0	23.0	25.5	29.0	32.0	35.5	38.0	42.0	45.0	48.0
C (kPa)	5.3	9.9	11.3	13.0	15.8	21.0	22.9	24.9	15.0	7.2
μ (-)	0.03	0.04	0.04	0.14	0.16	0.31	0.43	0.67	0.90	1.17
ff_c (-)	3.1	2.0	1.8	1.7	1.4	1.2	1.2	1.2	2.1	3.7

According to the standard classification of powder flowability [72,75,76], the behaviour of the pasty sludge is similar to that of very cohesive powder, while the divided granular-like sludge behaves like cohesive powders.

The extracellular polymeric substances (EPS) are the major components of sludge, with 50-60 % of the organic fraction [89]. A denser, stronger and stiffer rigid network of these biopolymers is formed with the increase of the TS content [90]. This induces an increase of the sludge glueyness [91] and thus an increase of the cohesion and a decrease of the flowability of sludge.

Then, the absence of moisture when $TS > TS_p$ in the sludge causes a strong increase of biopolymers viscosity to such an extent that the sludge may develop defects such as voids and cavities [92–94]. This transition is responsible for the formation of a crumbly material (divided granular-like sludge) and consequently the decrease of the cohesion and the increase of the flowability index.

Finally, the increase of TS content results in a gradual decrease in lubrication between sludge components. This favours the direct frictional contacts between sludge components and results in the increase of the friction coefficient. Moreover, cavities and voids formed into the sludge when $TS > TS_p$, lead to increase the roughness of sludge particles, increasing the friction coefficient.

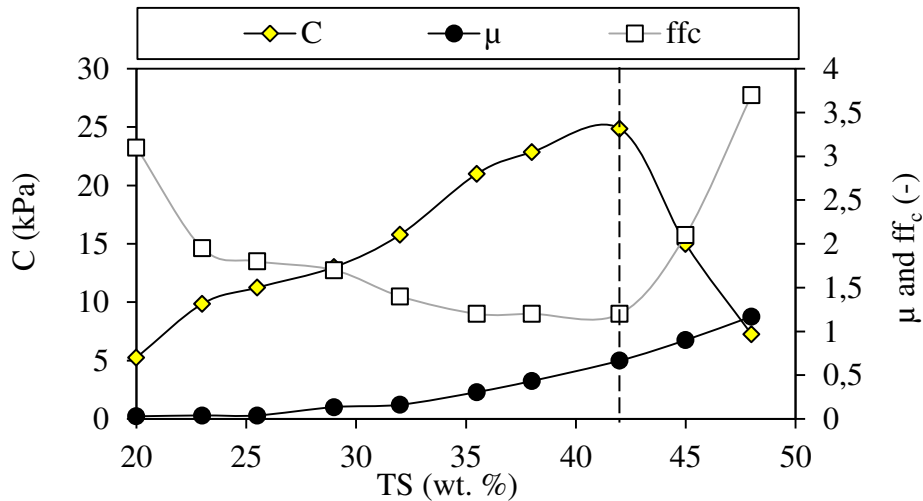


Figure 8. Variation of the friction coefficient, the cohesion and the flowability index according to TS content.

Figure 9 presents the evolution of the rupture stress as function of TS content for two different consolidations ($\sigma_i=1$ and $\sigma_i=16$ kPa) with the purpose of studying the influence of consolidation on the stickiness characteristics of sludge.

At low solid concentration (TS < 25.5 wt. %), the resistance was weak because of the high moisture in the sludge inducing lubrication between solids. As a result, the sludge showed a fluid-like behaviour ($ff_c=3.1-1.8$). With the increase of the TS content, sludge shows increasing resistance to movement. This behaviour can be attributed to the increase of cohesive forces as shown in Figure 8. When the TS content of the sludge achieves the plastic limit $TS_p=43$ wt. %, the shearing stress achieves a maximum. The maximum shear stress that was needed to cause the rupture of the sludge is found equal to 25 and 36 kPa for the two different consolidations $\sigma_i=5$ and $\sigma_i=16$ kPa, respectively. The high values of shearing stress at $\sigma_i=16$ kPa could possibly be explained by the increase of the sludge density at higher consolidation.

Li et al. [16] observed the same pattern when mapping the plastic (sticky) phase of sludge by the Jenike shear method and a device developed from the work of Peeters et al. [1]. For a sludge with a VS of 39 % VS of dry weight, they reported that the maximum shear stress needed to cause slippage is equal to 5.4 kPa for a consolidation of approximately 5 kPa. The peak value of the shear stress reported by Li et al. [16] was 5 times smaller than that obtained in this study. This difference is probably due to the distinctive nature of the sludge samples and to the different experimental set-ups. After the maximum, a rapid decrease occurs because of the formation of a crumbly material (divided granular-like sludge).

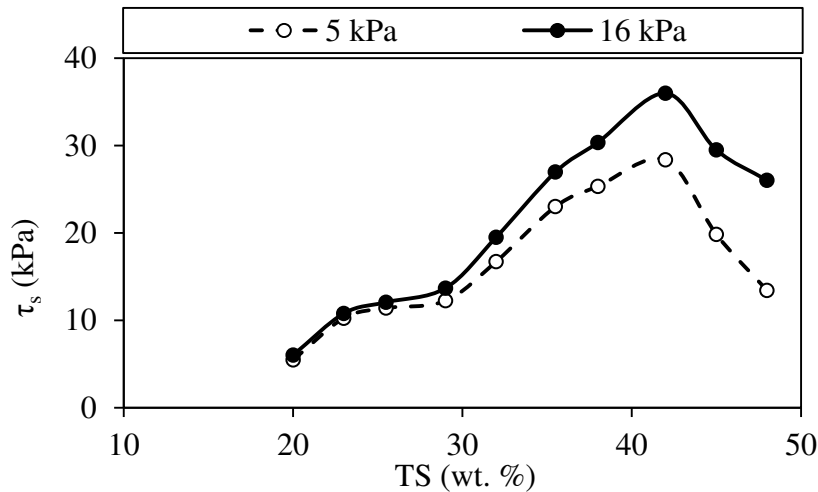


Figure 9. Shearing strength of the sludge as a function of TS content, for two different normal stresses.

3.2. Determination of the yield-like stress using the conventional rheometer (pasty sludge)

Figure 10 presents the evolution of the normal stress (σ) and the viscous (or dissipation) modulus (G'') under stress sweep measurements, for a pasty sludge having a TTTS of 26.3 wt. % TS. Viscous modulus and normal stress are nearly constant up to a critical shear stress $\tau_c=10$ kPa, reflecting that the sludge structure is preserved. Then, when the shear stress exceeds τ_c the normal stress abruptly decreases simultaneously with G'' . These decreases are concomitant with the appearance of fractures, that results from viscous-like dissipation. The critical shear stress at which the viscous modulus and normal stress start to decrease could be reasonably assumed as a yield-like stress. The same procedure is applied to obtain the yield-like stresses at various TS contents.

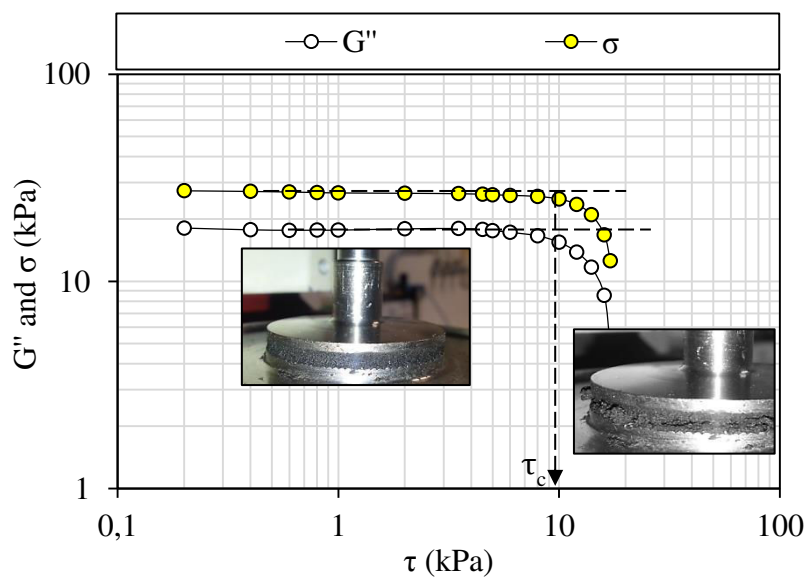


Figure 10. Evolution of the viscous modulus and the normal stress during stress sweep measurements for a sludge at 26.3 wt. % TS.

3.3. Validation of results obtained on the granular rheometer

In order to confirm the validity of results obtained on the granular rheometer, the rupture stresses (τ_s) obtained on the granular rheometer at various TS contents are compared to the yield-like stresses that can be obtained on the conventional rotating rheometer. But for a valid comparison, the normal stress recorded during measurements on the conventional rotating rheometer (Table 3) should be used to consolidate the material in the granular rheometer.

Table 3. Values of the normal stress for some TS contents during stress sweep measurement on the conventional rheometer.

TS (wt. %)	20.0	23.0	25.5	26.3	29.0	35.5	42
σ (kPa)	11	20	24	27	34	60	100

Figure 11 displays the evolution of τ_s and τ_c as function of TS contents. In the range of TS content between 20 and 43 wt. %, τ_s and τ_c are equal. This result supports the accuracy of measurements and the technical validity of the granular rheometer to characterise pasty sludge.

Eilers [95] law (Equation 4) is currently used to describe the evolution of solid-like properties (yield stress, viscous and elastic moduli, consistency index) of granular pastes [58–60]. It can be used to fit the data in the studied range of TS contents:

$$\tau_c = b \left(1 + \frac{1.25 \text{ TS}}{1 - \frac{\text{TS}}{\text{TS}_m}} \right)^2 \quad \text{Equation 4}$$

Where b is a model parameter and TS_m is the maximum packing solids concentration derived from Eilers equation. Theoretically it corresponds to the maximum of solid particles that can be placed in a volume of fluid. Its value depends on the shape and size of the particles in the suspension. In this study, a value of $\text{TS}_m=55$ wt. % and $b=0.008$ are found to best fit the experimental data. This value of the maximum packing solids concentration corresponds to a maximum packing volume concentration $\phi_m=42$ % according to Equation 1. This is very close to that that obtained on fresh tricalcium silicate (C_3S in short) equals to 45 % [36], but much lower than that of compact random packing of hard spheres value, where it is equal to 64 % [61,96,97]. This suggests that the sludge components are not hard and compact spherical particles but deformable objects.

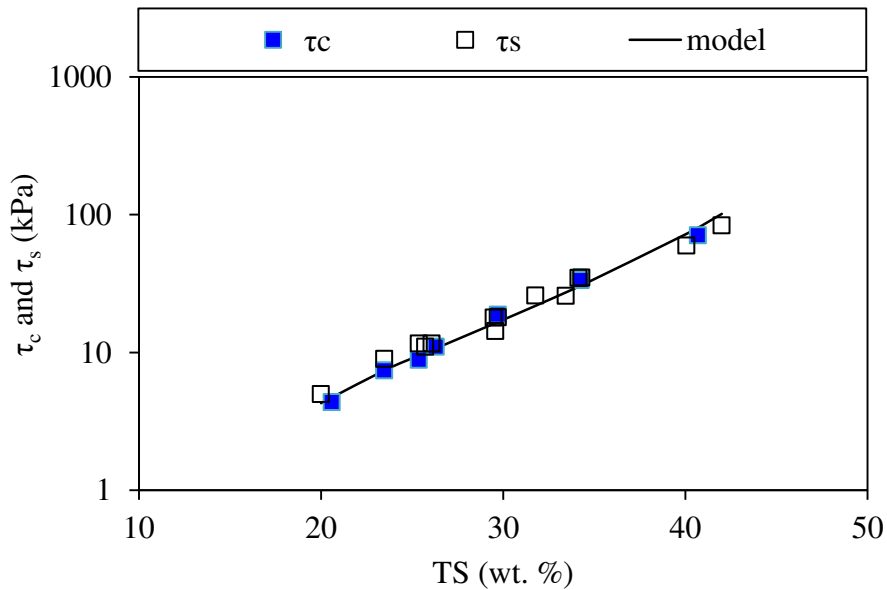


Figure 11. Comparison between τ_c and τ_s against the TS contents.

4. Conclusion

In this work, the rheological behaviour of a sludge is analysed by using a granular rheometer in a TS range of 20-48 wt. %. The transition between pasty and divided granular-like states occurring at around 43 wt. %. The results obtained can be modelled using the Mohr-Coulomb criterion which reflects a granular (or frictional) behaviour. The cohesion and the flowability index evolve inversely against TS contents, the cohesion increase resulting in a flowability index decrease and consequently an increase of the resistance to shear. As soon as TS exceeds 43 wt. %, a sharp decrease of the cohesion is highlighted which induces an increase of the flowability index and consequently a decrease in resistance to shear. Moreover, results reveal that the sludge behaves as a very cohesive powder when the TS content is lower than 43 wt. % and as a cohesive powder once exceeding 43 wt. %. Moreover, shear stresses/concentrations curves present the same shape with that obtained in the literature to map the plastic (sticky) phase of sludge.

The comparison with a classical rotational rheometer shows similar and equal characteristics. This makes it possible to confirm the validity of measurement and the use a granular rheometer to characterise pasty sludge.

3.2. Résumé

Dans cette étude, les propriétés rhéologiques des boues sont caractérisées dans une gamme de siccité allant de 20 à 48 % à l'aide d'un rhéomètre granulaire (FT4 - Freeman Technology, UK).

Ce travail démontre que les résultats obtenus avec le rhéomètre granulaire peuvent être modélisés à l'aide du critère de Mohr-Coulomb, mettant en évidence un comportement frictionnel.

On observe que la cohésion et l'indice de fluidité évoluent inversement en fonction de la siccité. Jusqu'à 43% de siccité, la cohésion augmente, entraînant une diminution de l'indice de fluidité et, par conséquent, une résistance croissante au cisaillement. Dès que la siccité dépasse 43 %, une forte diminution de la cohésion est mise en évidence, ce qui induit une augmentation de l'indice de fluidité et par conséquent une diminution de la résistance au cisaillement.

Selon la classification standard, les boues déshydratées à consistance granulaire se comportent comme des poudres cohésives tandis que le comportement des boues pâteuses est similaire à celui d'une poudre très cohésive.

De plus, ce travail montre que pour une consolidation donnée, les courbes contraintes de cisaillement/concentrations présentent la même forme que celle obtenue dans la littérature pour cartographier la phase plastique (collante) des boues. Il serait possible donc de caractériser la phase plastique en utilisant le rhéomètre granulaire.

La fiabilité des mesures sur les boues pâteuses est étayée par une comparaison expérimentale avec un rhéomètre rotatif conventionnel.

4 Conclusion

L'impact de la siccité, dans une gamme allant de 2 à 48 %, sur le comportement rhéologique des boues a été étudié. Le but était d'identifier et modéliser les différents régimes qui gouvernent le comportement rhéologique de la boue afin de mieux comprendre son écoulement dans le sécheur.

Pour étudier le comportement rhéologique des boues de consistances liquide et plastique, nous avons utilisé un rhéomètre conventionnel avec la mise en œuvre de la procédure de correction de surface présentée au chapitre 3. Cependant, pour étudier le comportement rhéologique des boues déshydratées de consistance granulaire, nous avons utilisé un rhéomètre granulaire avec la mise en œuvre du test de cisaillement utilisé classiquement dans les matériaux granulaires.

Sur la base de ces expérimentations, un schéma rhéo-physique général (Figure 1) peut être proposé, dans lequel le comportement rhéologique est lié à la siccité de la boue.

Lorsque la siccité est inférieure à 6 %, la boue est décrite comme une suspension diluée. Les interactions hydrodynamiques ou physico-chimiques gouvernent le comportement rhéologique et le régime est appelé visqueux.

Pour des siccités entre 6 et 14 %, une contribution additionnelle à la contrainte seuil apparaît lorsque la concentration dépasse le seuil de percolation rhéologique 6 %. Le système est dans une phase intermédiaire entre un régime visqueux et un régime frictionnel. Aucune fracture n'est observée dans cette gamme de concentration. La boue est décrite comme une suspension concentrée ayant une consistance liquide (selon la définition de limites d'Atterberg).

Lorsque la siccité se trouve dans la gamme 14-43 %, un régime frictionnel est mis en évidence par l'apparition de la dilatance, comme dans le cas des pâtes granulaires. C'est également la gamme de siccité dans laquelle le phénomène de fracturation apparaît dans les boues, même à petites déformations de cisaillement. Donc, la boue ne peut plus être considérée comme un matériau de consistance liquide. Elle est plutôt décrite comme un matériau de consistance plastique ou comme une pâte granulaire. Selon la classification des poudres, le comportement de la boue ayant des siccités entre 20 et 43 % peut être similaire à une poudre très cohésive dont l'écoulement est difficile.

A la fin, lorsque la siccité est supérieure à la limite plasticité, 43 %, la boue devient un matériau divisé avec un comportement similaire à une poudre très cohésive (pour des siccités entre 43 et 48 %).

		$Si_0 = 1,9 \%$	$Si_c = 6 \%$	$Si_j = Si_L = 14 \%$	$Si = 20 \%$	$Si_p = 43 \%$	$Si_m = 55 \%$
Rhéomètre rotatif conventionnel	Texture de la boue	Matériau continu					Matériau divisé
		Suspension diluée	Suspension concentrée à consistance liquide		Pâte granulaire ou plastique qui se fracture sous cisaillement		
	Forces dominantes	Forces visqueuses dominantes	Phase transitionnelle Le nombre de contacts directs entre les flocs augmente		Forces frictionnelles dominantes		
	Dilatance	Non-dilatant (aucun saut de force normale détecté)			Dilatant (saut de force normale détecté)		
	Seuil de contrainte τ_y	$\tau_y \sim \tau_{visqueuse}$	Contribution double visqueuse et frictionnelle $\tau_y \sim c \cdot \tau_{visqueuse} + (1 - c) \cdot \tau_{friction}$		$\tau_y \sim \tau_{friction}$		
Rhéomètre granulaire	Loi de comportement					Loi de Mohr-Coulomb	
	Similarité de comportement					$ff_c = 1-2$ Poudre très cohésive	$ff_c > 2$ Comme une poudre cohésive

Figure 1 : Diagramme de phase de la boue en fonction de la concentration et de son comportement rhéologique correspondant. Si_0 la siccité en dessous de laquelle aucune contrainte seuil n'est détectée. Si_c est le seuil de percolation rhéologique. Si_L et Si_j sont la limite de liquidité et la concentration de encombrement (jamming), respectivement. Si_p la limite de plasticité. Si_m la concentration maximale dérivée de l'équation d'Eilers. ff_c l'indice de fluidité.

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