Procédure expérimentale pour caractériser les boues pâteuses à hautes températures

Lors du séchage dans un sécheur par contact avec agitation (type sécheur à palettes ou sécheur à disques), la température de la boue est comprise entre 90 et 120°C [36]. Pour travailler dans des conditions de température similaires lors des mesures rhéologiques et ainsi évaluer l'impact de la température sur les propriétés rhéologiques, les boues peuvent être chauffées dans le rhéomètre.

Cependant, l'évaporation peut fortement biaiser les résultats expérimentaux. Les techniques traditionnelles recommandées dans la littérature pour contrôler l'évaporation ne se sont pas révélées très efficaces à haute température. Par conséquent, développer une procédure pour contrôler/limiter l'évaporation s'est avéré indispensable.

La procédure élaborée vise à contrôler l'évaporation à haute température en utilisant une configuration accessible à tout rhéomètre commercial. Le principe consiste à empêcher l'évaporation du solvant (l'eau dans le cas présent) en limitant le contact entre la boue cisaillée et l'environnement.

5.1. Description de la procédure pour contrôler l'évaporation de l'eau lors des mesures rhéologiques à haute température-Article 2

Ce travail a fait l'objet d'une communication orale au congrès SMICE 2018 (Rome, Mai 2018), puis a été sélectionné pour publication dans Water Science and Technology. Le document proposé ci-après est publié dans cette revue.

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Afin d'éviter le problème de la solubilisation de la matière organique à hautes températures, les boues utilisées dans cette partie sont préalablement traitées thermiquement.

How to avoid evaporation during rheological measurements of dewatered pasty sludge at high temperature

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Abstract

To control the residence time in paddle dryers and the drying efficiency, the knowledge of rheological behaviour of highly concentrated and pasty sludge and its temperature dependency is required. However, because of perturbing effects such as evaporation, rheological measurements are not fully representative of intrinsic sludge properties. Traditional techniques recommended in the literature to control evaporation are not efficient at high temperatures. This work demonstrates how to control the evaporation at high temperature using a configuration which is accessible for any commercial rheometer. The configuration concept is to prevent solvent (water) evaporation by limiting the contact between the sheared sludge and the environment. We demonstrate that this configuration allows preventing evaporation up to 80 °C at least during 2 hours. The efficiency of this configuration is confirmed at different total solid (TS) contents ranging from 20 to 47 wt.%.

Keywords: Bad data, Evaporation, High temperature, Pasty sludge, Rheology

1. Introduction

In EU, sludge production is increasing every year and is becoming a real challenge for the waste water treatment plants (WWTP) [9]. Thermal drying is one of the most commonly used operations to reduce volumes. Because of the difficulties to control the sludge flow rates in agitated dryers, a lot of energy is consumed unnecessarily [11,13,17,18]. Optimisation of the dryer energy consumption can be obtained by an accurate control of the operating parameters, among which the residence time which is directly connected to the flow rates in the dryer [11,13,59]. Controlling the residence time implies the knowledge of the rheological parameters of sludge for total solid contents (TS) higher than 20 wt.% and their temperature dependency during drying.

However, rheological measurements are hard to perform at high TS as perturbing effects appear such as fractures and evaporation [36,38,39]. Indeed, at high TS, the interactions between particles are mainly frictional ones leading to fractures under shear experiments and thus bad rheological data. In a previous work, it has been shown how to correct fractures impact at ambient temperature with a well-controlled procedure allowing the exact determination of the surface really sheared, and thus, of intrinsic rheological parameters [60].

Moreover, during a test with a long duration or at elevated temperatures, additional problems occur as solvent evaporation or sample drying. Consequently, the obtained rheological data are not fully representative of a controlled state of the sludge. To overcome this problem and minimize measurement errors, the most frequently used solutions in rotational rheometers (with plane-plane, coaxial-planar and coaxial-plane geometries) consist of applying a Newtonian oil film around the free surfaces of sludge. With such technique, measurements have been done at temperatures up to 80 °C for diluted sludge, that is TS<5 wt.% [61–63] but only 60 °C at higher concentrations, that is TS up to 16 wt.% [24,35,64]. This observation is due to the fact that rheological properties are more sensitive to solvent evaporation at high TS. However, the applicability of this method is limited at low experiment duration (lower than 20 min). Moreover, the applicability of this method is not adapted to pasty sludge, since fractures appear under shear measurements, which causes sample drainage and undesirable mass transport between the sludge and the surrounding oil layer.

The literature has underlined another solution to control the evaporation during rheological measurements consisting of keeping a saturated atmosphere during experimental tests. In that perspective, rheometer manufacturers and academic researchers have explored and tested a device consisting of an insulated sample chamber (cover) and a vapour trap to prevent heat transfer and limit the solvent transfer [65–67]. This device was implemented to study the rheological behavior of different materials, as for instance clay-polymer mixtures at ambient temperature [68,69], diluted CMC solutions (<5 wt%) at temperatures up to 80 °C [70] or pasty sludge with TS content up to 28.5 wt.% at temperatures up to 60°C [71–73]. Later, Quignon-Tosoni [74] used to keep a saturated atmosphere an apparatus that was adapted from a nebulizer to the geometry of the rheometer (coaxial cylinders) and observed no change in the viscosity of clay suspensions for 24 hours at room temperature. However, a gradual evolution in sample viscosity due to evaporation was highlighted even at 40 °C by Sato and Breedveld [75], leading to unrealistic rheological properties. Keeping a saturated atmosphere seems thus an efficient technique to prevent solvent evaporation in the case of pasty materials, but it seems to be adapted to temperatures lower than the ones used in thermal drying, typically from 90 to 120 °C.

Finally, the literature highlights the lack of techniques adapted to control the evaporation of solvent at high temperature during rheological measurements, in particular on highly concentrated suspensions such as pasty sludges. This article, therefore, aims at filling this gap by proposing a specific procedure to prevent the solvent evaporation at high temperatures during rheological measurements on pasty sludge. This allows to obtain intrinsic rheological parameters and thus a better understanding of pasty sludge behaviour in the dryer. The efficiency of this technique at high temperatures, is first validated on sludge having 20 wt.% TS, then confirmed at 28 and 47 wt.% TS.

2. Material and methods

2.1. Sludge

Pasty sludge was sampled at the waste water treatment plant from Albi city (France) at the outlet of the centrifuge. Its initial TS (standard EN 12880:2000) was 20 wt.% and the volatile solid (VS, standard EN 12879:2000) content was about 63 wt.% (of dry weight). Samples with higher TS contents have been prepared in a filtration/compression cell inserted in a hydraulic press (Carver USA). A sludge mass of 0.8 kg is pressed for 48 and 72 h at a pressure of 30 bar. The temperature of the laboratory is maintained at 20 °C.

Several authors have shown that the solubilisation of organic matter (from the solid phase to the liquid phase) at high temperature irreversibly modifies the structure of the sludge and therefore its rheology [1–4]. The time and the temperature of thermal treatment are the dominant factor influencing the organic solubilisation and thus the rheological parameters. Indeed, the organic matter solubilisation during thermal treatment, could reach a stable state within 30-60 minutes with respect to temperatures higher than 100°C [5,6] but ranged from hours to days at temperatures lower than 100°C [6–8]. Xue et al [8] and Zhang et al. [6] investigated the effects of thermal treatment on organic matter solubilisation on pasty sludge (TS around 17 %). They showed that the solubilisation of the organic matter increases as function of time but tends toward a stable state at around 24 hours of thermal treatment at temperature between 60 and 90°C.

Thus, to avoid the problem of solubilization during rheological tests at high temperatures, the samples are pretreated thermally at 90°C for at least 24 hours before the measurements.

The TS and VS contents have been determined before and after the thermal treatment to check that this treatment did not alter the sludge. The table 1 shows the result for the initial sludge.

Table 1. Measurement of TS and VS contents before and after the thermal treatment.

	Before thermal treatment	After thermal treatment
TS content (wt.%)	20.3	20.4
VS content (wt.% TS)	63.2	63.1

Then, the sludge obtained is mixed smoothly by hand and then stored at 4 °C to minimize impact of biological activity.

2.2. Rheological measurements

Rheological measurements are performed with a stress-controlled rheometer (HAAKE RheoStress 600, Thermo Scientific, Germany). The upper part supplies measurements, while the lower part is fixed. Two configurations described thereafter are implemented.

A constant dynamic strain (γ=0.3 %) in the linear viscoelastic range (LVE) is applied. Viscous modulus G", elastic modulus G' and loss tangent Tan δ (viscous to elastic modulus ratio) are tracked during enough time with 1 measurement every 10 seconds. This will help at evaluating the evaporation kinetics of sludge, and hence, at defining the appropriate conditions to prevent evaporation.

2.2.1. Plate-plate configuration

The geometry consists of a classic serrated plate-plate with a 35 mm diameter (Fig. 1). The gap is kept constant at 2 mm. A peltier temperature controller is connected to the lower plate. To prevent evaporation, measurements were carried out in a vapour saturated medium by using a cover and a vapour trap (Fig. 1). The vapour trap is connected to the shaft of the upper plate. Its principal role is to provide a tight seal between the shaft and the cover. A ring is mounted directly onto the cover: when the cover is placed, the ring lowers into the solvent in the vapour trap. The water on the lower plate is acting as liquid to saturate air trapped in the chamber (under the cover). It can contain up to 2 ml of water which is sufficient to saturate the air even at 80 °C. For example, based on humid air material balance calculi, the quantity of water needed to saturate the chamber at 80 °C is close to 0.5 ml. As a result, the chamber will be maintained saturated. Thereafter, only experiments without the vapour trap will be notified (no protection).

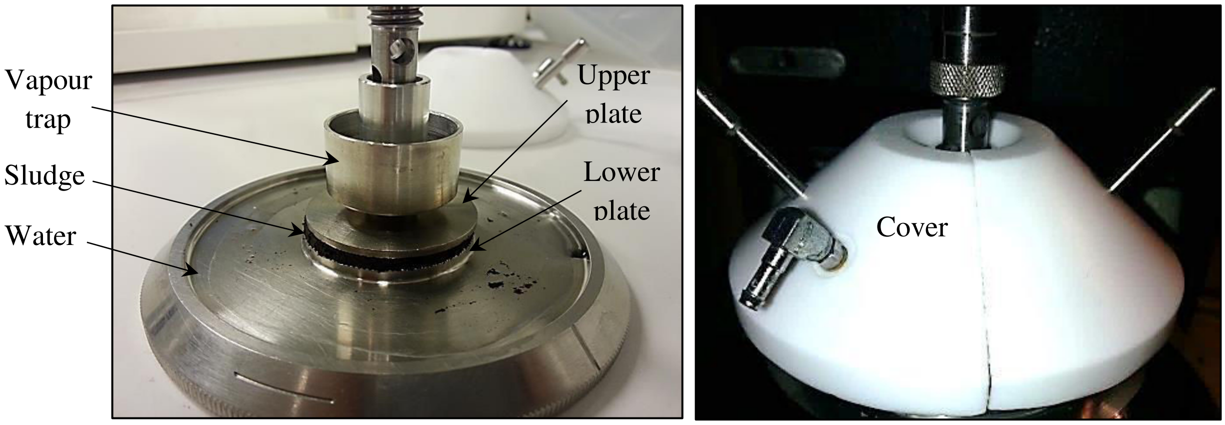

Fig. 1–Plate-plate configuration used to prevent evaporation.

2.2.2. Plate-cylinder configuration

The geometry consists of a serrated upper plate (35 mm diameter) coupled with a lower cylinder (36.88 mm inner diameter and 50 mm depth; see Fig. 2). A temperature-regulated bath is connected to the lower cylinder. The sludge sample (at a constant volume each experiment) is introduced into the measuring cylinder (Fig. 2-a). Then the upper plate is placed so as to form a sludge ring around the upper plate of height h=1.5 mm (Fig. 2-b). This step aims at limiting the contact between the sheared sludge and the environment. The upper plate is turned around ϕ=2 rad (Fig. 2-b) to eliminate residual stresses generated by the ring of sludge. Because of the latter step, the sample may not adhere correctly to the lower surface of the upper plate. To ensure this contact, the upper plate is lowered again of 0.5 mm: the new height of the sludge ring is thus h+0.5 mm (Fig. 2-c).

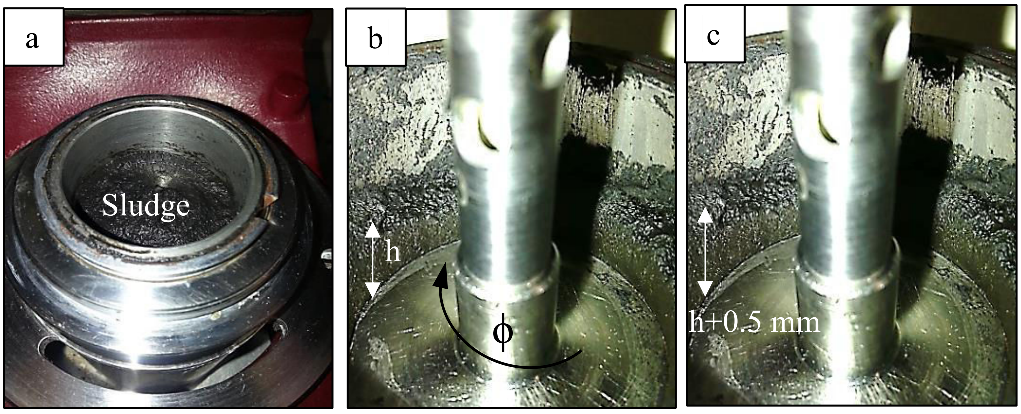

Fig. 2–Plate-cylinder configuration used to prevent evaporation.

Finally, to improve the protection against evaporation, measurements are carried out in a vapour saturated medium by using the vapour trap and the cover.

The TS contents of sludge is estimated prior and at the end of each test. For the plate-plate configuration, the whole sample between the measuring tools is extracted (Fig. 3-a). For the plate-cylinder configuration, only part of sample from where the upper plate was placed is extracted (Fig. 3-b).

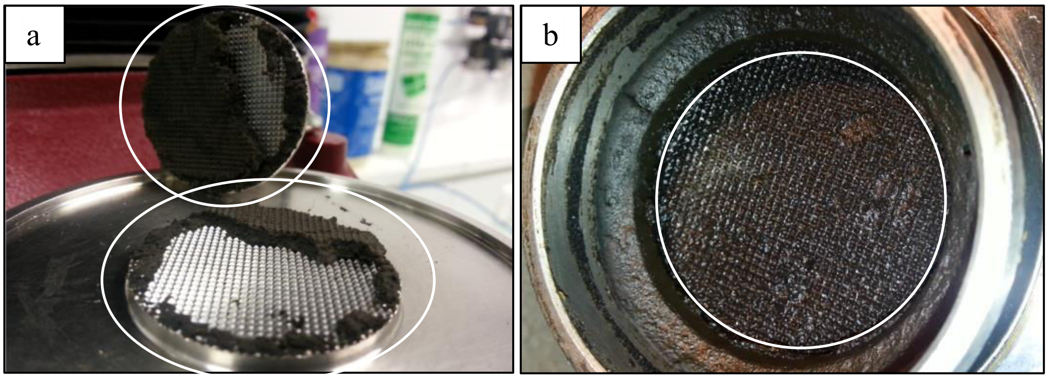

Fig. 3–The place where the sludge is extracted for TS content estimation. Plate-plate configuration (a) and plate-cylinder configuration (b).

3. Results and discussion

3.1. Elastic modulus or loss tangent?

Fig. 4 presents the evolution of the dimensionless elastic modulus (G'/G'_0 where G'_0 is the initial value) and the loss tangent (Tan δ) as function of time for three repeated tests on a sludge having the same TS content of 20 wt.%. The elastic modulus curves present the same shape but with a difference between measurements for times larger than 500 seconds. However, for the same tests, the loss tangent curves are stable and identical during all the time of experiment. All the tests show very good repeatability and reliability. This fact is notably due to the great sensitivity of the elastic modulus to sample preparation but also to the loading of sludge in the measurements device. This is true especially at very low strains as in our case. Therefore the elastic modulus cannot be used as a reference to track the evolution of solvent evaporation in this study. The tracking of the loss tangent is thus more relevant to verify whether the solvent present in the sludge evaporates or not.

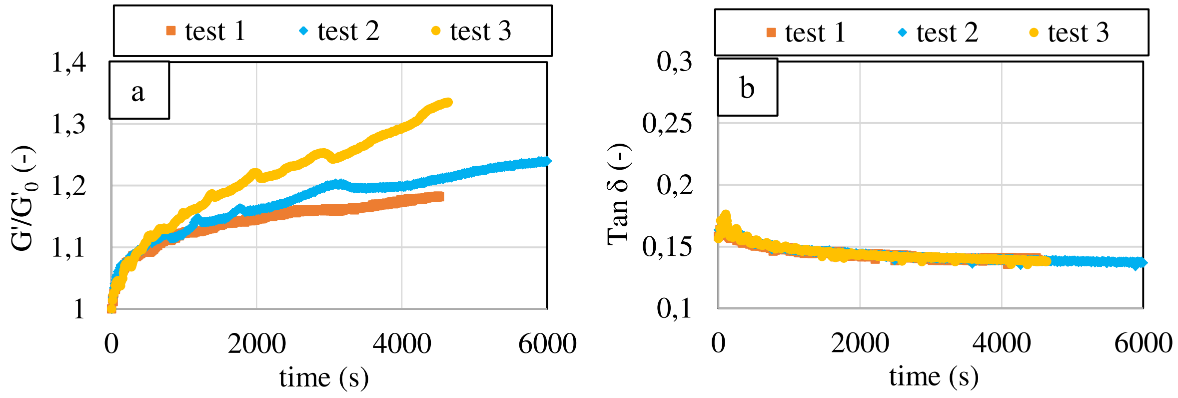

Fig. 4–Evolution of the dimensionless elastic modulus (a) and the loss tangent (b) as function of time under a constant strain (γ=0.3 %) for 20 wt.% TS sludge. G'_0 is the initial value of the elastic modulus G'.

3.2. Plate-plate configuration

The table 2 presents the increase in TS content at the end of experiment using the plate-plate configuration for sludge having initially a TS of 20 wt.%. Without protection, the evaporation takes place even at ambient temperature as sludge surfaces are free and in contact to an unsaturated atmosphere. This results in a TS increase of 19 % after 3 hours of experiment. For the same experiment duration at ambient temperature, no change in concentration (negligible increase of 0.2 %) is detected in a saturated atmosphere using the cover and the vapour trap. At a higher temperature of 80 °C, because of evaporation, a crusty material is formed on the free edge (Fig. 5) and as expected, the TS content of the sludge increases by 13 % after only 10 minutes of experiment. These results suggest that the combination of cover and vapour trap control the evaporation of solvent at ambient temperature over long periods of time, but is insufficient to perform measurements at high temperatures.

Table 2: TS increase for the plate-plate configuration for 20 wt.% TS sludge.

Protection	Temperature	Time of experiment	TS increase (wt.%)
no	20 °C	3 hours	19
yes	20 °C	3 hours	0.2
yes	80 °C	10 minutes	13

Fig. 5–Picture of the 20 wt.% TS sludge at temperature of 80°C after 10 minutes of experiment.

Fig. 6 presents the evolution of the loss tangent as function of time using the plate plate configuration under different conditions. Without protection at ambient temperature, the tangent loss drops significantly because of the sludge TS increase by 19 %. Using the cover and the vapour trap at ambient temperature, the loss tangent decreases until a critical time of about 1000 seconds (Fig. 5), indicating a restructuration is occurring and the material is becoming more and more elastic [57,60]. Then, after 1000 seconds the loss tangent tends toward a plateau highlighting a stable behaviour. In fact, sludge is mainly made of water and organic polymers: during shear, there is a strong competition between colloidal forces which tend to rebuild the solid structure (physical aging) and hydrodynamic forces which tend to maintain the solid structure broken. There is a critical strain γ_c below which the solid structure rebuilds even under shear [57]. This critical strain is without doubt higher than the strain applied in this study γ=0.3 %, that is why sludge becomes more elastic. At 80 °C, the formation of the crusty material on the free edge leads to instabilities in the loss tangent throughout the test. It confirms that trapping saturated vapour inside the sample chamber is not sufficient to prevent sample evaporation at high temperature.

The time-dependency of sludge rheological characteristics makes the estimation of the evaporation kinetic difficult during the first 1000 seconds. Therefore, to be able to estimate the evaporation kinetic, only the part of measurement where time is higher than 1000 seconds, is considered, i.e., when the sludge behaviour is stable.

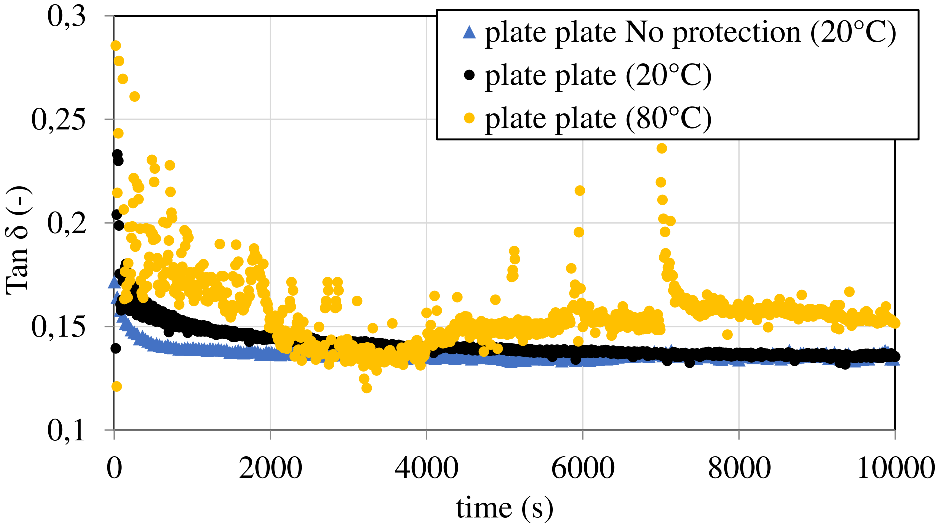

Fig. 6–Evolution of loss tangent as function of time under a constant strain (γ=0.3 %) for 20 wt.% TS sludge.

3.3. Plate-cylinder configuration

3.3.1. Validation test

Data of the plate-cylinder configuration are compared with those obtained using the plate-plate configuration at ambient temperature and in a saturated atmosphere (Fig. 7). During the first 1000 seconds, the sludge behaviour is impacted by the restructuration. Then, beyond 1000 seconds, all curves become superimposed and identical. The important noise previously observed in Fig. 5 at 80 °C with plate plate configuration disappeared.

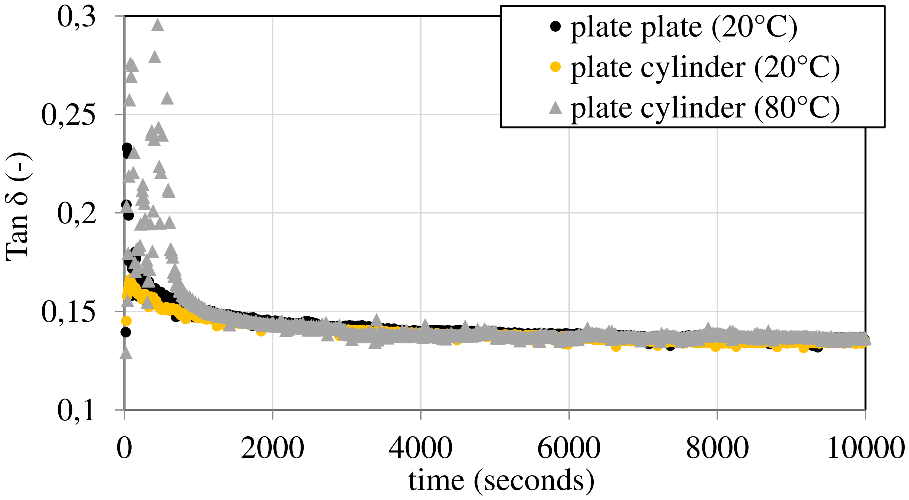

Fig. 7–Evolution of loss tangent as function of time under a constant strain (γ=0.3 %) for sludge with 20 wt.% TS.

Results in table 3 show that TS contents of the sheared sludge is kept constant during at least 3 hours confirming an efficient control of the evaporation phenomenon. Moreover, when the measuring tools are removed away, the texture of the sheared sludge looks like soft and shiny, while the sludge surrounding the plate (the ring of sludge) seems hard and crusty playing therefore the role of a protection layer (Fig. 8). Finally, this allows to validate the effectiveness of the plate cylinder configuration to prevent evaporation up to 80 °C.

Table 3: TS contents evolution for the plate cylinder configuration as function of time at 80 °C for 20 % TS sludge.

Time of experiment (h)	TS content (wt.%)
0	20.2
3	20.2
5	19.9
10	19.2

Fig. 8–Picture of the sludge after 3 hours of experiment at 80 °C for 20 wt.% TS sludge.

To evaluate the effectiveness of the plate cylinder configuration at temperatures higher than 80 °C, the experiment was repeated for several times at a more elevated temperature that is 90°C (Fig. 9). Large fluctuations are highlighted in data all along of experiment probably due to evaporation. It seems thus not possible to obtain reliable measurements for temperatures higher than 80 °C.

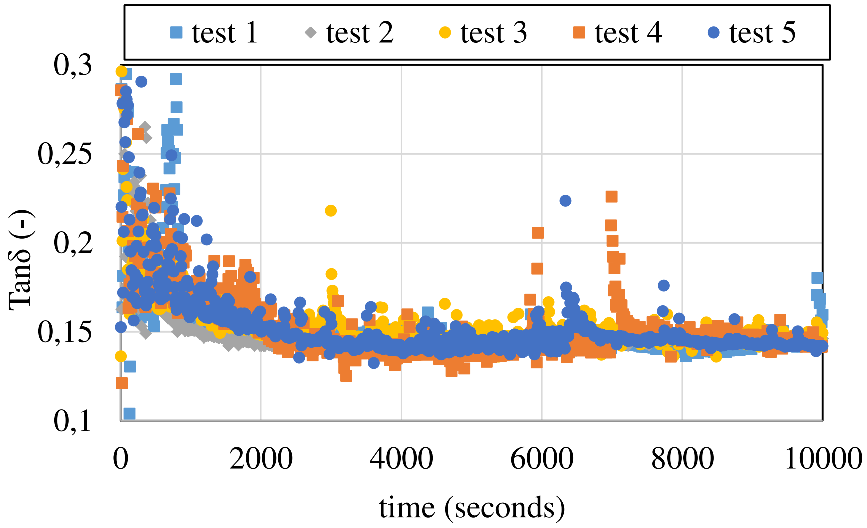

Fig. 9–Evolution of loss tangent as function of time under a constant strain (γ=0.3 %) at 90 °C using a plate cylinder configuration for 20 wt.% TS sludge.

3.3.2. Variation of TS contents at 80 °C

As a second practical example, the behaviour of sludge at higher concentrations (28 and 47 wt.% TS) is investigated at 80 °C. Because rheological properties are very sensitive to solvent evaporation at very high concentration, measurements are performed only for 6000 seconds (Fig. 10).

As expected, during the first 1000 seconds, the loss tangent signal is dramatically noisy due to inertia and transient conduction. Then, at higher durations, whatever the TS content, curves are identical and tend toward a plateau highlighting a stable behaviour and indicating the absence of evaporation.

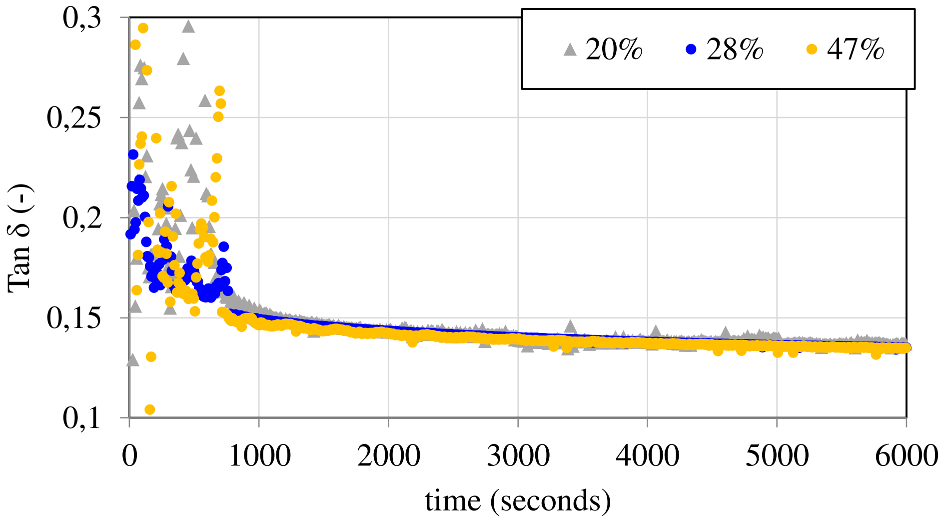

Fig. 10–Loss tangent evolution as function of time under a constant strain (γ=0.3 %) at 80 °C for different TS contents.

This configuration allows to prevent the evaporation whatever the temperature ranging from 20 to 80 °C and thus to keep a constant state of the sludge under rheological measurements. The door is now open to define how rheological parameters of pasty sludge evolve with temperature and thus to identify a kinetic function regarding temperature changes during the drying process.

4. Conclusion

The first part of this work demonstrates the difficulties in performing reliable rheological measurements due to the sensitivity of the elastic modulus to sample preparation and loading in the rheometer. However, the loss tangent exhibits very good repeatability and reliability and is thus used as a reference to track the evolution of solvent evaporation.

The next part shows how evaporation in rotational rheometry can be controlled, by implementing a simple configuration compatible with any commercial rotational rheometer. The configuration consists in an upper plate and a lower cylinder. It is based on the limitation of the contacts between the sheared sludge and the surrounding gaseous environment. It is shown that this configuration allows preventing evaporation whatever the temperature up to 80 °C for TS contents ranging from 20 to 47 wt.%.

The next step of this work will aim to control both fractures and evaporation at high temperatures in order to obtain realistic rheological parameters of pasty sludges. This leads us to understand how sludge evolves in the dryer and thus to control residence time and drying efficiency.

5.2. Résumé

Des essais préliminaires ont démontré que les techniques recommandées dans la littérature pour limiter/contrôler l'évaporation à hautes températures pendant les mesures rhéologiques ne sont pas efficaces pour des boues très concentrés chauffées.

Nous avons proposé, dans ce travail, l'utilisation d'une géométrie simple, compatible avec tout rhéomètre rotatif conventionnel et démontré avec succès son efficacité sur une large plage de température (20-80°C) et de siccité (20-47 %). Le suivi de la cinétique d'évaporation est réalisé en appliquant une faible déformation en mode dynamique et en suivant l'évolution de l'angle de perte. La méthode semble limitée pour décrire le comportement rhéologique dans le sécheur où la température de la boue varie entre 100 et 120°C.

6 Conclusion du chapitre

Dans ce chapitre, nous avons présenté les différents pré-traitements appliqués aux boues pour disposer d’une large gamme de concentrations, allant de 2 à 48 %. Cette plage de siccité inclut les différents états de la matière (liquide, pâteuse/plastique et granulaire-divisé) susceptibles d’être rencontrés dans la file boue d’une station d’épuration.

Nous avons présenté les deux techniques de caractérisation utilisées pour caractériser le comportement rhéologique des boues liquides, plastique et déshydratées à consistance granulaire. Nous avons également présenté, sous formes d’articles, les deux procédures expérimentales développées sur le rhéomètre conventionnel pour étudier le comportement rhéologique des boues pâteuses. La première procédure vise à corriger l’impact de la fracturation du milieu. La deuxième procédure permet de limiter/contrôler l’évaporation jusqu’à 80°C. Inspiré de la littérature sur la rhéologie des matériaux granulaires, nous avons également présenté la procédure qui permet de caractériser le comportement rhéologique des boues déshydratées à consistance granulaire.

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