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

M. Mouzaoui, J. C. Baudez, M. Sauceau and P. Arlabosse

ABSTRACT

Controlling the residence time in paddle dryers and the drying efficiency imply the knowledge of rheological behaviour of highly concentrated and pasty sludge and its temperature dependency. However, because of perturbing effects such as evaporation, measurements are not fully representative of intrinsic sludge properties. Classical techniques usually considered in the literature for evaporation control are not efficient at high temperatures. This work gives a method to control the evaporation at high temperature that can be used with any commercial rheometer. The configuration concept is to prevent water loss by limiting the contact between the sheared sludge and the environment. This configuration allows preventing evaporation up to 80 °C at least during 2 h. Its efficiency is confirmed at different total solid (TS) contents ranging from 20 to 47 wt.%.

Key words | bad data, evaporation, high temperature, pasty sludge, rheology

M. Mouzaoui (corresponding author)
M. Sauceau
P. Arlabosse
Université de Toulouse; Mines Albi; CNRS; Centre RAPSODEE,
Campus Jarlard, F-81013 Albi,
France
E-mail: *mouzaouimohamed1@gmail.com*

M. Mouzaoui
Irstea, UR TSCF, Domaine des Palaquins,
F-03150 Montoldre,
France

J. C. Baudez
IMT Lille Douai, Direction de la Recherche et de l'Innovation,
20 Rue Guglielmo Marconi,
59650 Villeneuve-d'Ascq, France

INTRODUCTION

In the EU, sludge production is increasing every year and is becoming a real challenge for the waste water treatment plants (WWTP) (Fytili & Zabaniotou 2008; Eshtiaghi *et al.* 2013; European Commission 2017). Thermal drying is one of the most commonly used operations to reduce volumes but part of the energy is unnecessarily consumed due to not optimised process (Chabrier 2007; Arlabosse *et al.* 2012; Charlou *et al.* 2015; Milhé *et al.* 2015). The dryer energy consumption can be optimised by an accurate control of the operating parameters, among which the residence time distribution which is directly linked to the flow rates in the dryer (Djerroud 2010; Arlabosse *et al.* 2012; Charlou *et al.* 2015). Controlling the residence time distribution implies the knowledge of the main rheological parameters of sludge especially for total solid (TS) contents 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 (Baudez & Coussot 2001; Chaari *et al.* 2003; Charlou 2014). Indeed, at high TS, the interactions between particles are mainly frictional leading to fractures during shear, inducing bad rheological data. In a previous work, it has been shown how to correct fracture impact at ambient temperature with a well-controlled procedure allowing the exact determination of the surface really sheared and, thus, of intrinsic rheological parameters (Mouzaoui *et al.* 2018).

During a long duration or high temperature tests, additional problems occur such as water loss and sample drying. Consequently, the measured 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 in applying a Newtonian oil film around the free surfaces of sludge. With such a technique, measurements have been done at temperatures up to 80 °C for diluted sludge, that is TS < 5 wt.% (Ortiz *et al.* 1994; Briscoe *et al.* 1998; Baudez *et al.* 2013b) but only up to 60 °C at higher concentrations, that is TS up to 16 wt.% (Baudez *et al.* 2013a; Jiang *et al.* 2014; Ségalen 2015). This observation is due to the fact that rheological properties are more sensitive to water evaporation at high TS. However, the applicability of this method is limited at low experiment duration (lower than 20 min) and is not adapted to pasty sludge, since fractures appear under shear

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measurements, leading to 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. Attachments/additional pieces to minimize the evaporation effects have developed in that perspective. For instance, consisting of an insulated sample chamber (cover) and a vapour trap (Gans *et al.* 1999; Nommensen *et al.* 1999; Ksapabutr *et al.* 2004). This method has been implemented in several works, as for clay-polymer mixtures at ambient temperature (Benchabane 2006; Ebagninin 2009), diluted carboxymethyl cellulose (CMC) solutions (<5 wt.%) at temperatures up to 80 °C (Benslimane 2012) or pasty sludge with TS content up to 28.5 wt.% at temperatures up to 60 °C (Dieudé-Fauvel *et al.* 2009; Hammadi *et al.* 2013; Feng *et al.* 2014). Later, to keep a saturated atmosphere, Quignon-Tosoni (2015) has used an apparatus adapted from a nebulizer to the geometry of the rheometer (coaxial cylinders) and observed no change in the viscosity of clay suspensions for 24 h at room temperature. However, a gradual evolution in sample viscosity due to evaporation was highlighted even at 40 °C by Sato & Breedveld (2005), leading to unrealistic rheological properties. Keeping a saturated atmosphere thus seems an efficient technique to prevent water 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.

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 (Pevere *et al.* 2009; Appels *et al.* 2010; Farno *et al.* 2015, 2014). 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 min with respect to temperatures higher than 100 °C (Carrère *et al.* 2010; Zhang *et al.* 2017) but ranged from hours to days at temperatures lower than 100 °C (Climent *et al.* 2007; Xue *et al.* 2015).

Finally, the literature highlights a lack of techniques adapted to control the evaporation of water at high temperature during rheological measurements, especially for on highly concentrated suspensions such as pasty sludges. This paper aims to fill this gap by proposing a specific procedure to prevent the water 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.

MATERIAL AND METHODS

Sludge

Pasty sludge was sampled at the WWTP from Albi city (France) at the outlet of the centrifuge. It is produced from extendedly aerated, thickened and digested municipal wastewater. 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.

To avoid the problem of solubilisation during rheological tests, samples were thermally pre-treated at 90 °C during 24 (in a hermitic container) prior to 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. 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

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: plate-plate and plate-cylinder configurations.

A constant dynamic strain ($\gamma = 0.3\%$) in the linear visco-elastic range (LVE) is applied. Viscous modulus G', elastic modulus G' and loss tangent Tan δ (viscous to elastic modulus ratio) are recorded over 3 h. This helps at evaluating the evaporation kinetics of sludge, and hence, at defining the appropriate conditions to prevent evaporation.

The TS contents of sludge is measured prior and at the end of each test. For the plate-plate configuration, the whole sample between the measuring tools is extracted while, for the plate-cylinder configuration, only part of the sample below the upper plate is extracted.

Plate-plate configuration

The geometry consists of a classic serrated plate-plate with a 35 mm diameter (Figure 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 (Figure 1). The cover (made of Teflon) and the vapour trap (made of steel) are manufactured by Thermo Scientific. 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 water in the vapour trap. The water on the lower plate is used 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. Thereafter, only experiments without the vapour trap will be notified (no protection).

Plate-cylinder configuration

The geometry consists in a serrated upper plate (35 mm diameter) coupled with a lower cylinder (36.88 mm inner diameter and 50 mm depth; see Figure 2). A temperature-regulated bath is connected to the lower cylinder. The sludge sample (at a constant volume for each experiment) is introduced into the measuring cylinder (Figure 2(a)). Then the upper plate is moved down in the sample so as to form a sludge ring around the upper plate of height h = 1.5 mm (Figure 2(b)). This step aims at limiting the contact between the sheared sludge and the environment. The upper plate is turned around $\phi = 2$ rad (Figure 2(b)) to eliminate residual stresses generated by the surrounding sludge ring. 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 by 0.5 mm: the new height of the sludge ring is thus h + 0.5 mm (Figure 2(c)).

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

RESULTS AND DISCUSSION

Pre-evaluation tests

Figure 3 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 triplicate using plate-plate configuration at 20 °C on a sludge at TS content of 20 wt.%. The elastic modulus curves present the same shape but with a difference between measurements for times longer than 500 seconds. However, for the same tests, the loss tangent curves are stable and identical during the whole test. 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 especially true at very low strains as in our case.

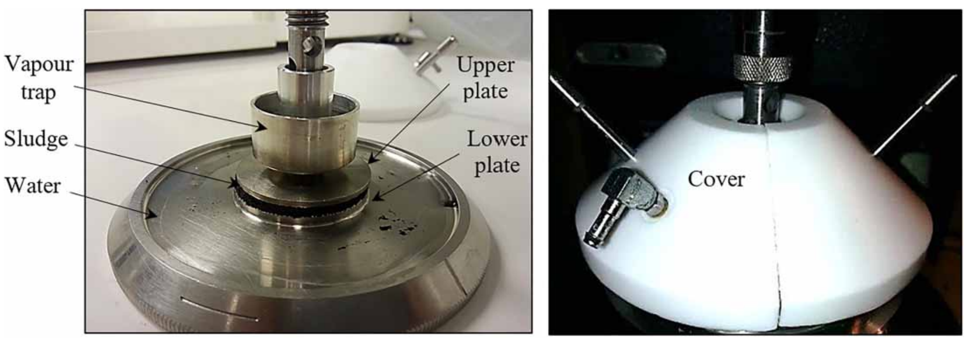

Figure 1 | Plate-plate configuration used to prevent evaporation.

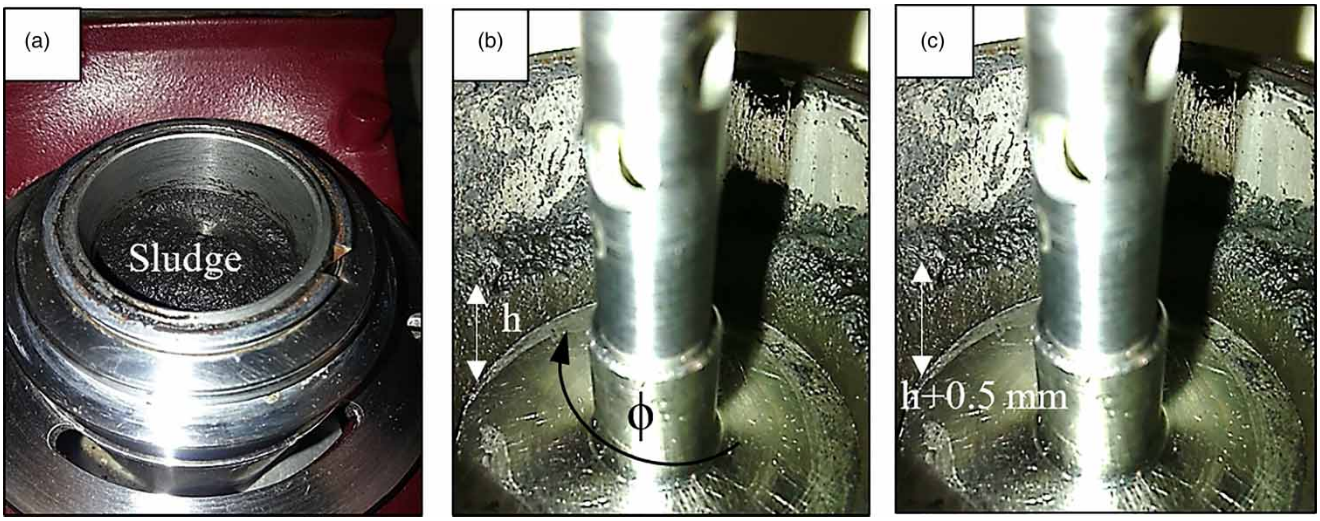

Figure 2 | Plate-cylinder configuration used to prevent evaporation.

Therefore, the elastic modulus cannot be used as a reference to track the evolution of water evaporation in this study. The tracking of the loss tangent is thus more relevant to verify whether the water present in the sludge evaporates or not.

Plate-plate configuration

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 from sludge free surfaces and in contact with an unsaturated atmosphere. This results in a TS increase of 19% after 3 h of experiment at 20 °C and of nearly 100% after only 10 min of experiment at 80 °C. For a 3 h 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 and as expected, the TS content of the sludge increases by 13% after only 10 min of experiment.

These results suggest that the combination of cover and vapour trap control the evaporation of water at ambient temperature over long periods of time, but is insufficient to perform measurements at high temperatures.

Figure 4 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 1,000 seconds, indicating that a restructuration is occurring and the material is becoming more and more elastic (Baudez 2008; Mouzaoui *et al.* 2018). Then, after 1,000

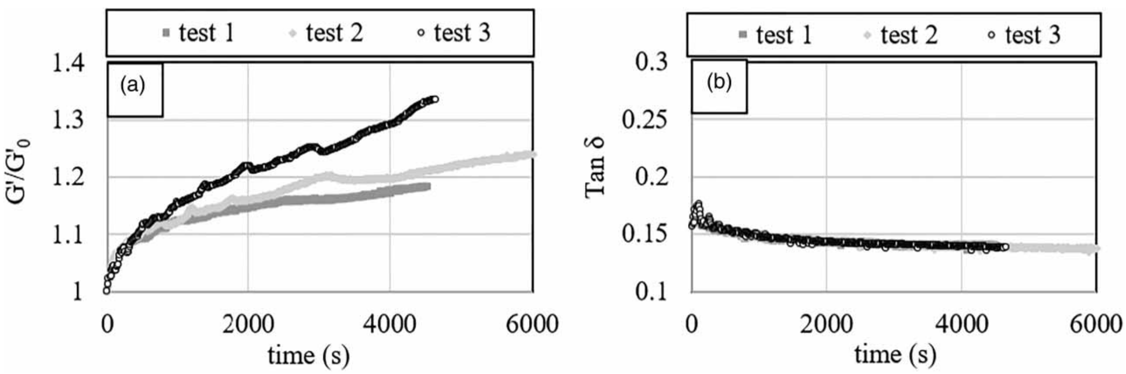

Figure 3 | Evolution of the dimensionless elastic modulus (a) and the loss tangent (b) as function of time under a constant strain ($\gamma = 0.3\%$) for 20 wt.% TS sludge. G_0' is the initial value of the elastic modulus G′.

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 h	19
yes	20 °C	3 h	0.2
yes	80 °C	10 min	13

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 broken solid structure. It results in a critical strain γ_c below which the solid structure rebuilds even under shear (Baudez 2008). This critical strain is without doubt higher than the strain applied in this study $\gamma = 0.3\%$, 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 1,000 seconds. Therefore, to be able to estimate the evaporation kinetic, only the part of measurement where time is higher than 1,000 seconds is considered, i.e. when the sludge behaviour is stable.

Plate-cylinder configuration

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 (Figure 5).

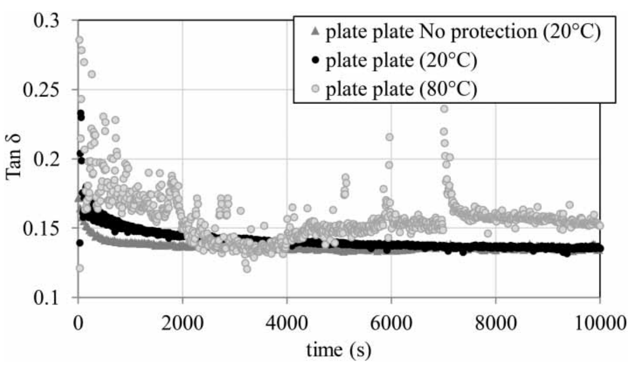

Figure 4 | Evolution of loss tangent as function of time under a constant strain ($\gamma = 0.3\%$) for 20 wt.% TS sludge.

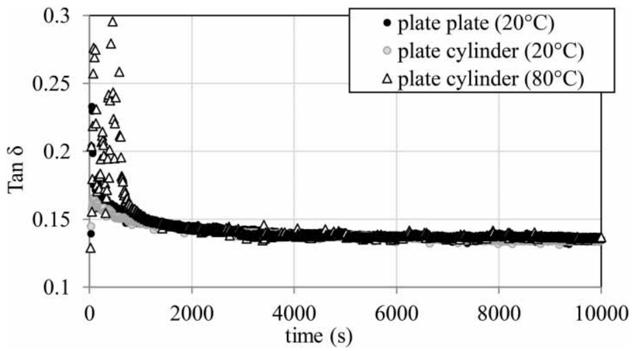

Figure 5 | Evolution of loss tangent as function of time under a constant strain ($\gamma = 0.3\%$) for sludge with 20 wt.% TS.

During the first 1,000 seconds, the sludge behaviour is impacted by the restructuration. Then, beyond 1,000 seconds, all curves become superimposed and almost identical. The important noise previously observed in Figure 4 at 80 °C with plate-plate configuration is weaker.

Results in Table 3 show that TS contents of the sheared sludge is kept constant during at least 3 h confirming an efficient control of the evaporation phenomenon. Moreover, when the measuring tools are removed away, the texture of the sheared sludge looks soft and shiny, while the sludge surrounding the plate (the surrounding ring of sludge) seems hard and crusty, therefore playing the role of a protection layer (Figure 6). Finally, this allows to validate the effectiveness of the plate cylinder configuration to prevent evaporation up to 80 °C.

To evaluate the effectiveness of the plate cylinder configuration at temperatures higher than 80 °C, the experiment was repeated several times at a more elevated temperature that is 90 °C (Figure 7). Large fluctuations in data are highlighted all along the experiment probably due to evaporation. It thus seems not possible to obtain reliable measurements for temperatures higher than 80 °C.

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

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

Time of experiment	TS content (wt.%)
0 h	20.2
3 h	20.2

Figure 6 | Picture of the sludge after 3 h of experiment at 80 °C for 20 wt.% TS sludge.

to water evaporation at very high concentration, measurements are performed only for 6,000 seconds (Figure 8).

As expected, during the first 1,000 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.

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 during 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.

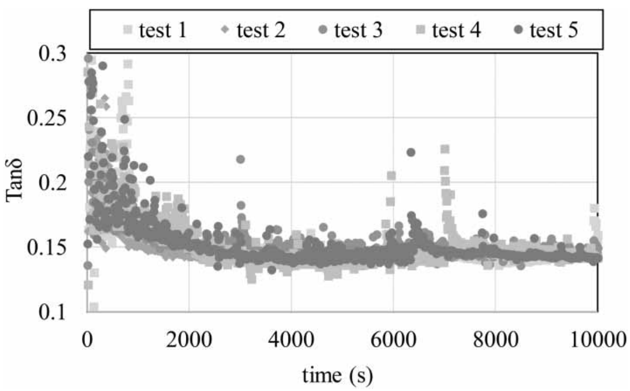

Figure 7 | Evolution of loss tangent as function of time under a constant strain ($\gamma = 0.3\%$) at 90 °C using a plate cylinder configuration for 20 wt.% TS sludge.

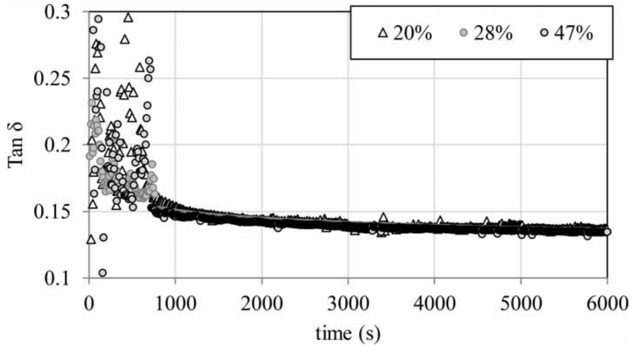

Figure 8 | Loss tangent evolution as function of time under a constant strain ($\gamma = 0.3\%$) at 80 °C for different TS contents.

CONCLUSION

The first part of this work demonstrates the difficulties to perform 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 water evaporation.

The second part shows how evaporation can be controlled in rotational rheometry, by implementing a simple configuration compatible with any commercial rotational rheometer. This 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 has been 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.

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