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Method Article

Impact of methodological artifact on digested sludge flow curve measurement

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ABSTRACT

Inconsistent experimental procedures have been used to characterize sludge rheology in literature. This often has resulted in proposing different rheological models for sludge as well as non-comparable data. Any collected rheological data needs to be interpreted considering the methodology used for its collection because otherwise they cannot be used by engineers for design and troubleshooting. This paper intends to shed light on the influential parameters during data collection procedure to produce a reliable and reproducible data.

This paper systematically investigates the impact of different geometries, preshear, equilibrium, rest and storage time on flow curve measurement for digested sludge in the range of 2.3 to 6% total solid and recommends a reliable procedure for reproducible sludge flow curve measurements. While the magnitude of impacts is different, we found all factors are significantly dependent on the sludge solid concentration. Besides, the method of the development of the protocol can be utilized to develop appropriate protocols for rheological characterization of any other sludge. The customization highlights are:

- Selecting the geometry according to the sludge solid concentration.
- Allocating an equilibrium time at each point of flow curve according to the sludge solid concentration.
- Flow curve data requires to visually inspected for instabilities.

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Specifications Table

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Method name	Sewage sludge flow curve measurement
Name and reference of original method	Nil.
Resource availability	https://figshare.com/articles/A_reliable_protocol_for_the_flow_curve_measurement_of_digested_sludge/6960023

Background

Sludge flow curve obtained by rheological measurement provides the most critical information on the design of sludge transportation systems. For non-Newtonian sludge with complex rheology, the flow curve data is fitted commonly with nonlinear model equations to obtain the fitting parameters which then used within the appropriate pipe flow model to estimate the design parameters (i.e., friction losses). Due to nonlinear nature of the rheological models as well as the pipe flow model, the whole procedure is quite sensitive in relation to the quality of the flow curve data which in details elaborated in Farno et al. (2018).

However, there are wide discrepancies between reported rheological data presented in literature due to using different rheological measurement procedures (Rheometry technique) (Ratkovich et al., 2013). Besides, the rheometry techniques are often missing from sludge literature (Eshtiaghi et al., 2013; Ratkovich et al., 2013) making it hard to assess the data quality. Common flaws and mistakes in sludge rheological measurements are highlighted as follows:

The geometry used in the rheometer for the data collection is one of the most critical information which can result in an invalid data collection. (Seyssiecq et al., 2003). Despite the importance of the geometry selection, there is no established guideline to follow for the geometry selection in sludge rheology. The most common geometry used in sludge flow curve measurement is concentric cylinders (CC), which is suitable for fluid samples because of higher surface area and better temperature control. Concentric cylinders geometry also prevents sample flowing out of the shear gap (Mezger, 2006). However, as sludge can become pasty with strong solid-like behavior depends on the solid concentration, concentric cylinders may not be always a right choice. In this case, a parallel plates geometry is more suitable (Mouzaoui et al., 2018). The fluidic sludge (sludge in liquid to pasty state) may be determined by slump test (Baudez et al., 2002) (ASTM D 4318) which shows whether the sludge sample flow under its weight by gravity or not (Liang et al., 2017). Besides, both concentric cylinders and parallel-plate geometries with smooth walls are vulnerable to wall slippage (Baudez, 2006), a measurement artifact causing the underestimation of viscosity. Generally, slippage occurs in suspensions measurement due to formation of a thin layer of low-viscosity fluid near the wall after migration of particles to the bulk of the fluids. Slippage is more prominent with concentrated suspensions as the difference between the viscosity of the slipping layer and the bulk of the fluid is more significant (Barnes, 1995). Despite, slippage has been widely reported in the characterization of sludge rheology, several literature did not report their methodology for avoiding the slippage during the sludge flow curve measurements (Ratkovich et al., 2013). Slippage can be prevented by using geometry with rough walls or serrated walls (e.g., vane-and-rough cup). However, these tools may limit the highest measurable shear rate compared to the smooth geometries as the roughness of geometries creates vortices inside the measurement geometry at high shear rates. These vortices impose additional stresses to viscous shear stresses and because of that the viscosity is overestimated (Mezger, 2006).

The gap size of a measurement geometry is another important factor in the accurate sludge rheological measurement (Mori et al., 2006, 2008). Generally, the choice of narrow gap geometry (normally -1 mm) is more appropriate particularly for material with yield stress because the calculation of the shear rate in narrow gap geometry is simpler and more accurate. Nevertheless, the size of the geometry gap should be 10 times larger than the largest particles of sludge which depends on the solid concentration and sludge type. Because of that, it is not always possible to use narrow gap geometries if there are particles larger than 1 mm size (Dick and Ewing, 1967). Choosing between narrow or wide-gap geometries as well as smooth, sandblasted or serrated geometries depends on the sample specifications (Mezger, 2006; Seyssiecq et al., 2003). Literature suggests that sludge physical specification such as particle size varies broadly with sludge origin, process and concentration (Ratkovich et al., 2013). Dick and Ewing (1967) performed a comprehensive equipment analysis for sludge application and noted that the narrow gap geometry was not suitable for rheological characterization of waste activated sludge. They proposed gap size at least ten times larger than sludge particles to ensure the device is sensitive enough to measure low viscosity substance. The minimum particle size to be measured in a parallel-plate geometry is suggested to be one-fifth of the gap size (Mezger, 2006). However, the rules of thumb for selecting suitable geometry size and roughness are useless for sludge because sludge is composed of aggregations called flocs. Sludge flocs form different sizes which varies according to the sludge type, concentration and even imposed shear rate.

A flow curve is the curve of shear stress and shear rate measured under steady-state conditions (Coussot, 2005). The shear rate ramp test (increasing or decreasing shear rate gradually) is the classic test to determine the flow curve. What is mostly ignored during the flow curve measurement is the fact that most materials with yield stress show a level of rheological time-dependency (Mewis and Wagner, 2011). Because a flow curve must be measured under steady state, all time-dependent behaviors (e.g., thixotropy) as well as the measurement artifact need to be removed to obtain reliable results. This is achieved by customizing the measurement procedure for a material. According to literature (Baudez et al., 2011; Feng et al., 2016; Mezger, 2006), an appropriate flow curve measurement procedure should include the following steps: 1. Preshear intervals—a constant shear rate that is usually applied to the sample before the test to homogenously distribute the sample in the shear gap and remove the sample shear history (Coussot, 2005). Several researchers (Baudez, 2008; Markis et al., 2014) showed that sewage sludge rheology is subjected to change with the shear history. Therefore, it is essential to preshear sludge for sufficiently long time at the highest shear rate of the actual test to ensure the material memory is removed to enable reproducibility of results. 2. Rest intervals—rest interval is performed by presenting a constant shear rate of zero for a specific time to enable the relaxation of the sample either after a preshear interval or at the start of the test (Mezger, 2006). Rest intervals help temperature equilibrium of the samples before performing the test. Another application for the rest intervals is that by keeping a constant rest time for different samples, all samples are allowed to rebuild their microstructural networks (which was entirely broken during the preshear intervals) to the same state which helps with reproducibility of results. Also, by controlling the rest time one may simulate the mechanical history sludge has undergone in the actual process. Generally, one obtains a more reproducible result for sludge flow curve by following the preshear - rest procedure (Baudez, 2008). 3. Equilibrium time—an appropriate equilibrium time for collecting data at each data point of the flow curve allows sludge to reach a steady state condition. This is vitally important because by gradual increasing the shear force during a flow curve measurement, materials microstructure is not just simply degraded, but as soon as a change in shear forces occurs, a competition is held between the applied forces acting towards microstructure degradation and the molecular attractive forces (derives usually by Brownian motion) acting towards rebuilding the microstructure which this creates instability. This competition continues until these forces balance out each other, and the microstructure reaches to a stable condition which depends on the material it takes different time to reach equilibrium. Logarithmically spaced data point in flow curve is often suggested because it allocates longer equilibrium times at low shear rate at which materials reach equilibrium longer and shorter equilibrium times at high shear rate at which materials reach equilibrium faster (Mezger, 2006).

Besides, flow instability and secondary flow (e.g., turbulence effect) may occur (Barnes, 2000; Tadros, 2010) in the measurement of the sludge flow curve at high shear rates. Development of turbulence leads to the formation of strong centrifugal force within the measuring gap. As a result of that, measurements (e.g., shear stress or shear rate) decay with time and erroneously show the time-dependent property or thixotropy (Slatter, 1997). The maximum measurable rotational speed to be applied on a liquid in the annular gap of concentric cylinders geometries can be estimated by Reynolds number and Taylor number (Mezger, 2006). Taylor number determines the stability criterion in which there is no vortex due to inertial effects and centrifugal forces. Reynolds number determines the rotational velocity at which the transition from the laminar regime to the turbulence regime is

Table 1
Selected geometries, storage, preshear, equilibrium and rest times for measuring flow curve of sludge at different solid concentrations (2.3–6%TS).

Geometry	Concentration (wt. %)	Storage time [day]	Pre-shear time [s]	Rest time [s]	Equilibrium time [s]
Small gap Vane-and-rough cup	6	0–30	0–1000	0–300	0–60
Small gap Vane-and-rough cup	3.5	0–30	0–1000	0–300	0–60
Small gap Smooth Cup-and-bob	2.3	0–30	0–1000	0–300	0–60

occurred (Mezger, 2006). But, since Taylors number and Reynolds number were not fully validated for non-Newtonian materials especially yield stress fluids (Coussot, 2005) they cannot provide a reliable estimation of the stable region. An easier approach to detect turbulence and secondary flow effects are to visually search the flow curve graph for a sudden increase in slope at high shear rates (Mezger, 2006).

This paper customizes the procedure of the flow curve characterization for sewage sludge in the light of sludge rheological complexity and measurement artifacts. The rest of this paper is structured as follows: In the materials section, the information on the origin and the composition of the sludge samples will be provided. In the protocol development and validation section, first, we investigate the suitability of different geometries, including small-gap-cup-and-bob, grooved-parallel-plate, vane-and-rough cup and wide-gap-cup-and-grooved-bob for the flow curve measurement of 2.3%, 3.5% and 6% digested sludge. Next, we investigate the appropriate parameters in the measurement sequence including preshear time, rest time and equilibrium time that is to be set in a rheometer software. Finally, the impact of storage time on the sludge flow curve is investigated. In conclusion, a validated protocol for a repeatable digested sludge flow curve measurement is provided. The protocol development steps in the following sections apply to any sludge type but the appropriate value of the parameters, such as preshear time or suitability of the geometry may differ from one sludge to another. In fact, all of the following procedures for finding the appropriate value for the flow curve measurement protocol need to be followed to ensure the accuracy of the collected data.

Materials

Digested sludge was sampled at Eastern Treatment Plant, Victoria at an original solid concentration of 2.3% (weight/weight). Samples were thickened to higher concentrations (3.5% and 6%) using the Buchner vacuum filtration process. The solid concentration of the samples was determined by heating the sludge at 105 °C for 24 h according to the APHA standards (APHA, 1992). The range of solid concentration studied here is based on current sludge solid concentration and future need for intensification of anaerobic digesters in a majority of wastewater treatment plants in Australia: 2–2.5% (about 20–25 g/l) is currently the output of most mesophilic anaerobic digesters in Australia, and 6% (about 60 g/l) is the future target concentrations in anaerobic digesters. Rheological characterizations were performed with a controlled-stress rheometer (HR3 TA Instruments) which was connected to a water bath to keep the test temperature at a constant value.

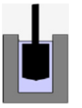
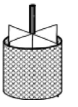
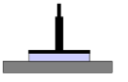
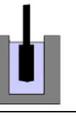
Secondly, the flow curves at each concentration were measured by varying the test parameters according to Table 1, using the most suitable geometry found in the first test. All data for this work is stored in Figshare (Farno and Eshtiaghi, 2018).

To be able to compare the errors of flow curve measurement obtained via different procedures, coefficients of variation (CV) or relative standard deviation was determined as follows:

$$CV = \frac{\text{Standard deviation}}{\text{mean}} * 100 \tag{1}$$

The average of CV was determined to be 5% for three repeats of the flow curve measurements using the same procedure and the same sample batch. So, errors of the flow curve measurements are within 5% accuracy.

Table 2
Geometry types used for flow curve measurements.

Geometry	Specifications	Measurement gap	Schematics
Small-gap-smooth- cup and bob	Bob diameter 28 mm Bob length 42 mm Cup diameter 30.4 mm	1 mm	
Small-gap-vane-and-rough-cup	Vane diameter 28 mm Vane length 42 mm Cup diameter 30.4 mm	1 mm	
Grooved-parallel-plates	Diameter 40 mm	1 mm	
Wide-gap-cup-and-grooved bob	Bob diameter 1 mm Bob height: 42 mm Cup diameter 30.4 mm	8 mm	

Protocol development and validation

Geometry

This section aims to investigate the suitability of four common geometries detailed in Table 2 (including small gap cup-and-bob, grooved-parallel-plate, vane-and-rough cup and wide-gap-cup-and-grooved-bob) for measurement of the sludge flow curve at low (2.3%), medium (3.5%) and high concentration (6%). To do so, flow curves at each concentration were measured at 20 °C using the same procedure discussed in (Farno et al., 2015; Farno et al., 2016a) which is 10 min preshear (at the highest shear rate) followed by 1-min rest and a decreasing ramp of shear stress. This procedure was selected according to the general rules of thumb by Mezger (2006) and the procedure used by sludge literature. Note, a decreasing ramp of shear rate or shear stress is preferred for sludge flow curve measurement according to the work of Baudez (2006).

Results from different geometries were evaluated based on considering two possibilities of slippage in smooth geometry and secondary flow in serrated-wall or vane geometry. Fig. 1(a)–(c) compares the flow curve of sludge at low (2.3%), medium (3.5%) and high concentration (6%) which was measured with grooved-parallel-plate, small-gap-smooth cup-and-bob, wide-gap-cup-and-grooved bob and small gap-vane-and-rough-cup geometries. As can be seen in Fig. 1(a) for dilute sludge (2.3%), smooth-cup-and-bob shows good agreement with grooved-parallel-plates while the vane data is significantly higher. Since two different geometries (smooth-cup-and-bob and grooved-parallel-plate) produced the same flow curve for the dilute sludge (−2.3%), that flow curve considered as the material flow behavior whereas small-gap-vane-and-rough-cup geometry overestimated the flow curve possibly due to secondary flow. In Fig. 1(b), small-gap-vane-and-rough-cup shows good agreement with grooved-parallel-plate while the flow curve data for wide-gap-rough-cup-and-grooved-bob deviates from the two above-mentioned geometries flow curve data beyond shear rate of $100 s^{-1}$ due to occurrence of secondary flow. The flow curve data generated from smooth-cup-and-bob is significantly lower than other geometries flow curve data. This result indicates the occurrence of slippage in the measurement of the 3.5% sludge flow curve with smooth-cup-and-bob geometry. Note, comparing the measurements between different-size geometries is the most common method for detecting the slippage (Barnes, 1995). As a result, either small-gap-vane-and-rough cup or grooved-parallel-plate can be selected for the flow

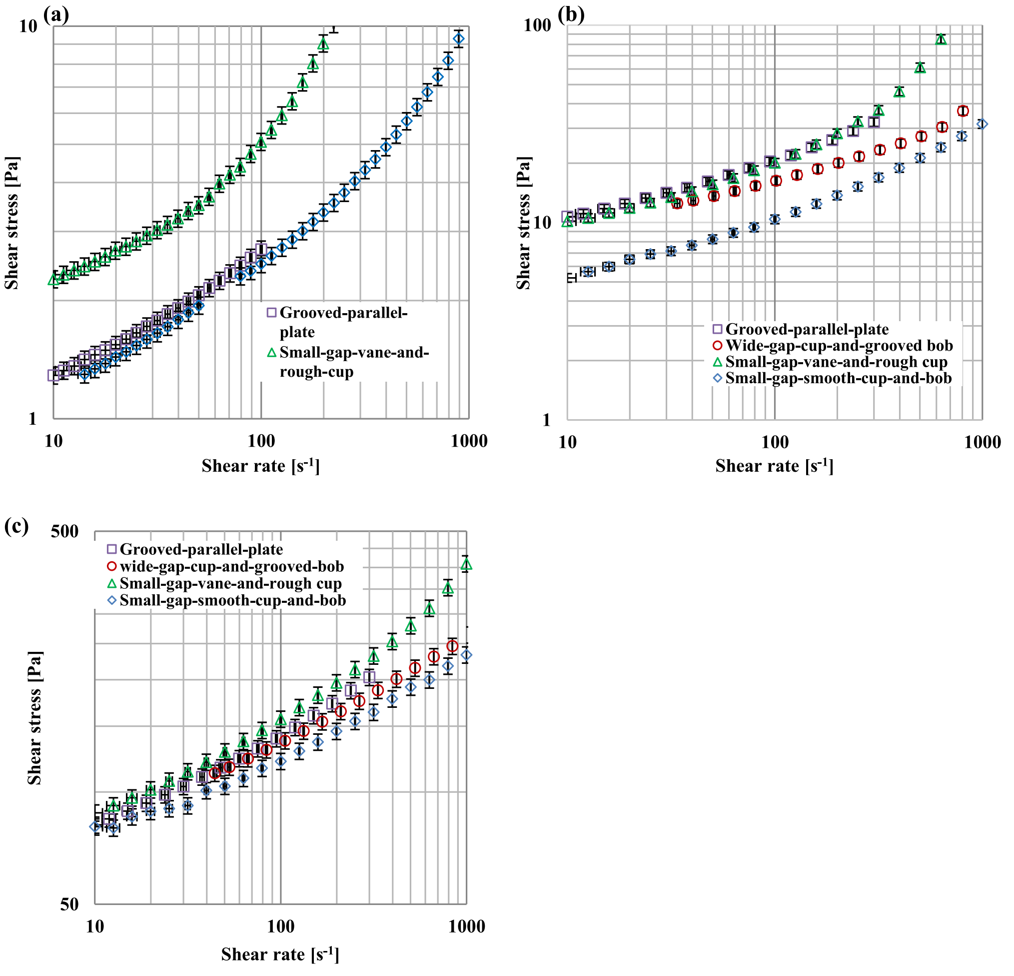


Fig. 1. Comparison of digested sludge flow curves measured via different geometries at 20 °C for (a) 2.3% (b) 3.5% and (c) 6% total solid.

curve measurement of 3.5% sludge. Slippage with smooth-cup-and-bob occurred during the flow curve measurement of 3.5% sludge in contrast to the flow curve measurement of 2% sludge as a result of increasing solid concentration of sludge and particle migration (Barnes, 1995). In Fig. 1(c), again smooth-cup-and-bob measured different values compared to other geometries indicating the slippage phenomena. The flow curve data collected using wide-gap-cup-and-grooved-bob and grooved-parallel-plate showed a good agreement while small-gap-vane-and-rough-cup measurements are slightly higher which may be due to occurrence of secondary flow. Note that although the flow curve data in grooved-parallel-plate overlapped with the flow curve data from different geometries across all measured concentration, it sounds that should be the best choice geometry for sludge. While that is true, however it is a difficult geometry to use because of pushing material out of gap as shear rate goes up. For example, in grooved-parallel-plate, if the range of shear rate or shear stress chosen for preshear and flow curve measurement is too high, the sludge pushes out from the geometry gap and measurement becomes erroneous due to creation of empty spot between two plates gap. As a result, the measurement range with grooved-parallel-plate is limited and may require a trial to be established right range for different samples. In addition, sludge dries out easily in a parallel-plate geometry, particularly where the test temperature is higher than the ambient temperature, and long preshearin time is required. Consequently, it is more suitable to use small-gap-smooth-cup-and-bob at low concentrations (−2.3%), small-gap-vane-and-rough-cup at medium concentration (−3.5%) and wide-gap-rough-cup-and-grooved-bob at high concentrations (−6%).

Nevertheless, it is not recommended to use wide-gap geometry at the first instance because large error occurs in the calculation of the shear stress and the shear rate by rheometers due to Newtonian assumption. For wide-gap geometries, it is necessary to manually calculate shear stress and shear rate according to the non-Newtonian fluid behavior (e.g., for sludge based on shear thinning viscoplastic behavior (Nguyen and Boger, 1987)). During a wide-gap measurement, sludge inside the shear gap can be partially or fully sheared depending on the applied shear stress and the yield stress. For the case in which sludge is fully sheared in the shear gap, literature (Estellé et al., 2008) suggests the Eqs. (2) and (3) to calculate shear stress and shear rate from rheometer torque and angular velocity, respectively.

$$\tau = \frac{\mathrm{M}}{2\pi\,\mathrm{h}\mathrm{R}_\mathrm{b}^2} \tag{2}$$

$$\dot{\gamma} = \frac{2\Omega}{\mathrm{n}}\left(\frac{\alpha^{2/\mathrm{n}}}{\alpha^{2/\mathrm{n}} - 1}\right) \tag{3}$$

Where M [N.m] is torque, h [m] the height of the bob, R_b [m] is the radius of the bob, Ω [rad/s] is angular velocity and, α [-] is the ratio of R_c to R_b and n [-] is the first derivative of $\ln(\tau)$ with respect to $\ln(\Omega)$.

Although wide-gap-cup-and-grooved-bob was indicated as the most suitable geometry for characterization of 6% sludge, in the following sections, the vane-and-rough-cup was used for pre-shear time comparison purpose to avoid misjudgment that may occur due to additional errors imposed on data because of recalculation of shear rate and shear stress by Eqs. (2) and (3).

Preshear time

The purpose of preshear is to remove any shear history present in the sludge (generally created due to gravity sedimentation) as well as homogenization of sludge in the measurement geometry. Preshear intensity is usually set to the highest value of the actual flow curve test to ensure no shear history remains to affect the measurements (Baudez and Coussot, 2001). There is a threshold preshear time which material shear history will be removed completely, and higher homogenization obtained. Numerous factors such as sludge storage time, sludge type, the way the sample is handled and transported before pouring into a geometry are important parameter impacting on creating shear history in the material. A correct preshear time is the minimum preshear time to guarantee removal of shear history and reproducibility of measurements no matter how samples were handled before the test. In fact, the minimum preshear time ensures the flow curve measurement is independent of shear history. Fig. 2 demonstrates the variation of flow curve of 2.3%, 3.5% and 6% digested sludge with varying the preshear time. As can be seen, the preshear time changes the flow curve until specific times above which the flow curves are almost superimposed on the previous measurement. In Fig. 2(a) flow curves measured with 10 min and beyond preshearing time are almost superimposed. While in Fig. 2(b) and (c), flow curves measured with 13 min and 16 min preshear time are almost superimposed. Therefore, it can be concluded that 10 min preshear duration is the minimum time required for preshearing 2.3% sludge while 13 min and 16 min preshear duration is the minimum time required for preshearing 3.5% and 6% sludge, respectively. Similarly, Table 3 shows the calculated coefficients of variation (CV) of sludge flow curve at different preshear time in comparison to the previous preshear time. As the preshear duration increased the variation of sludge flow curve (i.e., the difference between two measurements of flow curves) decreased until it reached a negligible value (e.g., below 5% error of measurement). It indicates all material shear history was removed. When

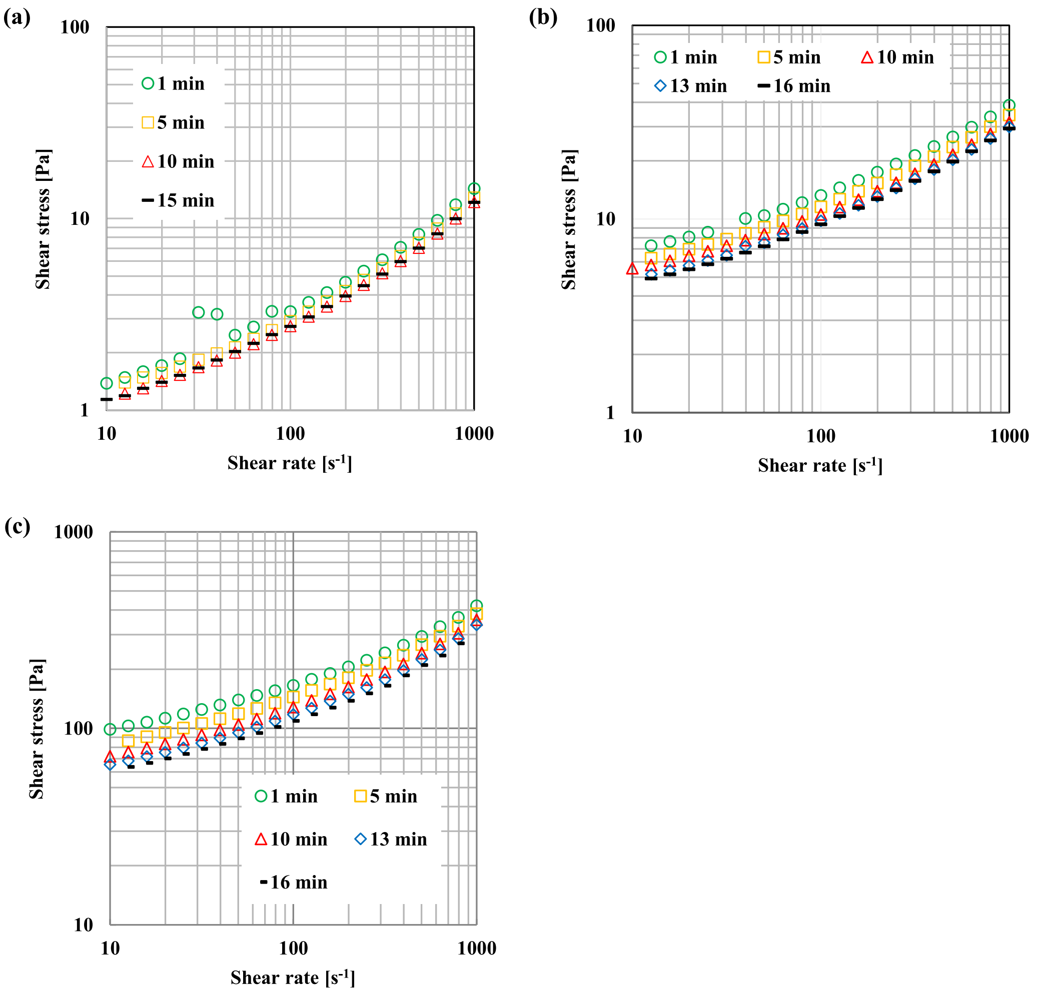


Fig. 2. Comparison of digested sludge flow curves measured at 20 °C with different preshear time for (a) 2.3%, (b) 3.5% and (c) 6% total solid.

preshear duration was extended from 1 min to 5 min and from 5 min to 10 min, 2%-sludge flow curves data varied on the average 13% and 7%, over the measurement range, respectively. As these figures (13% and 7%) were higher than the standard errors of flow curve measurement (5%), they were not a measurement error, but rather indicates that flow curves data is not yet reproducible as material shear history has not been removed completely with preshear duration of 5 min. However, by further extending the preshear duration to 13 min, the variation of flow curve in respect to 10 min preshear reduced to just 0.3% which was below the standard errors of measurements. As a result, 10 min preshear for 2.3% sludge was the minimum time to remove all shear history as the measurement with 13 min preshear resulted in the same figure. Also, Table 3 shows 13 min and 16 min were sufficient time for preshear 3.5% and 6% sludge, respectively. Note that this time is only valid for the sample stored in cooled conditions up to a few weeks before experiments for Australian sludge. For samples stored more than a few weeks or exposed to different storage conditions, the abovementioned preshear time test procedure should be performed to establish the

Table 3
Average variation of sludge flow curve with respect to the previous flow curve when preshear duration increased up to 16 min (Note, the first column shows the variation of flow curve data between 5 min and 1 min preshear duration; CV is the average of cumulative variance of all measurement with different preshear duration).

TS	CV	Variation			
		5 min	10 min	13 min	16 min
2%	11%	13%	7%	0.3%	_
3.5%	14%	13%	8%	7%	4%
6%	16%	13%	11%	8%	6%

Table 4
Average variation of sludge flow curve with respect to the previous flow curve when rest time is extended up to 5 min (Note, the first column shows the variation of flow curve data between 1 min and no rests; CV is the average of cumulative variance of all measurement with different rest duration).

TS	CV	1 min	2 min	5 min
2%	2.5%	3.4%	1.2%	_
3.5%	6.7%	2.5%	1.2%	3.5%
6%	2.2%	2.2%	2.5%	0.6%

appropriate preshear time. However, these preshear times are a good indication of the required range off preshear time.

Rest time

Once the shear history of the sludge is removed by preshear step, it is necessary to leave the sludge at rest for some time until all sludge flocs' structures to be relaxed after experiencing high stress during preshear. Sludge flocs are composed of biopolymers (e.g., extra polymeric substances) bonded together by secondary forces (Farno, 2016). Shear forces can overcome these secondary forces and temporarily degrade the sludge flocs structure. However, once the imposed shear forces are removed, the flocs gradually rebuild the original agglomerated structure (Barnes, 2000). In fact, this rest time allows sludge solids to retrieve their original floc structure (Barnes, 2000). This rest time can be set at a complete retrieval of floc structure where a repeat of the test does not affect the flow curve data, but it is important that this rest time is set according to the actual procedure that the sludge has undergone (or will undergo) in a plant for which the rheological data is required. For example, if rheological properties of sludge after a pump are required, there is no need for rest time more than 60 s as the sludge structure after the pump is fully broken due to the very high shear rates imposed by the pump. So, in this case, 60s-rest time is short enough to prevent the microstructure build up and long enough for materials to be relaxed and to cease the flow. But if sludge is flowing out of a tank by gravity force after a long residence time (For example 4 h) in the tank, the rest time should be chosen at 4 h, so that the floc structure rebuild as much as 4 h rest time. Therefore, the rest time should be selected based on the actual processes. Fig. 3(a)–(c) demonstrates the variation of the flow curve of 2.3%, 3.5% and 6% digested sludge with different rest time. As can be seen in Fig. 3(a)–(c), an increase in rest time up to 5 min did not significantly impact the results of the sludge flow curve measurements. Table 4 also confirms that there was no change in sludge flow curve data with a change of rest time within 5 min as CVs are below the 5% errors of measurements. Note that it is better to set the rest time at the minimum value required because this minimizes the effect of solid settling in addition to saving time in the measurement process.

Equilibrium time

Equilibrium time is the time required to reach a steady-state value at each point of measurement of a flow curve. When sludge is sheared, the flocs start degrading and rebuilding at the same time. In the beginning, the rate of degradation is faster than the rate of rebuilding the flocs, as a result;

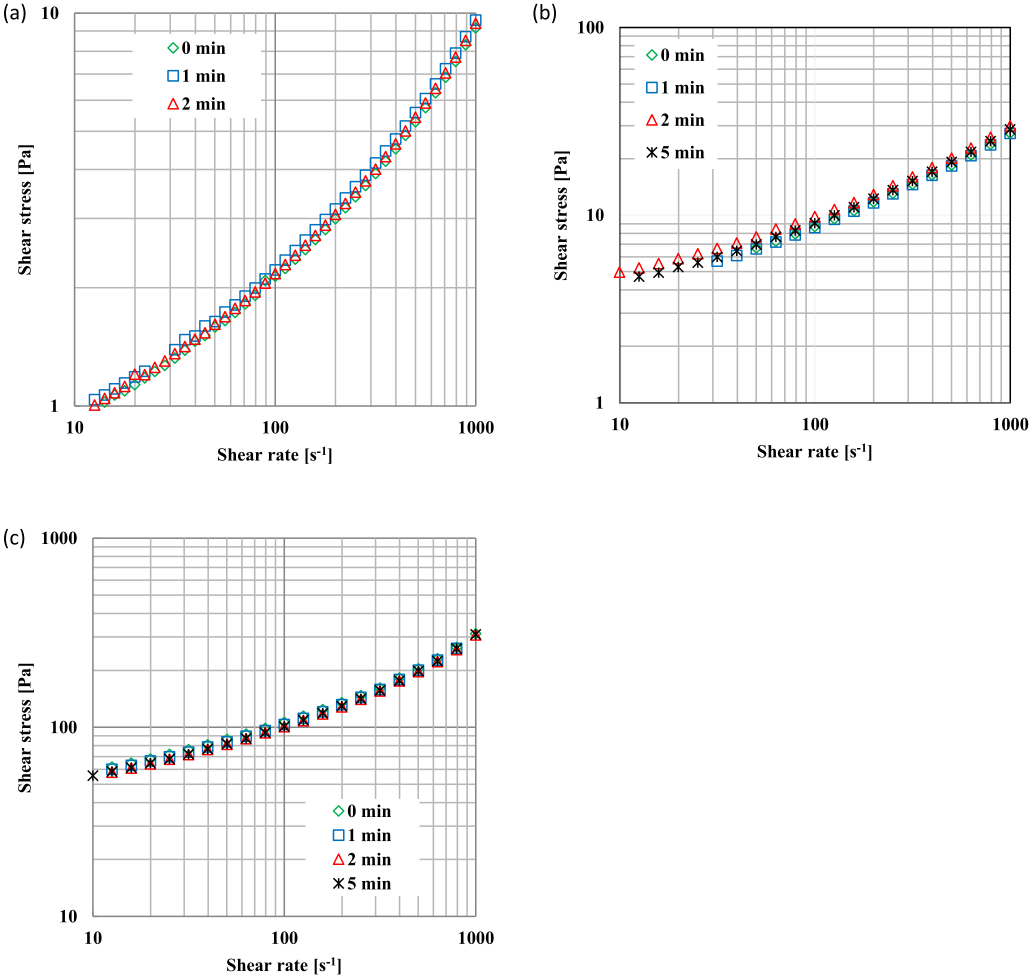


Fig. 3. Comparison of digested sludge flow curves measured with different rest time after 10 min preshear time for different total solid of (a) 2.3% at 36 °C, (b) 3.5% at 20 °C and (c) 6% at 20 °C.

more flocs are broken. This process continues until a balance is achieved in which a portion of flocs remained broken depends on the applied shear rate (Barnes, 2000). The higher the shear rate, the higher the proportion of flocs which remained broken. The steady state at each point of measurement is achieved when the rate of floc degradation balance out with the rate of floc build up. In Fig. 4, the variation of the sludge flow curve with the equilibrium time is shown for 2.3%, 3.5% and 6% digested sludge. As can be seen, there are small variations between flow curves measured with different equilibrium times. Also, there are large fluctuations in the 2.3% sludge flow curve which was measured with zero equilibrium. As presented in Table 5, the variation of flow curve data for 2.3% with 10 s equilibrium time and beyond are below the errors of measurement (5%). So, 10 s is determined as the minimum equilibrium time required for measurement of the 2.3% sludge flow curve. Similarly, the minimum equilibrium time was determined as 20 s and 40 s for 3.5% and 6% sludge, respectively. The minimum equilibrium time of 10 s, 20 s and 40 s for 2.3%, 3.5% and 6% digested sludge; respectively ensure that the data point in the flow curve are collected under a steady state condition. Note that for measurements that need to be quick, for example, the flow curve of

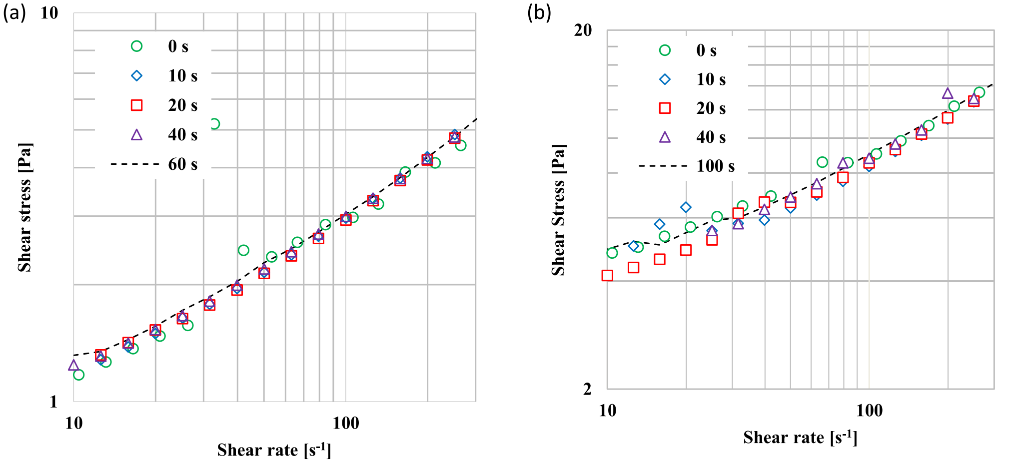


Fig. 4. Comparison of digested sludge flow curves measured at 20 °C with different equilibrium times at each measurement point for (a) 2.3%, (b) 3.5% and (c) 6% total solid.

sludge at elevated temperature in which sludge composition changes over time (Farno et al., 2015), the total number of measurement points can be reduced to avoid long measurement times while the required equilibrium time is maintained.

Storage time

Often, the characterization of sludge rheology is performed a few days after sampling. Sludge properties - including rheology - change with storage time as sludge contains microorganisms that constantly consume organic matter. Literature suggests that the best way to keep these changes at a minimum is to store the sludge in a refrigerator (Baudez, 2008). Table 6 compares the variation of Herschel–Bulkley model parameters fitted to sludge flow curves at different storage time. The Herschel–Bulkley model is given as

$$\tau = \tau_H + k\dot{\gamma}^n, \tag{4}$$

Table 5
Average variation of sludge flow curve in respect to the previous flow curve when equilibrium time is extended up to 60 s (Note, the first column shows the variation of flow curve data between 10 s and zero equilibrium; CV is the average of cumulative variance of all measurement with different equilibrium time).

TS	CV	Variation			
		10 s	20 s	40 s	60 s
2%	3.1%	1%	0.7%	1%	1.6%
3.5%	5.3%	5.1%	1.6%	2.7%	1.2%
6%	5.8%	12%	5.1%	2%	0.2%

Table 6
Variation of Herschel–Bulkley model parameters fitted to the flow curve of 2.3% and 3.5% digested sludge stored in fridge.

	2.3%			3.5%		
	Fresh	1-week	11-week	Fresh	1-week	4-week
τ_y	0.82	0.82	0.74	4.45	4.3	3.1
k	0.1	0.1	0.08	0.39	0.31	0.28
n	0.62	0.61	0.65	0.61	0.64	0.63

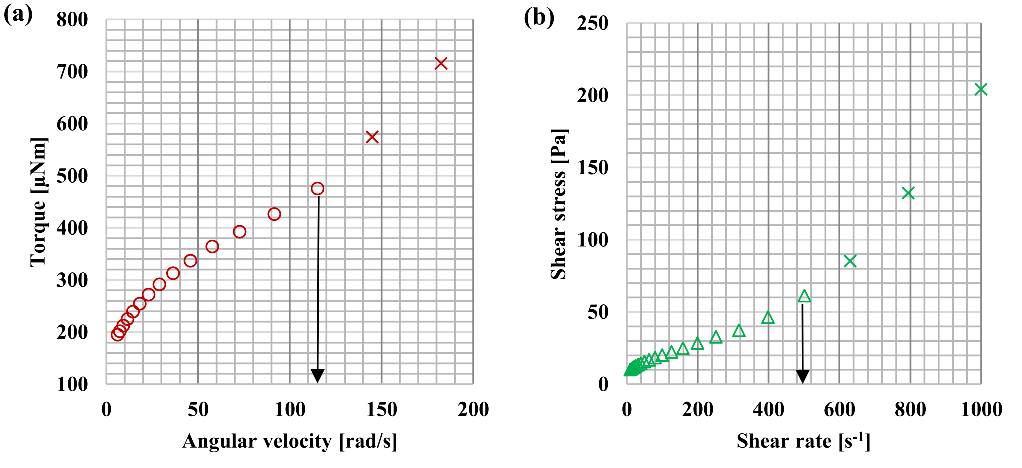


Fig. 5. Cutting turbulence data from flow curve data (a) 3.5% digested sludge measured with wide-gap-cup-and-bob, and (b) 3.5% digested sludge measured with small-gap-vane-and-rough cup.

where τ_H [Pa], k [Pa.s^n] and n [-] are model parameters, τ [Pa] and $\dot{\gamma}$ [s^{-1}] are shear stress and shear rate, respectively.

As can be seen, sample did not change significantly up to one-week storage in the fridge, considerable changes in model parameters were only observed for samples stored for several weeks in the fridge. Therefore, the results of flow curve measurements are closer to the sludge in the treatment plant if samples are stored in the fridge for less than a week before the tests.

Identifying errors

By finding the right geometry and appropriate values for preshear time, rest time, equilibrium time and storage time, most methodological errors in the sludge flow curve measurement are eliminated. Besides, sample drying during the measurement and flow instability are two common sources of errors that one needs to be cautious about them. So it is also important to consider the duration of test and operating temperature when selecting the right geometry to keep the sample from drying

Table 7
Proposed geometry, storage, preshear, equilibrium and rest times for measuring flow curve of digested sludge at different solid concentrations (2.3–6%TS).

TS (% wt.)	Geometry	Pre-shear time [min]	Rest time [min]	Equilibrium time [s]	Storage limit [month]	Measurement range [s^{-1}]
2.3	Small-gap-mooth-cup-and-bob	> 10	< 3	10	1	400 to 10
3.5	Small-gap-vane-and-rough cup	> 13	< 5	20	1	400 to 10
6	wide-gap- cup-and-grooved bob	> 15	< 5	40	–	1000 to 10

out (Farno et al., 2016a). To ensure the reproducibility of data, one may use another suitable geometry to repeat a flow curve measurement. Fig. 5(a) shows turbulence effects appeared in the result above the angular velocity of 115 rad/s using wide-gap-cup-and-grooved-bob. Fig. 5(b) shows turbulence effects appeared in the result above the shear rate of 500 s^{-1} using the small-gap-vane-and-rough cup. Therefore, the results need to be cut out from the shear rate of 500 s^{-1}.

Conclusion

This paper showed how systematically to investigate selecting a right geometry, preshear, equilibrium, rest and storage time for digested sludge in the range of 2.3 to 6%TS. The obtained value for the above-mentioned parameters is a good indication of the starting point for any type of sludge. However, an accurate value only can be obtained with a thorough investigation for sludge under investigation to generate a reproducible flow curve.

We showed that sludge rheology is highly influenced by its shear history, because of that sludge should be preshear-rest according to the actual process in the sludge treatment plant for which flow curve data are collected.

Rheological characterization of concentrated sludge by smooth geometries and by small-gap geometries leads to slippage and flow jamming, respectively. And depending on the sludge concentration (above 2% total solid), an equilibrium time of more than 10 s is necessary at each data point during sludge flow curve measurement. Furthermore, sludge storage time before doing measurement is dependent on sludge concentration as the higher the concentration, less storage time (one week) should be considered. Because sludge rheology is changing faster due to microbial activities.

Based on the obtained results, the recommended procedure to eliminate methodological artifact for rheological characterization of 2.3%, 3.5% and 6% digested sludge is listed in Table 7. Note if sludge contains large coarse particles (one tenth of the gap size) then the small gap is not appropriate geometry for flow curve measurement. A wide-gap-cup-and-bob should be replaced with small-gap-cup-and-bob. A sudden burst of noise in data at low shear rate can be related to trapping particles in the gap indicating an inappropriate measurement gap size.

Although a reliable procedure with specific values for influential parameters on flow curve data collection was developed here for sludge, using the recommended value should be handled with care. However, the procedure used here for obtaining parameters can be utilized to develop appropriate protocols for rheological characterization of any other sludge from any treatment plants in any countries.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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