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Eshtiaghi, N., Markis, F., Yap, S., Baudez, J., & Slatter, P. (2013). Rheological characterisation of municipal sludge: A review. Water Research, 47(15), 5493–5510. https://doi.org/10.1016/j.watres.2013.07.001
Document Version: Accepted Manuscript

Published Version: https://doi.org/10.1016/j.watres.2013.07.001

RHEOLOGICAL CHARACTERISATION OF MUNICIPAL SLUDGE: A REVIEW

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ABSTRACT

Sustainable sludge management is becoming a major issue for waste water treatment plants due to increasing urban populations and tightening environmental regulations for conventional sludge disposal methods. To address this problem, a good understanding of sludge behavior is vital to improve and optimize the current state of wastewater treatment operations. This paper provides a review of the recent experimental works in order for researchers to be able to develop a reliable characterization technique for measuring the important properties of sludge such as viscosity, yield stress, thixotropy, and viscoelasticity and to better understand the impact of solids concentrations, temperature, and water content on these properties. In this context, choosing the appropriate rheological model and rheometer is also important.

Keyword: Municipal Sludge, rheological models, yield stress, viscosity, thixotropy, viscoelasticity, physico-chemical properties

Paper outlines:

1. Introduction

Internationally, wastewater treatment plants are striving to achieve a sustainable sludge management strategy due to the legal banning of conventional sludge disposal methods such as landfill. However, the rapid growth of urban populations has resulted in the production of increasing volumes of sewage sludge. Existing municipal wastewater facilities are reaching capacity, requiring expansion and upgrades to handle the additional load that is anticipated in future. This means that a more concentrated and subsequently rheologically complex sludge will be fed into sludge treatment plants (Eshtiaghi, et al., 2012a). Optimal and efficient design and operation of sludge treatment processes requires accurate prediction of the hydrodynamic functioning of different equipment such as pumps, heat exchangers and mixing systems. Prediction of the correct flow behaviour of these engineering hydrodynamic processes requires accurate knowledge of the rheology of sludge (Slatter, 2011, Ratkovich, et al., 2013; Esthtiaghi et.al. 2012a, Baroutian et al., 2013). Slatter (1997; 2001; 2003; 2004; 2008) has consistently shown that sludge rheology plays a fundamentally important role in analysing the hydrodynamic behaviour of sludge, as it flows through the treatment process. Therefore a better understanding of the flow properties of sewage sludge is required in order to obtain useful parameters to improve the design of sludge treatment processes and to ensure sustainable sludge management.

Recently, Ratkovick et al. (2013) presented the importance of activated sludge rheology on pumping, mixing, bubble diameter, secondary settler hydrodynamic, etc. In particular Ratkovick et al. (2013) focused on the viscosity of activated sludge and compared the viscosity data published from different experimental set ups; this highlighted how changes in experimental protocol would give different results and finding an absolute value for viscosity is not possible. In the second part of this paper, Ratkovich et al. (2013) explains the correct procedure for modelling experimental data in order to obtain a reliable result.

In this paper, we describe a general overview of the different rheological properties of wastewater municipal sludge such as viscosity, yield stress, thixotropy, and viscoelasticity as well as the commonly used rheometers.

2. Sludge rheology and rheological models

Rheology is the science that studies the deformation and flow of matter. Dilute sewage sludge behaves closely to a Newtonian fluid (Sanin, 2002) however, at higher solids concentrations (3-10%) the behavior becomes non-Newtonian and for which the rheological characteristics are highly dependent on the treatment process (Lotito et al., 1997; Battistoni, 1997).

Figure 1: Rheological models (Linear axes)

The non-Newtonian rheological models more commonly used to describe sludge behavior in steady state laminar flow are the simple power-law or Ostwald model (Eq. 1) (Kurath and Larson, 1990; Moeller and Torres, 1997; Bougrier et al., 2006; Terashima et al., 2009; Wu et al., 2011), the Bingham model (Eq. 2) (Sozanski et al., 1997; Guibaud et al., 2004, Mu and Yu, 2006), the Sisko model (Eq. 3) (Mori et al., 2006; Pollice et al., 2007), the Herschel-Bulkley model (Eq. 4) (Slatter, 1997; Baudez, 2001), the Casson models (Eq.5)

(Chhabra and Richardson, 2008), the truncated power-law (Eq.6) (Baudez, 2008; Eshtiaghi et al., 2012b) and the Cross viscosity fluid model (Eq.7) (Sybilski, 2011; Eshtiaghi et al., 2012b)

$$\tau = K\dot{\gamma}^{m} \qquad \text{Eq. 1}$$

$$\tau = \tau_{o} + \eta\ \dot{\gamma} \qquad \text{Eq. 2}$$

$$\tau = \eta_{\infty}\dot{\gamma} + K\dot{\gamma}^{m} \qquad \text{Eq. 3}$$

$$\tau = \tau_{o} + K\dot{\gamma}^{m} \qquad \text{Eq. 4}$$

$$\tau = \sqrt{\tau_{Cy}} + \sqrt{\eta_{C}\dot{\gamma}} \qquad \text{Eq. 5}$$

$$\frac{\tau}{\tau_{C}} = \left(\frac{\dot{\gamma}}{\dot{\gamma}_{C}}\right)^{n} \qquad \text{Eq. 6}$$

$$\mu = \frac{\mu_{0}}{1 + K\dot{\gamma}^{m}} \qquad \text{Eq.7}$$

Depending on the presence of a yield stress, the power law (or Ostwald model) (Eq. 1) and Bingham are the most basic and common rheological models,

The Herschel-Bulkey fluid model is a general form of Bingham model; it is modified to embrace the non-linear flow curve. The Herschel-Bulkley model describes sludge as a shear thinning material and is most commonly used to characterize concentrated sludge (Baudez and Coussot, 2001; Baudez et al, 2011).

Recently, Khalili Garakani et al. (2011) utilized different types of rheological models to characterize activated sludge in a submerged type membrane bioreactor and used the Herschel-Bulkley model to describe the behavior of activated sludge at high concentrations, and the Bingham model to characterize dilute sludge. Also, they used the power law model to describe the viscosity of sludge in the low shear range.

Martin et al. (2011) further commented that the Bingham model is suitable for characterization of membrane bioreactor and anaerobic digested sludge at intermediate to high shear range.

The power law model fails at modeling the Non-Newtonian fluid behavior at high shear rate where viscosity ultimately remains higher than water viscosity. This failure can be rectified by using Baudez's model (2011) in which the Herschel-Bulkley and Bingham models are coupled (Eq.8) to represent the behavior of sludge over the full range of shear rates, where the apparent viscosity tends to a limiting value i.e. plateau:

$$\tau = \tau_o + (K\dot{\gamma}^{m-1} + \alpha_0)\dot{\gamma} \qquad \text{Eq. 8}$$

where α_0 is a plateau viscosity of sludge, describing the rheological behavior of sludge at high shear rates.

Several researchers have attempted to correlate both '*m*' and '*K*' with solids concentrations of sludge. For '*K*', the relationship had been described with a simplified correlation proposed by Landel et al. (1965) (Eq. 9) or exponential function (Eq. 10) (Mori et al., 2006; Moreau et al., 2009) and regression analysis (Eq.11, Eq.12) by Lotito et al. (1997) and Allen and Robinson (1990), respectively:

$$K = \eta_w \left(1 - \frac{TSS}{TSS_{\max}}\right)^{-m}$$ Eq.9

$$K = a\exp(b \times [TSS])$$ Eq.10

Where subscript *w* refers to water, *a* and *b* are empirical coefficients.

$$K = (a.TSS + b).TSS + c$$ Eq.11

$$K = a.TSS^b$$ Eq.12

Where a, b and c are correlation coefficients.

On the other hand, '*m*' can be correlated to the total suspended solids with either a polynomial (Eq. 13) (Slatter, 1997), linear (Eq. 14) (Mori et al., 2006), power-law function (Eq. 15) (Moreau et al., 2009) or regression analysis (Eq.16, and Eq.17) (Lotito et al. 1997 and Allen and Robinson, 1990, respectively):

$$m = b_1 TSS^2 + b_2 TSS + 1$$ Eq. 13

$$m = a - (b \times [TSS])$$ Eq. 14

$$m = a - (b \times [TSS]^c)$$ Eq. 15

$$m = (a.TSS + b).TSS + c$$ Eq .16

$$m = a.TSS^b$$ Eq .17

where TSS is total solids concentration (g/L) with a, *b* and *c* as the empirical coefficients. The flow behavior index and flow consistency coefficient may not be readily used for rheological characterization of sludge, but have proved to be useful indicators of the sludge behavior during rheological measurement.

However, Baudez et al. (2011) revealed similarities in the rheological behavior of anaerobic digested sludge at different solids concentrations by developing a master curve on which each single curve can be plotted. This means that the power-law index remains constant over a wide range of solids concentration. These results were also obtained for highly concentrated sludge from several origins (Baudez et al., 2006), pointing out that rheological parameters are only dependent of two characteristics, the yield stress and high shear viscosity.

Based on a review paper by Seyssiecq et al. (2003), the choice of rheological model is shown to be subjective and highly dependent on the experimental condition such as applied shear stress or sheear rate range as well as type of sludge. For concentrated suspensions, the Ostwald or Bingham model, in general, were the most common model used to describe the rheological behaviour of sludge. Baudez (2002) found that the behaviour of pasty sewage sludge is highly dependent on hydrodynamic and particle interactions. This is due to the competition between these two interactions when sludge is sheared; particle interactions induce structure build-up (aging) whilst hydrodynamic forces tend to resist particle interactions and keep the structure in a broken state (rejuvenation). Baudez (2008) also introduced a new technique to measure the dual rheological behavior of sludge using reconstruction of instantaneous velocity profiles based on repetitive creep measurements. He revealed that the sludge will only achieve homogenous flow (following a truncated power-model) once the shear rate and shear stress are higher than a critical value. As the critical shear rate and shear stress are highly dependent on solids content, this implies that thixotropy may be significant for thickened sludge.

The rheological data available in literature are rarely comparable as there is no standard protocol for characterizing the rheology of sludge. Sample handling and storage prior to characterization have a significant impact on the rheology of sludge. Furthermore, time dependent, thixotropic properties have eluded measurement accuracy. Therefore, there is a necessity for developing a standard protocol to characterize the rheological behavior of sludge so that consistent data can be reported in literature for comparison.

3. Commonly used rheometers for sludge characterization

The instrument used to measure the flow curve of sludge is known as a rheometer. At present, among the commercial available rheometers, rotational and capillary have been used for sludge applications. The test for sludge is carried out over a range of shear stresses or shear rates that are mostly encountered in practice at steady state flow. The rheological information on sludge in laminar region is often extrapolated by several orders of magnitude to predict the behavior of sludge at high shear region (turbulent regime). Therefore, the accuracy of measurement is of utmost important.

The papers reviewed in this work showed that the rotational viscometer, particularly concentric cylinder, has been involved in a wide range of sludge charaterisation work for various industrial applications. In the past, most research focused mainly on identifying the appropriate geometry for sludge measurement and the error associated with such measurement (Seyssiecq, Ferrasse et al., 2003). In the activated sludge process, the secondary clarifier is recognised as the main bottleneck and fulfils a triple-role as a clarifier, sludge thickener and sludge storage zone (Weiss et al., 2007). Therefore, most researchers have tended to sample sludge from the secondary clarifier for rheological study. They were utilising the rotational viscometer for the rheological study of the activated sludge process to develop a more effective and sustainable sludge treatment system. Also, rotational rheometers have been used widely for characterization of membrane bioreactor sludge in order to improve the conventional activated sludge treatment process. Most of the reseachers reported using either Brookfield or Haake type rotational rheometers with similar model or geometries to characterise their research works. Despite the fact that similar rheometers have been used, the results are not comparable. Ratcovich et al. (2013) has presented an overview on the problems associated with comparing activated sludge rheological data due to the lack of measurement protocol data.

This problem is further complicated by variations of sludge samples and its physico-chemistry used among the reseachers and the time-dependent properties of sludge. This makes it difficult to identify whether discrepancies in results are due to the thixotropic property of sludge, the origin of sludge or the artefacts of the measuring process and equipment. Because, measurement errors associated with different inner cylinder design has not been examined properly. Although these designs are said to overcome the wall or end effects during the measurement. Therefore, it is important to examine the physical properties of sludge as well as identifying the major biological components or the associated interparticle interactions that are responsible for its rheological behaviour. This information will be useful for developing models to quantify wall and end effects associated with rotational rheometer and enchance the measurement reliability in rheological study for sludge.

In the following section, an overview of the utilization of capillary and rotational rheometers with their brand and geometry used for sludge characterization is presented. Table 1 in supplementary provides a summary of research works utilized rotational rheometers.

3.1. Capillary rheometer

The capillary or tube viscometer, also known as Ostwald viscometer, employs a pressure gradient to cause fluid to flow in laminar region at a measured shear rate through a capillary tube of known diameter and length (Figure 2). It is the most common instrument for the fluid viscosity measurement due to its relative simplicity, low cost and accuracy (in the case of long capillaries) (Chhabra and Richardson, 2008). Slatter (1997) and recently Ratcovich et al. (2013) has reviewed the advantages and disadvantages of using capillary viscometer for sludge characterization. The principal advantages are mechanically simple (similar geometry to pipe flow), high shear ranges can be attained and enable measurement of diameter dependent effect. On the other hand, the disadvantages include larger sample volumes required, the same sample of fluid cannot be subjected to sustained shear for measuring time-dependent effects and the sample is subjected to a varying rate of shear over the tube cross section. Chhabra and Richardson (2008) added that cleaning could also be a problem due to the small diameter of the tube.

Figure 2.Schematic of capillary rheometer (image courtesy to google)

3.1.1. End effect and wall slip

The two common sources of error associated with capillary devices are so called end effects and wall effects. For purely viscous fluid, this effect is usually neglected as long as the length to diameter ratio (L/D) of the capillary tube is of the order of 100 to 120 (Nguyen et al., 2007a). As for viscoelastic substances larger L/D values are required and as of now there is no conclusive estimate of the desired ratio (Nguyen, et al., 2007a). On the other hand, wall slip mechanism, which is commonly accepted for a concentrated suspension, can be explained by formation of a slip layer adjacent to the wall due to particle migration (Baudez et al., 2007; Nguyen et al., 2007b). The slip phenomenon has been well documented by others (Rosenberger et al., 2002; Bellon et al., 2007; Paredes et al., 2011).

3.1.2. Overview of utilization of capillary rheometer for sludge characterization

One of the earliest attempts to study the rheological properties of sewage sludges using capilliary rheometers was done by Babbit and Caldwell (1939). However, their results were not satisfacory due to the difficulties faced during the measurement. These include insufficicent sludge sample, clogging and velocity control by means of a valve. Notable contributions from other reseachers in this area include Brisbin (1957) using a capillary rheometer to correlate the rheological properties sludge with solids concentration, by Sirman (1960) to characterize digested sludge. Bhattacharya (1981) realized the significance of the physico-chemical effect on the sludge rheological properities and utilized a tube viscometer to examine the effect of temperature and solids concentration on the sludge behaviours. Seyssiecq, et al. (2003) have acknowledged some of the works done on sludge characterization using a commercially available capillary rheometer in their review paper, notably anaerobic digested sludge (Behn, 1962) and concentrated sludge (Gasnier, et al., 1986; Hiemenz and Rajagopalan, 1997). Most recently, the capillary rheometer was used by Grant and Robinson (1990) to measure the rheological properties of filamentous broth, by Slatter (1997) to relate rheological properties of sludge to operating conditions in the sludge pumping process and also by Poitou, et al. (1997) to study the rheological and mechanical properties of pasty sludge.

Bache and Papavasilopoulos (2000) employed Ostwald rheometer in their research to analyze the viscosity of sludge in response of polymer conditioning and determine the optimum polymer dosage required for dewatering applications. Ward and Burd (2004) used modified Ostwald rheometer to perform viscosity measurements on conditioned sludge with different pHs and solids concentrations at 100^{o}C.

A recent article by Pullum et al. (2010) questioned the validity of tube viscometer in examining the rheological properties of stable ‘homogeneous’ suspension with coarse particles. The experiment was carried out with CMC solution and glass beads (~1mm) as pure carrier fluid and coarse particle, respectively, in small and industralized pipes. Their result showed that the stratified bed flow effect in homogeneous suspension may be negligible in tube capillary, but it dominated the transport pressure gradients in the industralized pipe. Their work has also cast doubts upon the validity of capillary tube data obtained with ‘normal’ slurry size distribution and whether it can be used directly in system design and process control for industrial scale. Clearly this phenomenon will also need to be studied to examine the validity of tube viscometer data for the rheological characterisation of primary sludge, if the design of high concentration pumping systems for primary sludge is to be performed with any certainty.

The most recent development of capillary rheometer technology in sludge application was presented in an article in which Slatter et al. (1996) described a modified capillary device called the Balanced Beam Tube Viscometer (BBTV). The device is composed of a transparent tube of various diameters that connects to two pressure vessels located at the either end of the beam. Compressed air with known pressure enables sludge to flow through the tube at a controlled rate. Mass in the load cell is registered over time, indicating the flow transferred through the tube. The principal advantage of this device is that sludge flow is not measured with a classical flow meter but calculated from the variations in mass measured by simple weighing. Therefore the accuracy is higher than that of a conventional flow meter and very low flow rates can be measured, which overcame the earlier velocity control issue by valve as depicted by Bobbit and Calldwell (1936). Slatter et al. (1998) improved the BBTV design to facilitate more accurate design of pipe and pumping plants for non-Newtonian slurries. This improvement allowed large number of data points to be collected in laminar and turbulent region and its transition point to provide useful data for process design which cannot be done with conventional capillary tube viscometer. However, the current design of BBTV is limited by maximum pipe diameter of 50 mm and still had not overcome the limitations of the previous design, such as inability to measure time dependency of materials and large sample volume is required. Nevertheless, their work was able to demonstrate that BBTV is a versatile and reliable instrument for both routine analyses and research work and can achieve more accurate measurement compared to typical tube viscometer. Most importantly, it has also demonstrated the potential to be adapted to enhance the accuracy and reliability for rheological measurement of activated sludge.

3.2. Rotational rheometer

The rotational rheometer with concentric cylinder geometry has became widely accepted and commercially available in recent years, and the most common class of rheometer used in sludge rheology (Figure 3). This device relates the measured torque to shear stress as well as angular velocity to shear rate, therefore enables evaluation of the rheological properties of sludge. Detailed theoretical analysis to develop basic equations for rotational viscometry is available in standard texts such as that by Van Wazer (1963).

Figure 3.Schematic of rotational rheometer with different geometry such as a) vane, b) concentric, c) cone-plate, d) parallel plate, e) double concentric (image courtesy to google)

The design of this rheometer offers unique features to study the rheological property of sludge, which is not available in other types of rheometer. These advantages include continuous operation to allow evaluation of time dependent properties, small sludge samples for testing, can be installed as bench top instrument and enables rheograms to be obtained when directly linked to a PC (Dick and Ewing, 1967; Slatter, 1997, Ratkovich et al., 2013).

3.2.1. Gap size

Dick and Ewing (1967) and Dick and Buck (1985) have provided a comprehensive equipment analysis and requirement for sludge application. Dick and Ewing (1967) noted that narrow gap rotational rheometer was not suitable for rheological measurement of sludge since the gap size was much smaller compared to the particle size in the suspension being investigated. They have commented that the gap size must be at least 10 times larger than particles in the sludge to ensure the device was sensitive enough to measure low viscosity substance. On the other hand, a wide gap would contribute to the development of turbulence which lead formation of strong centrifuge action within the measuring gap. Centrifuge action can cause the readings to decay with time and subsequently lead to erroneous identification of time-dependent property or thixotropy (Slatter, 1997). To minimize the effect due to centrifuge action, Chhabra and Richardson (2008) suggested that the ratio of diameter of inner to outer cylinder must be larger than 0.99. Seyssiecq, et al. (2003) has discussed this issue in his review paper and noted that the choice of concentric cylinder geometry depended on the type of sludge that one is working with. Indeed, the effect of measuring geometries on sludge rheology has been demonstrated by Mori et al. (2006). In their experiments, a rotational and controlled stress rheometer with concentric cylinder (CC) (measuring gap: 1mm) and double concentric cylinder (DCC) (measuring gap: 0.38 and 0.42 mm) were used to obtain the flow curves for activated sludge, which was composed mainly of macroflocs with mean diameter of 125µm. The experimental results indicate that the CC systems is suitable for characterization of sludge whereas the dimension of the DCC geometry are too small leading to blockage of flow as the suspension is sheared.

3.2.2. Overview of utilization of the rotational rheometer for sludge characterization

The Rotational viscometer has proven to be a useful tool to obtain rheological properties of sludge for process design and modelling as well as optimization. Most of reseachers employed this type of rheometer to examine the influence of operating conditions and physico-chemical properties on the viscosity of sewage sludge (summary presented in the Table 1 as a supplimantry material).

Several authors used a stress-controlled concentric cyclinder rheometer – DSR200 to evaluate rheological properties of anaerobic digested sludge at various solids concentrations (Esthiaghi, et al. 2012b) and temperature (Baudez et al., 2013b). The effect of measuring geometries on the rheological behaviour of sludge was reviewed by Seyssiecq et al. (2003), Mori et al. (2008) and by Mori et al. (2006) using different concentric cylinder geometries. Laera et al. (2007) and Pollice et al. (2007) as well as Pollice et al. (2008) have employed Rheotest 2.1, Haake Mendigen (GMBH) equipped with concentric cylinder to examine the rheology of bioreactor sludge at solids retentions times of 20 days at 20^{o}C. Several researchers employed rotational rheometer in examining the effect of pre-treatment and polymer conditioning prior dewatering on the rheology of sludge. In the recent years, influence of ultrasonification pretreatment on the rhelogical features of sludge has been widely studied with different types of rotational rheometer such as Brookfield type rotational rheometer (Pham et al., 2009; Pham et al., 2010) and RS 300 stress-controlled rheometer (Ruiz-Hernando et al., 2010). Kim et al. (2009) used a Brookfield type to investigate the rheology of secondary sludge after alkaline pretreatment and hydrogen peroxide oxidation to investigate the efficiency of each process for more effective excess sludge reduction. Jolis (2008) and Verma et al. (2007a) utilized rotational disk rheometer and Brookfield type rheometer, respectively, to demonstrate that solids after thermal hydrolysis pretreament, sludgeviscosity reduces and the fraction of soluble organic matter increases. Ayol and Dentel (2005), on the other hand, analysed the rheology of anaerobic digested sludge after enzymatic treatment with a Brookfield type rheometer to derive parameters that may be used to characterise drainability and filterability dynamic.

The Rotational rheometer is also commonly used for rheological characterisation of sludge samples obtained from different stages or processes in sludge treatment. Mu and Yu (2006) used a shear controlled rotational rheometer to determine the characteristic of granular sludge with average size of 150 to 250 µm in an upflow anaerobic reactor. Mu and Wang (2007) utilized a rotational rheometer equipped with double gap measuring system to determine surface characteristic of anaerobic granular sludge in acidgenic fermentative process. Fonts et al. (2009) employed a rotational rheometer for viscosity measurment as part of their works to evaluate the physico-chemical properties of pyrolysis liquid of sewage sludges for possible energy applications. Wang and Dentel (2011) used Brookfield rheometer equipped with ultralow adapter to determine the supernatant viscosity of raw anaerobic digested sludge after centrifuge. This type of viscometer was also used to characterise sewage sludge and wastewater that were incubated with different types of fermenter. For instance, Verma, Brar et al. utilized a Brookfield type viscometer to characterise *Trichoderma viride* fermented starch wastewater (2006) and activated sludge (2007b) as well as *Bacillus Thuringiesis* fermented primary secondary and mixed sludge (60% primary, 40% secondary) (Brar, Verma et al., 2005; Brar, Verma et al., 2008) to evaluate the optimum operating condition and to test their feasibility as potential growth substance on the basis of process performance and rheology when compare to other commercial medias. Recently, Seviour et al. (2009a) employed a strained controlled rheometer ARES with parallel plate to characterise aerobic sludge of a lab-scale sequencing batch reactor at different pH, temperature and salt concentration based on storage modulus ($G^{'}$) and loss modulus ($G^{''}$) to demonstrate that the granules were hydrogels. Khongnakorn et al. (2010) demonstrated that they were able to utilize a stress controlled Haake rheometer to evaluate the rheological properties of membrane bioreactor sludge during unsteady state flow condition at 21^{o}C. The experiment showed that change in applied stress could affect the solubility of organic materials in sludge and therefore influence the solids behaviour of sludge. Their work had highlighted the possibility to improve the performance of membrane bioreactor unit by modifying the presense of soluble microbial compounds i.e. the microbial activity induced by the fermenter.

The Rotational rheometer can also be used to analyse dewaterability of sludge through rheological study. Hou and Li (2003) used a Brookfield rheometer to evaluate the feasibility of using rheological properties to assess dewaterbility of inorganic water and organic activated sludge that were conditioned with fly ash and polymer. They have concluded that both minimum viscosity and rheograms peaks could be used to measure the dewaterability of inorganic water sludge, but not for organic sludge. Örmeci and Abu-Orf (2005) proposed a protocol to directly measure the overall network strength of sludge using concentric cylinder rheometers to evaluate the dewaterability of wastewater sludge. Indeed, Örmeci (2007) has also reported the problem associated with the reproducibility of the measurement using concentric cylinder due to difficulties to obtain representative sub-samples from well flocculated sludge in his work to optimize conditioning and dewatering process in wastewater treatment.

Several researchers employed this type of rheometer to determine characteristic of sludge in menbrane bioreactor to evaluate the process performance and optimization. Chu, Wu et al. (2007) used a shear rate-controlled Brookfield viscometer to test the dewaterability and perform structural analysis on the sludge sampled from a pilot-scale membrane bioreactor and estimated the appropriate polymer dose prior dewatering to improve the process performance via hystersis loop test. Van Kaam et al. (2008) used a Bohlin C-VOR 200 Rheometer to perform viscosity and oscillation measurement of mixed liquor. Ho and Sung (2009) used a Haake type viscometer to investigate the effect of solids concents and hydrodynamic conditions on microfiltratio (pore size = 1 µm) of anaerobic digested sludge. Recently, Brannock et al. (2010) utilized a rotational stress-controlled Haake rheometer to investigate mixing characteristic of full-scale membrane bioreactors and developed a computational fluid dynamics model framework for biological wastewater treatment which accounted for aerations, sludge rheology and geometries of the reactor itself. The validity of model has been verified with two full-scale membrane bioreactors and successfully predicted the overall reactor residence time distribution with high precision. Weiss et al. (2007) used a rotational viscometer to perform on-site rheology experiments to develop a computational fluid dynamic model that predicted the sedimentation of activated sludge in a full scale flat bottom circular secondary clarifier that is equipped with a suction-lift sludge removal system. The model prediction was showed to agree well with the measured sludge concentration profiles in the clarifier for two different treatment plant loadings. Efterkharzadeh et al. (2007) have employed a Haake type rheometer to obtain site-specific sludge rheology data to upgrade the wastewater treatment system to handle higher solids concentrations. The rheology data were used to prepare a scale-up model for the digester mixing system as well as develop a computational fluid dynamics model that can be used to assess the effectiveness of mixing. The paper demonstrated the benefits of analysing site-specific sludge rheology for assessing the effect of solids concentration on the mixing efficiency of anaerobic digester.

4. Viscosity

Viscosity is defined as the ratio of shear stress to shear rate, which can be evaluated by means of the flow curve. The more viscous and less flowable the fluid, the greater is the viscosity (Ratkovich et al., 2013). This parameter has been a fundamental measure for physical characteristic of sludge suspension relating to deformation and flow properties. Since sludge is non-Newtonian fluid as the viscosity changes with shear rate or applied stress. Therefore, the term 'apparent viscosity' is used to describe this behavior. A non-Newtonian behavior of sludge observed to be shear-thinning (Chaari et al., 2003), is commonly characterized by a decreasing apparent viscosity over increasing shear rate, but at extreme low and high shears rate exhibit Newtonian behavior. The resulting apparent viscosities at low and high shear rate are known as zero shear viscosity, η_0, and infinite shear viscosity, η_∞, respectively. Thus it is also valid to say that the apparent viscosity of shear thinning fluid reduces from zero shear viscosity to infinite shear viscosity with increasing shear rate.

Several researchers chose to characterize sludge rheology based on limiting viscosity (Tixier et al., 2003a; Pevere et al., 2006). Due to non-Newtonian behavior of sludge, the rheological property of sludge can be better described by a single parameter of limiting viscosity (Seyssiecq et al., 2003), which allows proper comparison of viscosity for different sludge samples (Tixier et al., 2003a). Limit viscosity corresponds to an asymptote value of the viscosity-time curve at high shear rate when the apparent viscosity becomes almost constant. It can be interpreted as being the viscosity of sludge corresponding to the maximum dispersion of floc under the influence of shear rate (Tixier et al., 2003b). This parameter has been employed to characterize a wide range of sewage sludge, such as anaerobic digested sludge (Battistoni et al., 1993; Pevere et al., 2006; Pevere et al., 2007; Li and Yu, 2011), aerobic sludge (Riley and Forster, 2001; Tixier et al., 2003b; Su and Yu, 2005), bioreactor sludge (Abu-Jdayil, Banat et al., 2010) and activated sludge (Tixier et al., 2003a). Besides characterizing sludge, limit viscosity serves as a good indicator of internal resistance (Battistoni et al., 1993) of different origins (Tixierb et al. 2003a,b) for the same treatment process. Several researchers have also attempted to use viscosity as means to evaluate thixotropic properties of sludge (Baudez and Coussot, 2001; Brar et al., 2005).

4.1. Effect of solids concentration on viscosity

For a suspension that is diluted enough and remains Newtonian, the relationship between viscosity and particle concentration can be described by the Einstein equation:

$$\eta = \eta_0 (1 + 2.5\phi) \qquad \text{Eq. 18}$$

where η is viscosity, η_0 is the viscosity of the fluid phase and ϕ is the particle volume fraction. The equation assumes the solids suspended in the fluid are spherical, non-interacting, insoluble and rigid (Sanin, 2002).

The effect of solids content on the limit viscosity of sludge has been examined in a great number of studies. It was found that the limit viscosity of sludge increases with solids content (Forster, 2002; Tixier et al., 2003b; Pevere et al., 2006; Mu et al., 2007; Moreau et al., 2009; Abu-Jdayil et al., 2010). At high solids content, structural units of suspension may be larger in size and closer to each other, leading to stronger inter-particle interactions and hence the higher apparent viscosity of sludge. This behavior has been mostly described with an exponential function (Battistoni et al., 1993; Rosenberger et al., 2002; Tixier et al., 2003b; Pevere et al., 2006; Abu-Jdayil et al., 2010) or power model (Lotito et al. 1997; Tixier et al. 2003b; Su and Yu, 2005). Recently, Baudez et al. (2011) demonstrated that the relationship between Bingham viscosity and solids concentration followed an exponential law, too. Considering most sludge has high fraction of suspension and interact with each other, it is unrealistic to expect that Einstein law is to be applied to these systems (Sanin, 2002). The effect of solids concentration on viscosity of sludge is in parallel with particle sizes, as both attribute to an increase of inter-particle interactions. As shown by Pevere et al. (2006), decrease in particle size at a constant solids concentration increased limit viscosity of sludge. This suggested that a decrease in particle size increases the surface area of particle to interact with each other. This also underlines the importance of the particle-particle interactions from a quantitative point of view (Pevere et al., 2006).

Recently, Khalili Garakani et al. (2011) have proposed a simplified correlation (Eq.19) to relate viscosity of activated sludge with mixed liquor suspended solids (ϕ_p) and shear rate ($\dot{\gamma}$) at 20^{o}C.

$$\eta = a \times \left(\frac{\phi_p{}^{b}}{\dot{\gamma}} \right) \qquad \text{Eq. 19}$$

where *a* and *b* are the empirical coefficient.

This correlation has been verified with the experimental data presented in work of Rosenberger et al. (2002) and Yang et al. (2009) and showed a better prediction capability, especially at lower and upper Newtonian regions. The authors also related the aeration intensity (U_g) to the viscosity of sludge based on the work of Popovic and Robinson (1984) and yields the following equation, which reveals the significance of air injection in an aerated fermenter:

$$\eta = \frac{a}{c} \times \left(\frac{\phi_p{}^b}{U_g} \right) \qquad \text{Eq. 20}$$

where *a* and *c* are the empirical coefficient.

Furthermore, they have emphasized the use of more sophisticated viscosity models such as Carreau or Cross model as they are able to provide the best prediction of viscosity in the whole wide range of shear rates for activated sludge. Saffarian et al. (2011) have applied modified Bingham model, based on the work of Papanastasiou (1987) , to simulate the sludge flow of a secondary clarifier in a sewage treatment, in which the plastic viscosity (η_p) can be expressed as below:

$$\eta_p = \left[1 - \exp\left(-m\dot{\gamma}\right)\right]^n \tau_B \dot{\gamma} + \eta_B \qquad \text{Eq. 21}$$

where *m* and *n* are shear rate and growth power rate, respectively, and τ_B and η_B are Bingham yield stress and viscosity, respectively, and are expressed as a function of temperature and concentration in the literature. The correlation has been verified and fitted well with the experimental data by Weiss et al. (2007).

Krieger – Doughety (1959) Viscosity model that takes into account the maximum packing fraction (ϕ_m), intrinsic viscsoity (η), and volume fraction of dispersed phase (ϕ) which modified by various authors (Behzadfar et al. (2009) and Kitano et al. (1981)) has been presented in Eq.22.

$$\eta_r = \left(1 - \frac{\phi}{\phi_m}\right)^{-[\eta]\phi_m} \qquad \text{Eq.22}$$

4.2. Effect of temperature on viscosity

The temperature dependent properties of sludge have been well-documented and examined. It is agreed in general that increase in temperature will result in a decrease in sludge viscosity (Battistoni et al. 1993; Sozanski et al., 1997; Mu et al., 2007; Abu-Jdayil et al., 2010; Baudez et al., 2013b). However, the temperature effect is not significant if the temperature range examined is approximately room temperature or even lower (Moreau et al., 2009). The relationship of sludge viscosity and temperature can be described with an Arrhenius type equation (Eq.23):

$$\eta_\infty = K \exp\left(\frac{E_a}{RT}\right) \qquad \text{Eq.23}$$

where η_∞ is limit viscosity, K is empirical constant, T is absolute temperature, R is universal gas constant, and E_a is the activation energy. This expression has been used to describe temperature effect on limiting viscosity of several types of sludge: bioreactor sludge (Yang et al., 2009; Abu-Jdayil et al., 2010), anaerobic digested sludge (Battistoni et al., 1993; Mu et al., 2007; Pevere et al., 2009; Baudez et al., 2013b) and diluted sludge (Sozanski et al., 1997). Several researchers have utilized different form of equations to estimate the temperature effect on viscosity of sludge. Sozanski et al. (1997) studied the effect of temperature on the Bingham plastic viscosity and yield stress. The relationship between temperature and rheological parameters was defined using a temperature factor "WT":

$$(WT)_1 = \frac{1}{T-273.45}\left[\frac{(\eta_B)_{273.45}}{(\eta_B)_T} - 1\right].100 \qquad \text{Eq.24}$$

η_B is the Bingham viscsoity and T is the temperature.

Dieudé-Fauvel et al. (2009) proposed a VTF model (Eq.25) to measure the viscosity of sludge as a function of temperature:

$$\eta = a\exp\left(\frac{b}{T-T_o}\right)+c \qquad \text{Eq.25}$$

where a, b and c are the dimensionless coefficients and T_o is the standard temperature (293.15 K).

Jiang et al. (2007), on the other hand, utilized another form of expression, (Eq.26), to estimate the temperature effect on the viscosity of sludge in their work to develop a hydrodynamic model for membrane reactor:

$$\ln(\frac{\eta}{\eta_o}) \approx a + b(\frac{T_o}{T}) + c(\frac{T_o}{T})^2 \qquad \text{Eq. 26}$$

where η and η_o are the viscosities that corresponded to T and T_o, respectively, and a, b and c are the empirical coefficients.

Yang et al. (2009) have presented a correlation that described the relationship between viscosity, mixed liquor suspended solids of bioreactor sludge (ϕ_p), and temperature at a constant shear rate (Eq.27):

$$\eta = a{\phi_p}^b e^{\frac{E_a}{R(T+273.15)}} \qquad \text{Eq.27}$$

Khalili Garakani et al. (2011) had modified this equation and proposed a generalized correlation (Eq.28) that includes the effect of shear rate on apparent viscosity and verified it based on the experiment results of Yang et al. (2009):

$$\eta = a\frac{\phi_p}{\dot{\gamma}}^b \; e^{\frac{E_a}{R(T+273.15)}} \qquad \text{Eq. 28}$$

The correlation shows a good agreement with the experimental data within the solids content of the work.

However, it was observed that thermal history may have a strong impact on the viscosity of sludge. Baudez et al., (2013b) have examined the viscosity of anaerobic digested sludge after heating and cooling and found that the Bingham viscosity increased. It was suggested that solids might have converted to dissolve compound and this process is partially irreversible. Therefore, the usual expression to model temperature dependence of sludge can no longer be applied due to change in sludge composition during the process of heating and cooling (Baudez et al., 2013b).

4.3. Effect of bound water content on viscosity

Few researchers have examined the effect of bound water content on the limit viscosity of sludge. Sozanski et al. (1997) observed a drop in sludge viscosity as the water content increased, which has previously reported by Forster (1983), and described the behavior with an exponential function (Eq.29). This behavior may be explained by the change in floc structure and presence of extracellular polymeric substances on the sludge surface (Liao et al., 2000).

$$\eta_B = \eta a \exp[b(W_{kr} - W)] \qquad \text{Eq.29}$$

W_{kr} *and* W are the critcal water content and water content of the samples. η_B, η are the plastic viscosity, apparent viscsoity (Bingham mode), respectively.

Recent development of new technologies in the wastewater treatment process, such as membrane bioreactor, has urged researchers to consider different experimental conditions when characterising sewage sludge. For instance, Seyssiecq et al. (2008) has considered the effect of aeration rate on the viscosity of sludge when performing an *in situ* rheological characterization of sludge in aeration bioreactors. It was observed that the viscosity of the sludge decreased significantly at low shear rate but was almost independent of aeration rates. At high shear rate, mechanical shearing was the dominant factor in that the structural reconfiguration of sludge was independent of the presence of air. The experiment has demonstrated an overall decrease in shear-thinning properties of aerated sludge compared to non-aerated, with a plateau at high aeration rates. The knowledge of flow behavior for aerated suspensions is important to understand the phenomenon occurring close to a membrane, such as fouling or clogging (Seyssiecq et al., 2008).

5. Yield Stress

The issue of whether yield stress really exists is still being debated. The main reason is that no equipment, so far, allows researchers to measure the shear stress of sludge at very low shear rates without being affected by wall-slip or end effects. Besides that, the concept of yield stress is not well-defined. There is variation in terms of rheological models and experimental methods used among researchers to determine the yield stress of a material. It is generally accepted that a rheological model that includes a yield stress term can be used to represent the flow behavior of sludge over a limited shear rate range, but does not necessary indicate that the sludge is a yield stress fluid (Barnes, 1999). Baudez and Coussot

(2001) as well as Mori et al., (2006) believed sludge exhibits yield stress in contrast to Valioulis (1980). Based on a review paper by Seyssiecq, et al. (2003), with the measuring apparatus being more advanced, it is commonly admitted among researchers that yield stress does exist in aggregated concentrated sludge. Indeed, a precise quantitative knowledge of the yield stress is vital to determine the optimum operating conditions of various operations in wastewater treatment, notably mixing and pumping. Yield stress is generally defined as minimum applied stress required for a material to flow continuously. Yield stress is often used to characterize sludge as it indicates the structure resistance due to applied shear rate or stress, therefore giving researchers a sense of the material's network strength and structure. With the presence of a yield stress, the sludge is known as viscoplastic material. Spinosa and Lotito (2003) summarises the importance of yield stress on the various sludge treatment operations (such as Stabilization, Dewatering, Storage/ Transportation, Agricultural use, Land filling, and Incineration) for three different types of sludge: liquid, paste, solid. They have highlighted that yield stress has high impact on storage and transportation of sludge regardless of being liquid, paste or solid.

For non-Newtonian fluid, such as sludge, two types of yield stress can be observed in the flow behavior, which are static and dynamic yield stress. Static yield stress corresponds to the transition stress between fully elastic and viscoelastic behavior, whereas, dynamic yield stress refers to the transition stress between viscoelastic and viscous behavior. In sludge application, it has not been made clear which type of yield stress is most of the researchers interested in measuring. It is assumed that the dynamic yield stress would be the interest of all because a material would flow continuously once this value is exceed, which is consistent with the general definition of yield stress in sludge application. The measurement method for yield stress materials with various types of rheometers has been well documented by Nguyen and Boger (1992) as well as Liddel and Boger (1996). For sludge application, yield stress measurement is mostly determined experimentally through dynamic or flow measurement. In dynamic measurement, a yield stress can be obtained by performing either an oscillatory strain or oscillatory stress sweep at constant frequency. On the other hand, in flow measurement, a rheogram is obtained and allowed yield stress value to be calculated by the extrapolation of flow curve to zero shear using rheological models of sludge eg. Herschel Bulkley (Slatter, 1997; Guibaud, Dollet et al., 2004) or Bingham model (Mikkelsen, 2001). This method heavily relies on the accuracy of measurement, which is difficult to obtain due to wall-slip effect. Few authors studied rheological properties of sludge by combining both dynamic and flow rheometry (Sutapa and Prost, 1996; Baudez, 2002; Baudez and Coussot, 2001). Sutapa and Prost (1996) noticed that the value of yield stress obtained from dynamic test is higher than yield stress of flow measurement. However, Mori et al. (2006) found that the flow yield stress was higher although both were in the same order of magnitude. They justified this by stating that the yield stress obtained from flow measurements corresponds to when the material begins to flow, whilst the dynamically measured yield stress is measured at the point just before the material flows. Recently, the same method had been adopted by Wang et al. (2011a) to determine the yield stress for conditioned and unconditioned sludge. It was found that the yield stress determined based on flow measurement correlated well with the ones obtained the dynamic measurement. Ayol et al. (2006) also conducted flow and dynamic measurement on conditioned and unconditioned sludge samples. The yield stress was determined using the complex modulus (refer to viscoelasticity section for definition) and critical strain value ($\tau_y = G^* \gamma_c$) where the G^* decrease dramatically beyond the critcal strain as the linear viscoelastic raegion ends at this point. They also found that the measured yield stress for synthetic sludge (Dursun et al., 2004) and anaerobic digested sludge (Ayol et al., 2006) are shown in good agreement with the peak network strength measured for the same sample. Although the peak network strength may correspond to the total energy required to break down the structure of sludge, it is not clear whether that the strength measured is equivalent to the yield stress of the same sludge samples as no work had actually been done to examine the relationship between these two. Furthermore, the authors commented that the geometry dependence in determing these two prevented a direct comparison.

5.1. Effect of solids concentration, bound water and temperature on yield stress

Most authors have examined the effect of solids concentration on the yield stress of sludge. It is generally agreed among researchers that yield stress tends to increase as the solids concentration of sludge becomes higher, even for pretreated or conditioned sludge (Mikkelsen, 2001; Riley and Forster, 2001; Forster, 2002; Seyssiecq et al., 2003; Spinosa and Lotito, 2003; Wilen et al., 2003; Abu-Jdayil et al., 2010; Khongnakorn et al., 2010, slatter, 1997). Slatter (1997) relates the yield stress with suspended solids concentration using the correlation presented in Eq.30. Mori et al. (2006) have examined the rheological properties of activated sludge with solids concentration range of 2.5 to 57.0 g/L and fitted the data using Herschel-Bulkley model. They were able to obtain yield stress of sludge through dynamic measurement and correlate it with solids concentration by using an exponential law model (Eq.31):

$$\tau_y = a \frac{TSS^b}{TSS_{max} - TSS} \qquad \text{Eq.30}$$

$$\tau_y = a \exp(b \times [TSS]) \qquad \text{Eq. 31}$$

where τ_y is yield stress, TSS is total solids suspended as well as a and b which are the empirical coefficients. Several other researchers have also expressed the relationship between yield stress of sludge and solids content with an exponential function similar to Eq. 31 (Battistoni et al., 1993; Riley and Forster, 2001; Abu-Jdayil et al., 2010). Seyssiecq et al. (2003) have provided a summary of the yield stress model used to describe different types of sludge under various experimental conditions. Most of the works derived yield stress value from Bingham model for various solids concentrations of sludge.

However, such models give a yield stress value even when the solids concentration is equal to zero, which is physically unacceptable. A minimum solids concentration is required to have a solids structure. In that sense, the power-law model suggested by Baudez (2008, 2011) or Forster (2002) appears more realistic.

Forster also studied the effect of conditioning and pretreatment by using ultrasound on the rheology of sludge. It was observed that yield stress of sludge reduced after pre-treatment and conditioning and the effect was not reversible. In his work, he was able to correlated yield stress to other two parameters, which are bound water content and surface charges of sludge, with a logarithmic relationship. This implies that the development of yield stress can be caused by surface-surface interactions (Forster, 2002). However, this contradicted with the results of his previous work (Riley and Forster, 2001) as he could not relate yield stress to bound water content of sludge. Sozanski et al. (1997) were able to express the relationship between yield stress for diluted sludge and water content in exponential function.

It is also worth noting that several authors, as summarized in the review paper by Seyssiecq et al. (2003), have devoted their works to examine the effect of factors, such as temperature (Manoliadis and Bishop, 1984; Battiston, 1997; Sozanski et al., 1997; Abu-Jdayil et al., 2010), critical water content, Eq. 32 (Sozanski et al., 1997), total volatile solids (Battiston, 1997) and storage time (Baudez, 2002) on the yield stress of sludge. The behavior of yield stress is usually related to temperature by an exponential function. Abu-Jdayil et al. (2010) and Battistoni et al. (1993) have examined the effect of temperature on bioreactor sludge and anaerobic digested sludge, respectively, and can describe the relationship with Arrhenius type equation (Eq.33) and Sozanski et al. (1997) presented Eq. 34 for the correlation between Bingham yield stress and tempreature:

$$\tau_y = c\exp\left[d\left(W_{kr} - W\right)\right] \quad \text{Eq.32}$$

W_{kr} are the critical water content of the sample. τ_y is the yield stress (Bingham model).

$$\tau_y = C\exp\left(\frac{E_a}{RT}\right) \quad \text{Eq. 33}$$

where C is the pre-exponential constant, and E_a is the yield stress activation energy, T is absolute temperature, R is universal gas constant. As for other parameters, the general form of yield stress model cannot be confirmed due to lack of literature data.

$$(WT)_2 = \frac{1}{T-273.45}\left[\frac{(\tau_y)_{273.45}}{(\tau_y)_T} - 1\right].100 \qquad \text{Eq.34}$$

Mikkelsen (2001) demonstrated that apparent viscosity of activated sludge was directly proportional to the Bingham yield stress and commented that these parameters can be used to reflect the number of particle interactions which oppose the flow of suspension.

It seems that most researchers rely on the indirect method which utilizes extrapolation of various flow models to obtain the yield stress value. The direct measurement of yield stress should also be done using the vane method, stress growth and stress relaxation method to verify and compare the yield stress obtained using extrapolation of flow models. However, it is important to review the suitability of the measurement method to ensure its compatibility with type of sludge studied and identify any related errors may need to be considered for correction. For instance, inclined plane test proposed by Coussot and Boyer (1995) may not be suitable for yield stress measurement of sludge as it cannot cover a wide shear range and is not relevant for thixotropic fluid.

Various authors such as Ogawa et al. (1997), Zhou et al. (2001) and Berli and Quemada (2000) have derived yield stress models to determine the yield stress values of colloidal suspensions which can be useful for primary sludge as it acts as a suspension. These models are presented in Table 1 as well as a description of their application.

Table 1: Yield stress models for various suspensions

Author	Model	Description
Ogawa et al. (1997)	$\tau_y = \frac{\phi U(r)}{ad^3}$	Yield stress model that takes into account the volume fraction (ϕ), total interaction potential $U(r)$, and particle diameter (d); a is a model parameter.
Zhou et al. (2001)	$\tau_y = B\frac{\phi^v}{d^2}$	Yield stress model that takes into account the volume fraction (ϕ), Bond strength coefficent (B), particle diameter (d), and power law exponent (v) that is related to the microstructure.
Berli and Quemada (2000)	$\tau_y = X\tau_c$	Yield stress model that is valid for dense suspensions (i.e. $\phi > \phi_c$), X is a rheological parameter, and τ_c is the critical shear stress.

At the moment, there is also no consistent correlation that relates yield stress to any of the physical parameters of sludge such as the origin of the sludge and the experimental conditions employed in each research work different from one to another. This implies that yield stress model can only be determined empirically, which is not desirable. The effect of physico-chemical properties such as temperature or pH on yield stress of sludge has not been examined properly. This could be due to the fact that most yield stress results are not reproducible and can vary by several orders of magnitude even if the experimental conditions were to remain the same. The result inconsistencies are usually associated with thixotropic property of sludge and equipment defects when measuring at low shear rate (Moller, Mewis et al., 2006). Effect of thermal history on yield stress of sludge should be examined as well. It is observed that yield stress of sludge which undergone heating and cooling is less than original sludge at the same temperature and without thermal history (Baudez et al., 2013b). Therefore, it is important to develop a simple, systematic and relevant procedure to characterize yield stress of sludge. Besides that, it is also important to clarify the type of yield stress one is measuring i.e. static or dynamic yield stress. This allows researchers to compare results and discuss any issues related to the measurement easily. This hopefully can accelerate the development of rheological model that can be used to evaluate significance of yield stress in sludge rheology.

6. Thixotropy

Thixotropy refers to the time-dependent disintegration of internal structure (Figure 4) as a result of the application of shear stress (Baxter 1988; Battistoni 1997; Tixier et al. 2003 a, b ; Baudez 2006; Baudez 2008).

According to Baudez (2008), below a critical shear stress, colloidal forces tend to rebuild the solids structure (physical aging) and shearing forces tend to break the solids structure (shear rejuvenation). As soon as the critical shear is reached, the solids structure is completely collapsed, and fluid starts flowing which the relationship between the shear rate and the shear stress can be defined with a truncated power-law (Baudez, 2008). In practice, thixotropic effects can alter pipe transportation by producing clog if the wall shear stress is not high enough to maintain a homogenous flow. Therefore, change of flow behavior of sludge over time is important to be considered in pipeline and pumping system design. This worsens by increasing sludge concentration as shear stress for continuous flow is a power law function of solids concentration. Besides that, the thixotropic behavior would results in structural build-up of sludge over a long retention time in the mixing tank or reactors and form stagnant region if not sheared properly, which is undesirable. Hence, a good knowledge of thixotropic property is crucial to enable development of an efficient stirring or mixing mechanism to optimize the treatment process with minimum cost.

Figure 4: Change of viscosity over time when stress applied and removed for just shear-thinning material (black line) versus shear-thinning thixotropic material (red line), image courtesy to google

Several studies had highlighted the controversies of whether thixotropic property of sludge was existed or merely an erroneous interpretation. This property makes it extremely difficult to characterise sludge according to a specific rheometric technique (Seyssiecq et al. 2003; Mori et al. 2006). Hence, there is always inconsistency in literature in terms of sludge characterisation and behaviour (Seyssiecq, Ferrasse et al. 2003; Mori et al. 2006).

Tixier et al. (2003a,b) found that the area of the hysteresis loop varied according to the nature of sludge.

However, Baudez (2006) demonstrated that the hysteresis loop mostly comes from the rheological procedure and the accuracy of the rheometer.

That may explain why Seyssiecq et al. (2003) showed that few researchers had attempted to model the thixotropic characteristic of sewage sludge but was unsuccessful, while most of them merely mentioned this property in their studies to remind possible errors might exist in the rheological measurement.

Other characterization methods include step change in shear rate and shear stress as well as dynamic moduli, which are detailed elsewhere (Mewis and Wagner, 2009). These two methods were able to provide a basis to evaluate the thixotropic effect although the level of understanding of shear history dependence of microstructure is still limited (Mewis and Wagner, 2009).

Recently, Baudez (2004; 2008) has presented a new technique, which is the reconstruction of the velocity profile, to measure the dual rheological behavior of sewage sludge. In this work, he was able to model the behavior of sludge using a unique equation which consisted of a solid and liquid component as well as a structural parameter, λ, measured as a function of time, to characterize the time-dependency of sludge. This parameter had also been adopted by several other researchers to develop thixotropic model that can be used to characterize time-dependent behavior of yield stress fluids (Labanda et al., 2004; Dullaert and Mewis, 2005; Alexandrou et al., 2009; Mewis and Wagner, 2009; Livescu et al., 2011). It is defined as a measure of the degree of structure in the suspension, having a value in the range of zero (fully broken) to 1(fully structured) (Toorman, 1997). Several researchers had demonstrated the possibility to relate the structural parameter to the rheological parameters of non-Newtonian fluid, notably yield stress (Toorman, 1997) and viscosity (Labanda and Llorens, 2008). However, most of the models proposed are not readily used in sludge application as they are still in developing stage and has not been verified with experimental results. Most importantly, these models involve multiple variables, which are complex to solve, and required significant simplification to improve the practicability of these models.

In contrast to the large number of models that have been proposed, there are few systematic data that can be used to evaluate the thixotropy of sludge for model verification, which has seriously hinders the progress in this field. Recently several researchers had devoted their works to study the impact of sludge age on the sludge treatment operations, but did not present any correlations that could contribute to the characterization of the thixotropic property of sludge (Ekama, 2010; Çiğgin et al., 2011; Hocaoglu et al., 2011).

There has been a growing interest among researchers to develop a reliable model for thixotropic characterization of various yield stress materials, but not specifically for sludge. Currently, most of the models are general. The measurement accuracy of thixotropic properties is often met with skepticism from researchers as there are no consistent laboratory protocols, reliable devices or even established parameters that can characterize this property.

7. Viscoelasticity

Sludge exhibits viscoelasticity which means that it behaves as elastic solids and liquid and when the applied stress reduces to zero, a partial elastic recovery is observed (Figure 5). The partial recovery may be related to storage of energy in inter-particle bounds.

Figure 5: Elastic and viscous response of a viscoelastic material to applied and removed deformation, image courtesy to google

Under applied stress, the sludge will behave as solids initially, but as a liquid eventually due to the breakdown of floc structure. The viscoelastic properties are obtained through dynamic measurement by applying a sinusoidal deformation and measuring a sinusoidal stress (stress and viscous component) in response to deformation (Chhabra and Richardson, 2008). The storage modulus (G', ratio of elastic stress over strain) and loss modulus (G'', the ratio of viscous stress over strain) are corresponding to the amount of energy stored and dissipated during deformation. The effects of these two moduli are combined into the complex modulus$\left(G^* = \frac{\tau^*}{\gamma^*}\right) = G' + iG''$, which indicate the sludge's overall resistance to deformation (Ayol et al., 2006). When $G' > G''$, implies that elastic behavior is more dominant than viscous behavior and vice versa. They can be calculated from Eq. (35) (Seyssiecq, et al. 2003).

$$G^{"} = \frac{\eta\omega^{2}}{1+\lambda^{2}\omega^{2}}, G' = \frac{\eta\lambda\omega^{2}}{1+\lambda^{2}\omega^{2}} \qquad \text{Eq.35}$$

where ω is oscillation frequency and λ is structural parameter. A complete review on the concept of dynamic measurement with sinusoidal oscillations can be found in the work of Seyssiecq et al. (2003). At present, there is no consistent correlation that can relate the parameter in dynamic measurement to the rheological parameters in flow measurement.

Chen et al. (2005) has demonstrated that the complex modulus of sludge can be significantly affected the addition of coagulant polymer. The addition of polymer would cause all the sludge samples to form more rigid solids and therefore, storage modulus increases with increasing polymer dosing, which is consistent with the results obtained by (Wang, et al., 2011a). The authors believed that the variation in G^* due to polymer addition may be explained by change of network strength of floc caused by the formation of bridging between cationic polymers and negatively charged sludge particles. Frequency sweeps from the work of (Wang et al., 2011a) revealed that the G’> G” indicating that the elastic behaviour was dominant over the viscous behaviour until a critical point was reached then G” >G’. This trend was also present for conditioned sludge, however, it extended over the viscous region, suggesting that for unconditioned anaerobic digested sludge, the water hold capacity was greater and exhibited less elastic behaviour. The crossover from G’>G” to G”>G’ is similar to that of solids and pastes suggesting that sludge behaves in a similar manner. Wang et al. (2011a) also observed gel like behaviour for low viscosity sludges at high shear rates in the linear viscoelastic regions. They argued that more energy is stored in the rigid structure of the conditioned anaerobic digested sludge which increases its elasticity (G’). Ayol et al.(2006) also conducted dynamic measurements on conditioned and unconditioned sludge samples and found that the storage modulus was greater than the loss modulus in the linear viscoelastic range,and the loss modulus increased whilst the storage modulus decreased beyond the linear viscoelastic range.

The hydrogel property of granular sludge has been identified by Seviour et al. (2009a) through dynamic measurement. This work has established a protocol for characterization of granular sludge and revealed that the macromolecular association is responsible for the formation of granular sludge under various environmental conditions as well as the yield response, which can be useful to promote flocculation in wastewater treatment. Also, they have utilized this technique to explain the structure difference between aerobic sludge granules and floccular sludge based on the sol-gel transition of extracellular polymeric substance (EPS) derived from the sludge (Seviour et al., 2009b). Recently, Baudez et al. (2013a) have identified strong similarity of the viscoelastic behavior of anaerobic digested and raw sludge with soft glassy material using dynamic measurement. Elastic and loss modulus is constant in linear viscoelastic region and $G' > G''$ but at cross over point G'' reaches its peak, then $G' < G''$ which is the hallmark of soft-glassy materials. This showed that soft-glassy material can be used a model fluid.

Based on the literature reviewed, it is shown that the application of dynamic measurement in sludge characteristion have been restricted to evaluation of visco-elastic properties as well as yield stress determination. Besides that, the reliability of these experimental works is unsure as there are too few studies or results that can be used for evaluation. More researchers should incorporate this type measurement into their work to explore its application and potential in sludge characteristaion as it is complementary to a better understanding of sludge rheology in static mode. Dynamic measurement has proved to be a useful analysis method to determine the elastic properties of sludge, which can provide a meaningful insight to the technical matters, such as mixing and pumping, in the wastewater treatment process. With better understanding of the dynamic behaviour, engineers may incorporate this parameter into their design to improve the process efficiency.

8. Relationship between sludge rheology and physico-chemistry interaction

There is little understanding between the rheological properties and actual sludge physico-chemical behaviour. The works of Forster (1981;1982; 2002) illustrate the relationship between surface chemistry and rheological properties. According to Forster (1982; 2002), the non-Newtonian behaviour of sewage sludges is related to the materials surface chemistry, so the surface charge carried by each component. Forster (1981; 1982) studied activated, anaerobically digested and aerobically digested sludges and found that the relationship between surface charge and rheological properties is controlled by the ionic strength of liquoras well as the chemical nature of sludge surfaces. For activated sludge, Forster (1982) found that polysaccharides influenced the surface charge. Forster (1982) found that the viscosity was reduced by adding cellulose;hence, the influence of polysaccharide on surface charges is significant. Forster (1982) was unsuccessful in determining the relationship between surface charge and rheological properties for other types of sludge and emphasised the neccessity of research on the surface chemistry of sludge and its influence on the rheological properties. No model was developed to be able to describe the relation between surface charge and viscosity of activated sludge. However, in his 2002 study of the rheological and physico-chemical characteristics of sewage sludges, Forster was able to develop a rule that described the influence of surface charge (Eq.36) and water content (Eq.37) on yield stress (Forster 2002).

$$Surface\ charge = -a\ Ln(\tau_y) - b \qquad \text{Eq.36}$$

$$water\ content = a\ Ln(\tau_y) + b \qquad \text{Eq.37}$$

Where τ_y is the yield stress and a and b are model parameters.

Tixier et al. (2003a) have investigated the effect of surface charge on limiting viscosity of activated sludge by varying pH and the cation concentration (calcium and sodium ions). A smallr decrease in pH and cation concentration decreased limiting viscosity which indicates that the sludge particle surface charge affects viscosity. This intraction was shown through the linear correlation between zeta potential and limiting viscosity. They have suggested that the effect of pH variation on viscosity could be related to the change of repulsion forces between flocs and thickness of double layer, as indicated by the zeta-potential. This is inconsistent with Sanin (2002) observation and their conclusion that increasing PH increas negetive charge on flocs which increases repulsion and hence expansion of floc matrix. However, Mu et al. (2007) commented that the limiting viscosity of sludge did not respond well to pH variation. Recently, Li and Yu (2011) have commented in their review paper that this matter still remains contraversial whether limiting viscosity is sensitive to pH change or not.

The effect of cation concentration on limiting viscosity was shown to be in good agreement with the work of Sanin (2002) and Pevere et al. (2007) and may be related to the compression of double layer, change of electrostatic repulsion between sludge floc and the salt concentration in the suspension. Sanin (2002) also examined the influence of conductivity on the rheology of activated sludge. They observed thatincreasing conductivity decreased the apparent viscosity Sanin (2002) argued that this was due to the compression of the electrical double layer around particles which results in a more compact floc structure.

Mori et al. (2006) calculated the magnitude of the energy of cohesion (E_c) of the 3D network of sludge (Eq. 38). This energy was used to determine the extent of interaction in flocculated structure. This method requires dynamic measurements.

$$E_c = \frac{1}{2}\tau_{y,dynamic}\,\gamma_c \qquad \text{Eq. 38}$$

The dynamic yeild stress ($\tau_{y,dynamic}$) and energy of cohesion of the 3D sludge network (E_c) were found to be proportional ($\tau_{y,dynamic} = \alpha . E_c$) because γ_c is almost constant for different concentration of sludge. Mori et al. (2006) developed an empirical model to describe the relationship between the energy of cohesion of the 3D network of sludge and suspended solids concentration (Eq. 39).

$$E_c = a exp[b(TSS)] \quad \text{Eq.39}$$

where a and b are parameters.

9. Conclusion

Rheological measurements have proved to be of great importance to quantitatively estimate the physical consistency of sewage sludge, and impart important data for wastewater treatment process optimization and design. Of all the rheological properties, the characterization of sludge thixotropic property has been the most difficult measurements. Even though many models have been proposed for this, there is little consistent data that can be used to verify the models due to the lack of reliable methodology to measure this property. A review of the literature presents:

- Sludge is always non-Newtonian
- exhibits a yield stress or not,
- is shear-thinning and thixotropic.
- At high shear rate, sludge behaves as thixotropic colloidal suspension, but
- At low shear rate exhibits polymeric behavior.
- Sewage sludge at high solids concentrations (3-10%) behaves as a complex mixture whose rheological behavior is highly dependent on the treatment process it is undergoing
- A combined Herschel-Bulckley and Bingham model describes sludge behavior over the full range of shear rates

- Limiting viscosity and yield stress proved to be reliable rheological parameters for sludge characterization as they correlate well with physico-chemical properties of sludge, and solids concentration.
- To ensure the consistency of characterization methods and tools used in sludge research, a laboratory protocol should be developed to help maintaining the uniformity of data presented in the publications and enable researchers to directly compare their experimental results and examine the validity of the methodology used for their investigation. Hopefully with this, it is possible to accelerate the development of research in sludge characterization and achieve a better understanding of sludge behavior to optimize all the operations that involve sludge.

Acknowledgment:

The authors are grateful for financial support which provided by RMIT Research and Innovation Office, Melbourne Water and South-East Water companies.

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