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Graphical abstract:

Anaerobic digestion is a key treatment process for solids treatment and pathogen reduction. Due to the inherent opacity of sludge, it is impossible to visualize the mixing and flow patterns inside an anaerobic digester. Therefore, choosing an appropriate transparent model fluid which can mimic the rheological behaviour of sludge is imperative for visualization of the hydrodynamic functioning of an anaerobic digester.

CLEAR MODEL FLUIDS TO EMULATE THE RHEOLOGICAL PROPERTIES OF THICKENED DIGESTED SLUDGE

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ABSTRACT

Optimizing flow processes in wastewater treatment plants requires that designers and operators take into account the flow properties of the sludge. Moreover, due to increasingly more stringent conditions on final disposal avenues such as landfill, composting, incineration etc., practitioners need to produce safer sludge in smaller quantities. Anaerobic digestion is a key treatment process for solids treatment and pathogen reduction. Due to the inherent opacity of sludge, it is impossible to visualize the mixing and flow patterns inside an anaerobic digester. Therefore, choosing an appropriate transparent model fluid which can mimic the rheological behaviour of sludge is imperative for visualization of the hydrodynamic functioning of an anaerobic digester.

Digested sludge is a complex material with time dependent non-Newtonian thixotropic characteristics. In steady state, it can be modelled by a basic power-law. However, for

short-time processes the Herschel-Bulkley model can be used to model liquid-like properties.

The objective of this study was to identify transparent model fluids which will mimic the behaviour of real sludge. A comparison of three model fluids, Carboxymethyl Cellulose (CMC), Carbopol gel and Laponite clay revealed that these fluids could each model certain aspects of sludge behaviour. It is concluded that the rheological behaviour of sludge can be modelled using CMC in steady state flow at high shear rates, Carbopol gel for short-time flow processes and Laponite clay suspension where time dependence is dominant.

Keywords: Thickened municipal digested sludge, rheology, thixotropic properties, Short time process, Steady state operation, model fluids,

1. Introduction

Optimal and efficient design and operation of waste water treatment plants requires accurate prediction of the hydrodynamic functioning of the associated plant such as pumps, heat exchangers and anaerobic digester mixing. Accurate flow behaviour prediction of these engineering hydrodynamic processes requires the rheological characteristics of the sludge as input (Slatter, 2011).

Since the beginning of the seventies with the pioneering work of Colin (1970), and Bhattacharya (1981), rheological measurements have proved to be of great interest to quantitatively estimate the physical consistency of sewage sludge. Sewage sludge is often

seen as a complex mixture. The literature presents a wide variety of results: sludge is always non-Newtonian (Campbell and Crescuollo, 1982), exhibits a yield stress (Baudez and Coussot, 2001; Mori et al., 2006) or not (Valioulis, 1980), is shear-thinning (Chaari et al., 2003) or thixotropic (Tixier et al., 2003). Most of the rheological models used to describe sludge behaviour are a simple power-law (Moeller and Torres, 1997), the Bingham model (Sozanski et al., 1997; Guibaud et al., 2003) or the Herschel-Bulkley model (Slatter, 1997; Baudez, 2001). The Sisko model, Cross viscosity model, truncated power law model (Baudez, 2008) have been also used. Slatter (2008) has shown that the sludge rheology plays a fundamentally important role in analysing the hydrodynamic behaviour of the sludge, as it flows through the tertiary treatment process.

$$\tau = K \dot{\gamma}^n \quad \text{Eq.1}$$

$$\tau = \mu_B \dot{\gamma} + \tau_y \quad \text{Eq.2}$$

$$\tau = K \dot{\gamma}^n + \tau_y \quad \text{Eq.3}$$

$$\tau = \mu_\infty \dot{\gamma} + K \dot{\gamma}^n \quad \text{Eq.4}$$

$$\frac{\mu - \mu_\infty}{\mu_0 - \mu_\infty} = \frac{1}{1 + K \dot{\gamma}^m} \quad \text{Eq.5}$$

$$\frac{\tau}{\tau_c} = \left(\frac{\dot{\gamma}}{\dot{\gamma}_c} \right)^n \quad \text{Eq.6}$$

A power-law fluid (Eq.1) is a type of generalized non-Newtonian fluid, where τ is the shear stress, $\dot{\gamma}$ is the shear rate, 'K' is the fluid consistency index, 'n' is the flow behaviour index. The pseudoplastic model can be used to model the shear thinning zone of

the rheogram of sludge suspensions (an intermediate shear rate range). In Bingham plastic model (Eq.2), sludge is rigid when shear stress (τ), has a value less than a critical value τ_y . Once the critical shear stress (or "yield stress") is exceeded, the material flows. The Herschel-Bulkley model (Eq.3) compared to the power-law model, the additional term yield stress (τ_y) quantifies the amount of stress that the fluid may experience before it yields and begins to flow. The physical reason for this behaviour is that sludge may contain particles or large molecules with have some kind of interaction, creating a weak solid structure and a certain amount of stress is required to break this structure and produce flow. Once the structure has been broken, the particles move with the liquid under viscous forces. If the stress is removed, the particles associate again (Chhabra and Richardson, 2008). This process also exists under shear at low shear stress (Tabuteau et al., 2006; Baudez, 2008). The advantage associated with employing the Herschel-Bulkley fluid model (Eq.3) describes both pseudoplastic (rheogram curvature) as well as the Bingham model (yield stress) (Slatter, 1999); however, the model does not take into account the Newtonian asymptotes at low and high shear rates (Slatter, 1999). At high shear rate, the model suggested by Baudez et al. (2011a), can be seen as a start to improve rheological characterisation of sludge. Both Bingham plastic and Herschel-Bulkley models can characterize the sludge behaviour from rest to flow. It is also important to note that in some cases, sludge may present slight thixotropic behaviour (Tabuteau et al., 2006, Terashima et al, 2009), which causes hysteresis on flow curves although (Baudez, 2006) demonstrated that hysteresis loop inside the rheogram cannot be used to define the thixotropic character

of a sludge. This is most frequently explained by the time-dependent disintegration of internal structure of a liquid suspension as a result of the application of shear stress.

The Sisko model (Eq.4) can be used to characterize the flow behaviour of sludge in the intermediate and high shear rates range, where the apparent viscosity tends to a limiting value (plateau) (Mori et al. , 2006). The Cross viscosity fluid model (Eq.5) is a four parameter model that relates the fitting parameters K (parameter with dimension of time) and m (dimensionless material parameter) to the zero shear viscosity, μ_0 and infinite shear viscosity, μ_∞ (Sybilski, 2011). Eq.5 is further simplified and presented in Eq. 6 because high shear rates cannot be accurately measured and thus the value of μ_∞ cannot be determined; it can also be assumed that $\mu > \mu_\infty$ (Sybilski, 2011).

$$\mu = \frac{\mu_0}{1 + K\dot{\gamma}^m} \quad \text{Eq.6}$$

Baudez (2008) defined a critical shear stress, $\dot{\tau}_c$ (with corresponding critical shear rate, $\dot{\gamma}_c$) which below that the solid structure rebuilds even under shear. In fact, below the critical shear stress, colloidal forces tend to rebuild the solid structure (physical aging) and shearing forces tend to break the solid structure (shear rejuvenation). As soon as the critical shear is reached, the solid 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 (Eq.6) model. He also correlates the higher degree of thixotropy of the material to stronger interactions between the solid particles.

Sludge rheology is complex and always evolves with time due to ageing and microbial activity. The time-dependent characteristics of sludge rheology make the measurement of physical parameters difficult and the results obtained are often unreliable because of this. Therefore it cannot be used as a reference material for industrial process design or controlled experiments. As a result of this, researchers have been trying to find a suitable transparent model fluid that mimics the behaviour of sludge. Most studies have focussed on the characterisation and modelling of activated sludge, but there are very few studies on digested sludge. For activated sludge, the proxy materials that have been studied so far include kaolin suspension for the yield stress determination (Spinoso and Lotito, 2003) polymeric gels (Legrand et al., 1998), polyvinyl chloride (PVC) suspensions (Bongiovanni, 1998) and polystyrene latex (Sanin and Vesilind, 1996; Örmeci and Vesilind, 2000).

To model the flow properties of activated sludge in liquid regime, kaolin suspensions are often used (Héritier et al., 2010) due to its similarity in rheological behaviour and can be described by a Herschel-Bulkley model (Baudez, 2001; Masalova et al., 2006). Sanin and Vesilind (1996) reported a synthetic sludge made up of polystyrene latex particles and polysaccharide-alginate. Their experiments demonstrated that it is possible to create chemical surrogate sludge by using bacteria like particles, extracellular resemble polysaccharides, and cations common to activated sludge at typical quantities. However, their synthetic sludge shows poor flocculation behaviour due to the absence of filamentous microorganism which is the backbone for overall floc structure in activated sludge (Nguyen et al., 2007a). Örmeci and Vesilind (2000) improved the characteristic of synthetic sludge by adding cellulose fibres to simulate the filamentous microorganisms in activated sludge,

which results in stronger floc formation and better dewaterability as well as settling, though the fibres are hard to dissolve in water (Baudez et al., 2007). They observed that both calcium ions and alginate are important to promote the floc formation in synthetic sludge, which is later confirmed by Nguyen et al. (2007a; 2007b; 2008,). Dursun et al. (2004) compare the physical characteristic of the synthetic sludge to activated sludge. They noted that the synthetic sludge may be an adequate surrogate in terms of electrokinetic and rheological properties but does not duplicate the properties and conditionability of activated sludge. Therefore the application of this synthetic sludge is only valid for qualitative or mechanics studies. Baudez et al. (2007) suggested that it is necessary to consider granular arrangement by adding micro-aggregates (Jorand et al., 1995) into synthetic sludge as real sludge does not contain individual bacterial cells only.

Baudez et al. (2007) commented that standard methodologies should be developed to prepare synthetic suspensions that mimic behaviour of both inorganic and organic sludge for lab testing. They showed that a mixture of kaolin and calcite/quartz sand in water with relative ratio ranges from 90/10% to 75/25% was able to well describe the behaviour of real inorganic sludge. As for organic sludge, it is developed based on the recipe by Müller and Dentel (2002) and Dursun et al. (2004) and consists of alginate, cellulose, yeast, bovine, serum albumin, stearic acid, and calcium as well as potassium ions in water. However, preparation of such recipe could be difficult as it requires one to have extensive knowledge on surface chemistry and colloidal system to ensure the synthetic sludge behaves exactly the same as the real sludge. Also, rheological measurement needs to be performed on the synthetic sludge to examine its behaviour to compare with activated sludge.

153

154 It is known that anaerobic digestion is a key treatment process for solids treatment, and
155 odour and pathogen reduction. Due to the inherent opacity of sludge, it is impossible to
156 visualize the mixing and flow patterns inside an anaerobic digester. Therefore, choosing an
157 appropriate transparent model fluid that can mimic the rheological behaviour of sludge is
158 imperative for visualization of the hydrodynamic functioning of an anaerobic digester. The
159 idea of model fluid for sludge is to find a material that is less complicated but at the same
160 time still be able to simulate the sludge behaviour. There is no study to find the
161 representative, safe and non-smelly fluid which could mimic the rheological behaviour of
162 sludge. Rheological properties of Carbopol, CMC and Laponite have been investigated in
163 the past decades for various applications in food science and surface coating but very few
164 compared their properties to the behaviour of sludge. Baudez et al. (2011b) recently have
165 examined that the similarity between soft glassy material and sludge, based on its dynamic
166 visco-elastic behaviour, i.e. the storage modulus (G') and loss modulus (G''), in the
167 temperature range of 10 to 60°C using strain and stress sweep tests. This work provides the
168 basis for future work in sludge characterisation utilizing the known characteristic of soft
169 glassy materials. Bonn (1999; 2002) has shown that Laponite is soft glassy material. This
170 might be because Laponite is also a complex fluid like sludge, which complicates the
171 rheological analysis. The experimental results obtained here do indicate some similarities
172 between the rheological behaviour of Laponite and sludge under the influence of solid
173 suspension. But due to thixotropic behaviour of Laponite, in order to study the yield stress
174 effect of sludge, for example, on mixing system, a simple system is required. Coussot et al.

(2009) also highlighted that the Carbopol gel is a type of material which can be an excellent model of yield stress fluid. Therefore, Carbopol has been chosen to study for yield stress effect of sludge. CMC sample also is a good example of fluid with no yield stress which can be used to study the sludge property once a critical stress was applied and the yield stress of sludge overcome. This paper aims to identify model fluids which will mimic the behaviour of real sludge. These materials are widely available, non toxic and less complicated to handle when use for sample preparations. Besides that, the rheological properties of these materials have been well documented and therefore provide a strong database to be adapted for sludge characterization. This enable a standard methods for sludge characterization, as proposed by European Committee for Standardization (CEN) (Spinosa, 2001), to be established so that technical language, methods and practice used among researchers across the world for sludge characterization can be synchronized. For this purpose the flow property and thixotropic behaviour of real sludge were measured then by measuring similar property in model fluids, the model fluid could represent sludge rheological behaviour in high shear, low shear. The best fit rheological model for experimental data of model fluids and sludge is also presented.

2. MATERIAL AND METHODS

2.1 Material:

The digested sludge obtained from Mount Martha waste water treatment plant with total solid suspended of 1.65% was concentrated to 3.23% TSS via vacuum filtration (Buchner Funnel) for 6 hours. The model fluids which were used in this study were CMC, Carbopol, and Laponite. Carboxymethylcellulose sodium salt (CMC) purchased from Sigma Aldrich

(M_w 700 000, D.S 0.9). Various aqueous CMC solutions were prepared by dissolving the correct amount of CMC in deionised water at room temperature with continuous stirring using an impeller mixer (Eurostar, power control Visc (Mixer)) at 1000 RPM. Carbopol ®940 purchased from ACROS ORGANICS. Laponite RD purchased from Southern Clay Products. Various concentrations of Carbopol were prepared by dissolving the correct amount of Carbopol in deionised water at room temperature with continuous stirring using an impeller mixer (Eurostar, power control Visc (Mixer)) at 1000 RPM. In order to neutralize these Carbopol solutions, 1M NaOH was added in a drop like manner whilst the pH was simultaneously measured until a pH of 7 was reached. 1.7, 9.2, 19.0, 28.6 mL NaOH are required neutralizing 200g of 0.1, 0.5, 1, and 1.5% Carbopol, respectively. The concentration of Laponite was prepared by dissolving the specific amount of Laponite powder into the corresponding amount of tap water whilst continuous mixing it simultaneously. After 48hr, the Laponite suspension made a gel structure.

2.2 Method:

The flow curve for thickened digested sludge at $25(\pm 1.0)^\circ\text{C}$ was determined using the Dynamic Stress Rheometer (SR200, Rheometrics) fitted with a couette geometry (inner diameter: 29mm, outer diameter: 32mm, length: 44mm). A constant shear stress was first imposed in order to pre-shear the sludge for 15 minutes and a relaxation time of 1 minute was allowed. The flow curve was then obtained by imposing an increasing stress that coincided with a shear rate less than 1000s^{-1} . This measurement was repeated for 3 different samples coming from the same withdrawal at the WWTP in order to determine the consistency of the results. The flow curve for model fluids at a constant temperature

(25 ± 1.0 °C) were carried out by applying a shear stress within the range of 0.017 to 20 Pa for CMC and 30 to 300 Pa for Carbopol using the SR 200 rheometer. For higher shear rate, shear rate controlled rheometer (Rheologica instruments AB, Scientex) with the couette geometry (gap diameter = 3.0 mm) for 50 to 500 RPM range was used as the SR200 was reached to its upper limit for torque measurement in the high shear rate range.

The thixotropic property of the concentrated digested sludge was measured at 25°C by applying a pre-shear (corresponding to 200s^{-1}) for a duration of 15 minutes. Then without relaxation time, a first shear stress corresponding to a shear rate of 100s^{-1} was applied for 15 minutes; a second shear stress corresponding to 5s^{-1} was then applied and the first shear stress was reapplied, each for duration of 15 minutes. The resulting shear rate versus time graph was analyzed in order to describe the thixotropy of digested sludge. The thixotropic property of Laponite at a constant temperature of 25 ± 1.0 °C, was measured the same way of sludge measurement by applying a pre-shear (corresponding to 400s^{-1}) for 15 minutes then a first shear stress corresponding to a shear rate of 350s^{-1} for 15 minutes; a second shear stress corresponding to 100s^{-1} for 15 minutes and reapplying the first shear again for another 15 minutes. The resulting shear rate versus time graph was analyzed (Amemiya and Shoemaker, 1992; Baudez, 2008).

3. RESULT AND DISCUSSION

The viscosity flow curve for thickened digested sludge at 3.23% is presented in Figure 1. As illustrated in Figure 1, this sludge sample can be modelled with Herschel-Bulkley model with shear thinning behaviour which is consistent with other researcher's observation

(Chaari et al., 2003). The parameters of model are $\tau_y = 1.504 Pa$, $K=0.296 Pa.s^n$, and $n=0.606$. Figure 2 shows the thixotropic characteristic of thickened digested sludge at 25°C.

Fig.1: Viscosity curve of thickened digested sludge with 3.23%TSS at 25°C

Fig.2: Thixotropic behaviour of thickened digested sludge with 3.23%TSS at 25°C

3.1 Similarity of sludge rheological behaviour during steady state operation to the rheology of CMC solution

If only high shear rate range (above $20s^{-1}$) of the sludge viscosity curve (Fig.1) is to be considered, one straight line or in another word the power law model is adequate for modelling experimental data. Therefore, sludge at high shear rates and at steady state operation can be modelled similar to CMC at shear rate higher than $10 s^{-1}$ with a power-law model although Cross model decres whole shear rate range of CMC solution. Figures 3 show the viscosity curves of different CMC concentrations, respectively.

Fig.3: Viscosity Curves of different CMC concentrations

Figure 3 was obtained by combining both SR200 and Rheologica data. But before merging these data, it is required that the raw data from the rheometer to be corrected for the effect of non-Newtonian flow behaviour in the wide gap measurement technique. The true shear rate was determined through the technique described by Coussot (2005). After finding true shear rate and shear stress of fluid in the wide gap of couette geometry, the data was combined with the raw data of SR200 and then the Cross viscosity model (Sybilski, 2011)

was used to describe the flow curve of CMC. In order to determine the Cross model parameters, the error between the predicted viscosity (using the Cross Model) and the measured viscosity according to Eq.7, was minimised. Table 1 shows the Cross model viscosity parameter for various CMC concentrations.

$$SSE = \frac{(\mu_{Predicted} - \mu_{Measured})^2}{(\mu_{Measured})^2} \quad \text{Eq.7}$$

Tab.1: Summary of Cross Model viscosity parameters

CMC (Wt.%)	m	K	μ_0
0.5	0.633	0.024	0.373
1	0.603	0.273	3.322
1.5	0.309	2.313	36.250

Figure 3 demonstrates that CMC exhibits shear thinning behaviour similar to sludge at high shear rate and follows the Cross model for power law fluids (Barnes, 1999). The error associated with measured data and Cross model calculations is relatively low suggesting that the Cross model can be used to model the fluid properties of aqueous CMC solutions at low concentrations (Benchabane and Bekkour, 2008). The plateau illustrated in Figure 3 suggests that the fluid behaves as a Newtonian fluid at low shear (Barnes and Walters, 1985), and also behaves as a non-Newtonian fluid following the power law model at higher shear rates.

3.2 Similarity of sludge rheological behaviour during short time process

operation to rheology of Carbopol fluid

The issue of whether yield stress really exists is still debatable until today. The main reason is that no equipment, so far, allows researchers to measure the shear stress of sludge at very low shear rate without being affected by the wall-slip or end effects. Besides that, the concept of yield stress is not well-defined. There is variation in terms of rheological model and experimental method used among researchers to determine yield stress points 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 necessarily indicate that the sludge is a yield stress fluid (Barnes, 1999). Based on a review paper by Seyssiecq et al. (2003), with apparatus being more advanced, it is commonly admitted among researchers that yield stress does exist in aggregated concentrated sludge.

Yield stress is defined as minimum stress that needs to be applied for a material to flow steadily. It is generally agreed among researchers that yield stress tends to increase as the solid concentration of sludge becomes higher, even for pre-treated or conditioned sludge (Mikkelsen, 2001; Forster, 2002; Seyssiecq et al., 2003; Spinosa and Lotito, 2003; Wilen et al., 2003, Khongnakorn et al., 2010;)

The viscosity curve for Carbopol in Figure 4 is plotted by using data obtained from SR200. The Herschel-Bulkley model was fitted to the data and its parameters are presented in Table 2. It is evident that Carbopol at the studied concentrations follows the Herschel-Bulkley Fluid model with shear thinning behaviour as illustrated a value below one for 'n'. A

critical yield stress must be applied in order for Carbopol to flow, which is evident for all Carbopol concentrations; this also increases with increasing concentration. Coussot et al. (2009) also highlighted Carbopol gel as a type of material which can be an excellent model for yield stress fluid. Therefore Carbopol will be a suitable material for studying the yield stress effect on sludge for different short time process such as pumping.

Tab.2: Rheological properties of Carbopol using the Herschel-Bulkley fluid Model

Carbopol (Wt. %)	n	K	τ_y
0.1	0.513	3.2	24.50
0.5	0.502	27.50	118.33
1.0	0.484	37.20	143.20
1.5	0.456	53.67	169.33

Fig.4: Viscosity curves for 0.1% ($\diamond$), 0.5% (Δ), 1.0% ($\square$) and 1.5 % ($\circ$) Carbopol samples

This study's observation is consistent with the results of the other researchers (Curran et al., 2002; Putz and Burghilea, 2009; Gomez et al., 2010,).

These results are compared with other researchers who also have investigated the rheological properties of Carbopol in their works in various applications (Table 3). It is agreed among researchers that the Herschel-Bulkley model fluid is a suitable model to describe the behaviour of Carbopol solution at various concentrations (Roberts and Barnes,

2001; Kim et al., 2003; Coussot et al. 2009; Gomez et al., 2010), but none of them use Carbopol as model fluid for sludge.

Tab.3: Rheological characterization of Carbopol for various applications

Author	Year	Application	Suspension concentration (%)
Robert and Barnes	2001	Examine slip effect on Carbopol dispersion	0.045 – 1.00
Kim et al.	2003	Evaluate viscoelastic property of Carbopol	0.1 – 4.0
Gomez et al.	2010	Model fluid for pulp mixing	0.1 – 1.5

In general, behaviour of Carbopol may seem similar to sludge as it is shear thinning and exhibit yield stress. As suspension concentration increases, both viscosity as well as yield stress of sludge and Carbopol increases. However, it is well known that Carbopol is a more stable system, i.e. non-thixotropy (Moller et al., 2009) when compare to sludge, therefore making it suitable to be used as a model fluid for sludge when thixotropic property is not a major concern.

3.2 Time characteristic of sludge

Figure 2 demonstrates that the property of sludge depends on shear history and processes that the sludge undergoes. Indeed, thixotropy demonstrates time-dependency of sludge structural changes at a given shear rate which is indication of a competition between flocculation and de-flocculation (Baudez, 2008). In practice, there is a possibility of

clogging the sludge transportation pipeline if the wall shear stress is to be below critical shear stress (yield stress). Because at shear stress below yield stress, the earlier deflocculated sludge will re-flocculates , and solid characteristic becomes predominant during shear (Baudez, 2008) and gradually solid deposition builds up from wall to the centre of the pipe. In Figure 2, there is 17% difference between shear rate data obtained for the same imposed shear stress (at the first and final stage) corresponding to 100 s^{-1} shear rate.. This indicates that the sludge flocculated again when the shear rate dropped from 100 s^{-1} to 5 s^{-1} and for this reason, the response for final and the same shear stress is not the same as of the first one. Because before the first shear at 100 s^{-1} , sludge undergoes a pre-shearing stress at 200 s^{-1} , but before the final shear at 100 s^{-1} , sludge undergoes shear at 5 s^{-1} , which demonstrates the thixotropic behaviour of the sludge. This characteristic of sludge is important when sludge undergoes different shearing stress. For example, there is a difference between the shearing stress in a heat exchanger and inside of anaerobic digester. Therefore, it is important to consider thixotropic property (flocculation and de-flocculation) of sludge for all engineering design such as pipeline design for pumping or heating purpose in order to prevent clogging; mixing system design in order to prevent deposition of sludge at the bottom of tank. Using external heat exchangers is common in wastewater treatment plant in order to keep anaerobic digester temperature constant at 37°C or 55°C depending on mesophilic or thermophilic condition. Thus, choosing a clear model fluid which can capture the time dependent characteristics of the sludge in the anaerobic digester is important.

The viscosity curve of Laponite clay can also be modelled with the Herschel-Bulkley model which is presented in Figure 5. Like Carbopol, Laponite presents shear thinning behaviour ($n < 1$) with a critical shear stress and rate which below that there is no flow. In Figure 5, it is evident that Laponite viscosity increases as the suspension concentration is increased. Table 5 demonstrates that the parameters of this model (‘K’, ‘n’ and τ_y).

Fig. 5: Viscosity curve for different concentration of Laponite: 1.0% (●), 2.0% (X), 2.5% (○), 3.0% (Δ), 3.5% (◇) and 4.5 % (□)

Tab.5. Rheological properties of Laponite using the Herschel-Bulkley fluid Model

Laponite (Wt. %)	n	K	τ_y
1.0	0.90	0.01	0.001
2.0	0.38	0.78	0.587
2.5	0.36	2.06	3.205
3.0	0.34	2.41	17.614
3.5	0.32	3.96	29.818
4.5	0.32	5.2	77.667

Figure 6 clearly shows that the Laponite possesses thixotropic properties. In this paper, it is observed that the thixotropic effect cannot be correlated to the rest time or even suspension

concentration. There is no clear trend or pattern in suggesting whether thixotropy becomes more apparent or not as the rest-time or suspension concentration is increased. Also it is noticed that the thixotropic test results are not reproducible and may vary by several orders of magnitude even if the experimental conditions were to remain the same. The thixotropic effect of Laponite might not be apparent over a short rest time difference. Many researchers have investigated the thixotropic properties of Laponite on daily basis or over the course of a longer period (Joshi et al., 2008; Labanda and Llorens, 2008) based on the change in rheological properties (Knabel et al., 2000; Labanda and Llorens, 2008).

Fig.6: Thixotropic behaviour of Laponite with 30 minutes rest time between each shear:

1.0% (X), 2.0% (Δ), 2.5% ($\circ$), 3.0% ($\diamond$) and 3.5 % ($\square$) and 4.5 % (--)

It seems that Laponite has proved to be a better model fluid for sludge compared to Carbopol as it can capture both the yield stress and thixotropic properties of digested sludge, as well as presenting with similar rheogram curvature, as is demonstrated in Figure 7. Unlike Carbopol, Laponite may be a temperature sensitive fluid, as indicated by the work of Shukla and Joshi (2008), which is important as sludge rheology is affected by thermal history (Baudez et al., 2011b). Nevertheless, further investigations need to be done to examine the effect of physical chemistry such as surface charge or temperature on the rheology of Laponite and compare it to sludge.

Fig.7: Comparative flow curves of all model fluids a) Carbopol, b) Laponite and c) CMC)

with 3.23% digested sludge

389

390 **4. CONCLUSION**

391 A thickened digested sludge has been rheologically characterized and the results showed
392 shear-thinning with time dependent (thixotropic characteristics) behaviour for samples
393 taken from one of Melbourne's wastewater treatment plants. Candidate transparent fluid
394 model materials to mimic the sludge behaviour were also rheologically characterised. In
395 comparison between real sludge and model fluid, it was concluded that the real sludge in
396 steady state and at high shear rate operation such as pipe flow, mixing, aeration can be
397 modelled by a basic power-law approach, similar to CMC. Also, Carbopol can be a good
398 proxy for real sludge for short-time processes such as pumping and its liquid-like property
399 can be modelled with the Herschel-Bulkley model. The time characteristic of sludge can be
400 modelled using Laponite clay, but further study is required at different temperatures in
401 order ensure that Laponite can emulate behavioural changes due to temperature variation.

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Research highlights:

Real sludge can be modelled:

- As non-Newtonian shear thinning thixotropic material
- In steady state, and at high shear rate operation with a power-law similar to CMC
- In short-time processes such as pumping with carbopol and the Herschel-Bulkley model
- With laponite for thixotropic property characterization

Fig.1: Viscosity curve of thickened digested sludge with 3.23% TSS at 25°C

Fig.2: Thixotropic behaviour of thickened digested sludge with 3.23% TSS at 25°C

Fig.3: Viscosity Curve of different CMC concentrations

Fig.4: Viscosity curves for 0.1% (◇), 0.5% (Δ), 1.0% (□) and 1.5% (○) Carbopol samples

Fig. 5: Viscosity curve for different concentration of Laponite: 1.0% (●), 2.0% (X), 2.5% (○), 3.0% (Δ), 3.5% (◇) and 4.5 % (□)

Fig.6: Thixotropic behaviour of Laponite with 30 minutes rest time between each shear: 1.0% (X), 2.0% (Δ), 2.5% (○), 3.0(◇) and 3.5 % (□) and 4.5 % (---)

(a)

(b)

(c)

Fig.7: Comparative flow curves of all model fluids a) Carbopol, b) Laponite and c) CMC) with 3.23% digested sludge