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Rheological characterisation of thermally-treated anaerobic digested sludge: Impact of temperature and thermal history

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Abstract

This study investigated the partially irreversible effect of thermal treatment on the rheology of digested sludge when it was subjected to temperature change between 20°C and 80°C and then cooled down to 20°C. The yield stress, infinite viscosity and liquor viscosity of sludge were measured at 20°C for different thermal histories and were compared to the evolution of the solubilised chemical oxygen demand (COD) of sludge liquor.

The results showed that thermal history irreversibly affects sludge rheology as the yield stress of sludge which was heated to 80°C then cooled down to 20°C was 68% lower than the initial yield stress at 20°C. This decrease was due to the irreversible solubilisation of solid matter during heating as underlined by soluble COD data which did not reach its original level after thermal treatment. Measured soluble COD of sludge which was heated and cooled down was much higher than the soluble COD of initial sludge. We found a proportionality of the increase of soluble COD with the decrease of the yield stress as well as increase of infinite viscosity.

Keywords: digested sludge rheology, infinite viscosity, soluble COD, composition change, yield stress

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1. Introduction:

Due to tightening environmental regulations the volume of solid residue from wastewater treatment is increasing. This wastewater solid residue or in other word sludge is an odorous semisolid material which contains organic, inorganic and microorganism and needs to be treated to reduce the adverse effect of its constituents to the environment.

Anaerobic digestion is one of the most important processes for reducing sludge volume by reducing of about 30% of the organic load (part of the solid organic matter is converted into gas) and for producing biogas. The process operates either at 35-37°C or at 55-58°C and sludge needs to be heated by external heat exchangers or direct steam injection. Also, every 3 or 4 hours the anaerobic digested sludge inside the digester is mixed with raw sludge which most likely will be at a different temperature compared to digested sludge (Sanin et al. 2011). Thus the temperature-dependant rheological characteristics of sludge are important in design and operation of digesters.

Sludge is a non-Newtonian, temperature dependent suspension of microorganism, organic and inorganic chemicals which form suspended flocs in watery medium. Literature showed that sludge is a shear thinning dispersion (Dick and Ewing 1967, Manoliadis and Bishop 1984, Lotito et al. 1997, Monteiro 1997, Sozanski et al. 1997, Baudez et al. 2011). Many researchers have been used Herschel-Bulkley model (Monteiro 1997, Baudez et al. 2011, Slatter 1997, Eshtiaghi et al. 2012) to describe rheological behaviour of sludge.

$$\tau = \tau^H + \dot{\gamma}^n \quad (1)$$

Where τ is shear stress (Pa.s), $\dot{\gamma}$ is shear rate (s^{-1}), τ^H is the Herschel-Bulkley yield stress (Pa.s), K and n are flow consistency and flow behaviour index, respectively.

A temperature-dependence of sludge rheological characteristics has been reported by several authors (Sozanski et al. 1997, Battistoni et al. 1993a, Mu et al. 2007, Abu-Jdayil et al. 2010, Baudez et al. 2013, Baroutian et al. 2013). An Arrhenius-like model is widely used to describe the relationship between temperature and rheological parameters (Baudez et al. 2011, Battistoni et al. 1993a, Mu et

al. 2007, Pevere et al. 2009). However, in a recent paper, Baudez et al. (2013) objected these results. They showed that the rheological behaviour of sludge is irreversibly altered by thermal history since the initial yield stress decreased after cooling the heated sludge. As they reported the infinite (Bingham) viscosity -of 3.2% sludge with thermal history at 60°C and 80°C increased almost 65% and 400%, respectively. They suggested that the sludge composition may be altered by temperature and may explain the observed evolution of rheological characteristics.

Based on a literature survey; these authors assumed their observations may result from a conversion of solid to dissolved constituents, a process which is partially irreversible. In order to confirm this assumption and improve on previous studies which mainly focused on direct effect of temperature, this paper investigated the effects of temperature and thermal history on the rheological properties of digested sludge as well as sludge composition. The digested sludge viscosity curve, yield stress and soluble chemical oxygen demand (COD) were measured at different temperatures and for different duration of heating. The rheological and composition characteristics of sludge with and without thermal history were compared. It was observed that thermal treatment of digested sludge induced a transfer of the organic matter from the solid compounds to the dissolved constituents and this transfer is proportional to the evolutions of the yield stress and liquor viscosity.

2. Materials and Methods

Digested sludge was collected at the Mount Martha waste water treatment plant (Melbourne, Victoria, Australia) and carried in sealed containers. To reduce the impact of microbial activities inside the sludge, the samples were stored at 4°C for 30 days before experiments. This procedure helps with stability of samples which results in reproducible data for several days (Curvers et al. 2009).

To measure the dry solids content (wt.% DS) of the sludge, the sample was heated at 105°C for at least 24hours to reach a constant weight. The residue was then quickly weighted to avoid absorbing

74 water. By burning the sample to 605°C, then the mineral dry solids content (MDS) of sludge was
75 acquired (Appels et al. 2010). Table.1 provides sample specifications data.

76 Rheological measurements were performed with a stress-controlled rheometer (ARG2) from TA
77 INSTRUMENTS which was equipped with a cup and bob geometry (inner diameter: 29mm, outer
78 diameter: 32mm, length: 44mm). The temperature varied between 20 to 80°C using a water bath
79 connected to the rheometer. The following procedure pre-set for all rheological tests: low-speed
80 mixing to reach the desired temperature, 5min pre-shearing at the highest shear rate of actual test to
81 erase material history (Baudez et al., 2011), following with 1min equilibrium at zero shear rate, and a
82 decreasing ramp of shear stress (starting at a shear stress corresponding to a shear rate of 1000s⁻¹)
83 was applied with duration of 10s for each point of measurement. This procedure prevents transient
84 effects on result (Mezger 2006). To prevent evaporation a thin layer of oil, which is not soluble with
85 sludge, was used for covering sludge (Baudez et al., 2011). For determination of yield stress, the
86 procedure was repeated with an increasing ramp of shear stress from sufficiently low shear stress to
87 the shear stress corresponding to a shear rate of 10s⁻¹. The yield point was found on strain-stress
88 graph where the viscoelastic deformation behaviour ended and irreversible deformation or viscous
89 flow began.

90 The flow curve was initially measured at 20°C then the sludge was heated to different temperatures
91 (50, 60, 70 and 80°C). For each test, the temperature was kept constant for 30min while the flow
92 curve was measured after 1, 15 and 30 min of thermal treatment. Then, the sludge was cooled down
93 to 20°C and the flow curve was determined again.

94 The effect of thermal treatment on sludge composition has been studied by Appels et al.(2010), and
95 Paul, Camacho et al. (2006). These studies showed that low-thermal treatment of digested sludge
96 (<100°C) results in organic matter solubilisation representing by chemical oxygen demand (COD).
97 Paul, Camacho et al. (2006) reported that the maximum of solubilisation yield can be achieved in the
98 first 30 min of low-temperature thermal treatment of digested sludge. To study the effect of

temperature and thermal history on composition, the soluble COD has been measured at the different experimental temperatures of 50, 60, 70 and 80°C and heating duration of 1, 15 and 30min.

The sludge liquor was obtained by vacuum filtration using mixed cellulose membranes (with pore size of 0.45µm). The soluble COD was then determined by HACH procedure (Method 8000) using DR 4000 Spectrophotometer and DRB200 Single Block Reactor.

The percentage of releasing soluble COD was calculated using following equation:

$$rsCOD(\%) = 100 * \frac{(sCOD_{T1} - sCOD_{T0})}{sCOD_{T0}} \quad (2)$$

where rsCOD is the percentages of releasing soluble COD and sCOD_{T1}, sCOD_{T0} stand for soluble COD (mg/L) at temperature of T₁ and T₀ (20°C) respectively.

The advantage of COD analysis is it can measure the energetics of the system for the purpose of tracking biological reactions and it is relatively fast and reproducible. The COD measure the majority of sludge biodegradable and non-biodegradable organic matter including proteins, carbohydrates and fats. In addition, COD analysis is much more practical compare to specific analysis of sludge constituents (Ellis 2004).

3. Result and discussions

3.1. Effect of temperature and thermal history on digested sludge composition

Fig.1 shows that the soluble COD increases by increasing the temperature and time of heat treatment. Increase in the level of solubility induces that some particulate organic matter (COD content) inside the sludge, changed their phase and solubilized into liquid medium which was as a result of destructing flocs or cell layers (Appels et al. 2010).

Furthermore, after cooling down the previously 30min-long heated sludge, the soluble COD still remained at high level although a slight decrease from its highest value was observed, indicating the process looks partially irreversible. Also, this irreversible effect of temperature and duration of heat

treatment is more significant at higher temperatures since the soluble COD at 20°C with thermal history at 80°C is almost 4 times higher than soluble COD at 20°C without thermal history.

3.2. Effect of temperature and thermal history on the rheological behaviour of digested sludge

Fig.2a shows that sludge becomes less viscous as the temperature increases which is consistent with the literature (Sozanski et al. 1997, Mu et al. 2007, Baudez et al. 2013, Battistoni et al. 1993b).

Fig.2b highlights the effect of thermal history on sludge flow curve at 20°C. When the sludge heated to 70°C and 80°C and kept at that temperature for 30min, then cooled down to 20°C, its rheological behaviour is more viscous at high shear rate compared to the initial sludge (before heating). These results are in agreement with Baudez et al. (2013).

However, as it can be observed in Fig.3, increasing the time of heat treatment at different shear rates causes different impacts on sludge flow curve. By keeping the temperature (at 50°C to 80°C) for 15 and 30min the sludge becomes less viscous in the low shear rate range and more viscous in high shear rate range (see Fig.3 a and b). The effect of duration of heat treatment at low shear rate is much more evident at 50°C as well at high shear rate at 80°C since keeping the temperature for 30min at 50°C and 80°C cause almost 13% decrease and 17% increase in sludge viscosity, respectively (data are not shown for viscosity). The longer the heating time, the more agglomerated particles can be destructed and the size and fraction of particles be reduced and solid matter dissolves into liquid (Paul et al. 2006). At low shear rates or pasty region in which the soft interaction predominate, the potential energy of mutual interactions depends on the spatial configuration of particles. The particles separating distances or configuration, thus is different by decreasing fraction and size and dissolving solid into liquid medium. As a result of that, by increasing the duration of heat treatment, the potential of mutual interaction decreases and thus, the viscosity decreases at low shear rates. By contrast, in high shear rates or hydrodynamic region, in which the viscous dissipation increases significantly with the shear rate, the suspension viscosity is not only a function of particle fraction,

but it is most likely a stronger function of interstitial fluid (Cousot 2005). In the following, we will present data which shows that increasing the duration of heat treatment, increased the viscosity of liquid medium (interstitial liquid), as a result of that the viscosity of sludge (the mixture of particles and liquid medium) increased by increasing the duration of heat treatment at high shear rates. Fig.4a compares the soluble COD and the infinite viscosity (viscosity at highest shear rate; almost 1000s^{-1}). As it can be observed, the increase of soluble COD with temperature of thermal history was linearly proportional to the increase of infinite viscosity. Fig.4b also shows the decrease of soluble COD with temperature of thermal history was linearly proportional to the decrease of yield stress. It means that, as assumed by Baudez et al. (2013), when digested sludge was thermally treated for 30min at different temperatures (50, 60, 70 and 80°C) and then cooled back to 20°C , there was an irreversible transfer of organic chemicals from particulate state to solubilized one which resulted in increasing viscosity in high shear rates and decreasing the yield stress.

3.3. Effect of thermal history on the viscosity of liquid medium

3.4. The viscosity of sludge liquid medium was measured to confirm the effect of organic matter solubilisation (COD) on the rheological properties of sludge. Fig.5 compares the sludge liquid medium viscosity at 20°C for the liquors which were obtained at different heating temperatures (50- 80°C) using vacuum filtration. As it can be seen, the viscosity of liquid medium increased by increasing the temperature at which the liquor was collected. It follows the same trend of COD solubilisation data (shown in Fig.1). As reported in Fig.1, the solubilized COD after thermal treatment remained higher than solubilized COD without thermal treatment which was confirmed in Fig. 5: the liquor medium viscosity at 80°C was 6% higher than sludge liquor viscosity without thermal treatment. This increase could result in thermal denaturation of proteins (Anson 1954) which is effective on viscosity (Anson and Mirsky 1932). Indeed, most of proteins irreversibly denature by exposing to high temperatures. This in fact results in unfolding and increasing the amount of water bound by the protein molecules. The unfolding increases the hydrodynamic radius of the molecule

which increases the viscosity of the solution. Furthermore, when the proteins cooled back, kinetic barriers will prevent proteins to form as the native format. The increased water binding reduces the solubility (following by hydrophobic aggregations) which decreases the particle-particle interactions (Anson and Mirsky 1932). This decrease in particle-particle interactions can significantly reduce the yield stress of a suspension (Coussot et al. 2002). In the following, we will show that yield stress of a 30min-long heated sludge after it cooled back to 20°C was much lower than the sludge at 20°C without thermal treatment.

3.5. Effect of temperature and thermal history on solid behaviour of digested sludge

Fig.6a shows the yield stress of sludge at 50°C and 60°C and for different time of heating. Keeping the temperature (at 50°C and 60°C) for 15min and 30min caused a little bit decrease in yield stress. This effect is similar to the liquid behaviour observation presented in Fig.3. This was because, at higher temperature, destructing of flocs was faster, which resulted in large quantity of flocs to be destructed even after one minute and further heating time did not impact further.

As it is depicted in Fig.1, increasing the time of heat treatment at studied temperatures, the flocs destructed gradually which resulted in increase of soluble COD. Comparing Fig.1 and Fig.6a, this trend confirms relationship between the rate of increasing soluble COD (or in other word decrease of particulate COD) and the rate of decreasing yield stress.

Fig.6b compares the yield point of sludge with different thermal history. As it can be seen, the yield stress decreased by increasing the temperature of thermal history. The sludge which was heated for 30min at higher temperature and then cooled down to 20°C has lower yield stress, since the sludge flocs were destructed more at higher temperature. As result of destructing the sludge flocs, the mutual interaction between particles decreased which resulted in decreasing yield stress. Fig.6b also shows thermal treatment of sludge at 70°C and 80°C for 30min reduced the yield stress of sludge at 20°C up to 55% and 68%, respectively. It can significantly reduce the cost of pumping and mixing as well as

increasing biogas production (Climent et al. 2007), however, further studies are required since heating is costly and may increase the cost of system maintenance.

4. Conclusion

The apparent viscosity and the yield stress of sludge decreased by increasing the temperature and increasing the duration of heating. The effect of duration of heating was much more evident on apparent viscosity and yield stress of sludge in lower temperatures and at lower shear rates since 30min-long heat treatment at 50°C decreased the apparent viscosity at the shear rate of 20s⁻¹ by almost 13% compared to 1% decrease in apparent viscosity of 30min-long heated sludge at 80°C. The yield stress and the infinite viscosity of cooled 30min-long heated sludge at 80°C were 68% lower and 18% higher than the yield stress and the infinite viscosity of sludge without thermal history, respectively. Similarly to the yield stress data, COD value of cooled heated sludge did not return to original value before thermal treatment. Moreover we found a linear relationship between increasing soluble COD and increasing infinite viscosity as well as decreasing yield stress, indicating that thermal treatment induced solubilisation of organic compounds which affected sludge rheology.

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Table title

Table 1 Total solid and mineral solid content of digested sludge

Figure captions

Figure 1 Impact of temperature and duration of heat treatment on releasing COD

Figure 2 Viscosity and flow curve of 2% digested sludge; (a) Impact of temperature (20-80°C) (b)

Impact of thermal history in comparison to sludge without thermal history measured at 20°C

Figure 3 Impact of duration of heat treatment on flow curve of 2% digested sludge at different

temperatures (50°C to 80°C); (a) at a typical low shear rate (b) at a typical high shear rate

Figure 4 Evolution of soluble COD versus (a) infinite viscosity, and (b) yield stress for 2% digested

sludge with thermal history at different temperatures (50-80°C)

Figure 5 Impact of thermal history at different temperature on viscosity of sludge liquid medium

Figure 6 Measured yield stress of digested sludge (2%) (a) at different temperatures and different

duration of heat treatment (50°C and 60°C) (b) at different thermal histories with different

temperatures (50, 60 70 and 80°C) compared to sludge without thermal history measured at 20°C.