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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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25 **1. Introduction:**

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

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

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

$$43 \quad \tau = \tau^H + \dot{\gamma}^n \quad (1)$$

44 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
45 and n are flow consistency and flow behaviour index, respectively.

46 A temperature-dependence of sludge rheological characteristics has been reported by several authors
47 (Sozanski et al. 1997, Battistoni et al. 1993a, Mu et al. 2007, Abu-Jdayil et al. 2010, Baudez et al.
48 2013, Baroutian et al. 2013). An Arrhenius-like model is widely used to describe the relationship
49 between temperature and rheological parameters (Baudez et al. 2011, Battistoni et al. 1993a, Mu et

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

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

66 **2. Materials and Methods**

67 Digested sludge was collected at the Mount Martha waste water treatment plant (Melbourne,
68 Victoria, Australia) and carried in sealed containers. To reduce the impact of microbial activities
69 inside the sludge, the samples were stored at 4°C for 30 days before experiments. This procedure
70 helps with stability of samples which results in reproducible data for several days (Curvers et al.
71 2009).

72 To measure the dry solids content (wt.% DS) of the sludge, the sample was heated at 105°C for at
73 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

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

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

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

$$105 \quad rsCOD(\%) = 100 * \frac{(sCOD_{T_1} - sCOD_{T_0})}{sCOD_{T_0}} \quad (2)$$

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

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

113 **3. Result and discussions**

114 **3.1. Effect of temperature and thermal history on digested sludge composition**

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

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

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

124 **3.2. Effect of temperature and thermal history on the rheological behaviour of digested** 125 **sludge**

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

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

132 However, as it can be observed in Fig.3, increasing the time of heat treatment at different shear rates
133 causes different impacts on sludge flow curve. By keeping the temperature (at 50°C to 80°C) for 15
134 and 30min the sludge becomes less viscous in the low shear rate range and more viscous in high
135 shear rate range (see Fig.3 a and b). The effect of duration of heat treatment at low shear rate is much
136 more evident at 50°C as well at high shear rate at 80°C since keeping the temperature for 30min at
137 50°C and 80°C cause almost 13% decrease and 17% increase in sludge viscosity, respectively (data
138 are not shown for viscosity). The longer the heating time, the more agglomerated particles can be
139 destructed and the size and fraction of particles be reduced and solid matter dissolves into liquid
140 (Paul et al. 2006). At low shear rates or pasty region in which the soft interaction predominate, the
141 potential energy of mutual interactions depends on the spatial configuration of particles. The particles
142 separating distances or configuration, thus is different by decreasing fraction and size and dissolving
143 solid into liquid medium. As a result of that, by increasing the duration of heat treatment, the
144 potential of mutual interaction decreases and thus, the viscosity decreases at low shear rates. By
145 contrast, in high shear rates or hydrodynamic region, in which the viscous dissipation increases
146 significantly with the shear rate, the suspension viscosity is not only a function of particle fraction,

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

159 **3.3. Effect of thermal history on the viscosity of liquid medium**

160 **3.4.** The viscosity of sludge liquid medium was measured to confirm the effect of organic matter
161 solubilisation (COD) on the rheological properties of sludge. Fig.5 compares the sludge liquid
162 medium viscosity at 20°C for the liquors which were obtained at different heating temperatures (50-
163 80°C) using vacuum filtration. As it can be seen, the viscosity of liquid medium increased by
164 increasing the temperature at which the liquor was collected. It follows the same trend of COD
165 solubilisation data (shown in Fig.1). As reported in Fig.1, the solubilized COD after thermal
166 treatment remained higher than solubilized COD without thermal treatment which was confirmed in
167 Fig. 5: the liquor medium viscosity at 80°C was 6% higher than sludge liquor viscosity without
168 thermal treatment. This increase could result in thermal denaturation of proteins (Anson 1954) which
169 is effective on viscosity (Anson and Mirsky 1932). Indeed, most of proteins irreversibly denature by
170 exposing to high temperatures. This in fact results in unfolding and increasing the amount of water
171 bound by the protein molecules. The unfolding increases the hydrodynamic radius of the molecule

172 which increases the viscosity of the solution. Furthermore, when the proteins cooled back, kinetic
173 barriers will prevent proteins to form as the native format. The increased water binding reduces the
174 solubility (following by hydrophobic aggregations) which decreases the particle-particle interactions
175 (Anson and Mirsky 1932). This decrease in particle-particle interactions can significantly reduce the
176 yield stress of a suspension (Coussot et al. 2002). In the following, we will show that yield stress of a
177 30min-long heated sludge after it cooled back to 20°C was much lower than the sludge at 20°C
178 without thermal treatment.

179 **3.5. Effect of temperature and thermal history on solid behaviour of digested sludge**

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

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

189 Fig.6b compares the yield point of sludge with different thermal history. As it can be seen, the yield
190 stress decreased by increasing the temperature of thermal history. The sludge which was heated for
191 30min at higher temperature and then cooled down to 20°C has lower yield stress, since the sludge
192 flocs were destructed more at higher temperature. As result of destructing the sludge flocs, the mutual
193 interaction between particles decreased which resulted in decreasing yield stress. Fig.6b also shows
194 thermal treatment of sludge at 70°C and 80°C for 30min reduced the yield stress of sludge at 20°C up
195 to 55% and 68%, respectively. It can significantly reduce the cost of pumping and mixing as well as

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

198 **4. Conclusion**

199 The apparent viscosity and the yield stress of sludge decreased by increasing the temperature and
200 increasing the duration of heating. The effect of duration of heating was much more evident on
201 apparent viscosity and yield stress of sludge in lower temperatures and at lower shear rates since
202 30min-long heat treatment at 50°C decreased the apparent viscosity at the shear rate of 20s⁻¹ by
203 almost 13% compared to 1% decrease in apparent viscosity of 30min-long heated sludge at 80°C.
204 The yield stress and the infinite viscosity of cooled 30min-long heated sludge at 80°C were 68%
205 lower and 18% higher than the yield stress and the infinite viscosity of sludge without thermal
206 history, respectively. Similarly to the yield stress data, COD value of cooled heated sludge did not
207 return to original value before thermal treatment. Moreover we found a linear relationship between
208 increasing soluble COD and increasing infinite viscosity as well as decreasing yield stress, indicating
209 that thermal treatment induced solubilisation of organic compounds which affected sludge rheology.

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- 273

274 **Table title**

275 Table 1 Total solid and mineral solid content of digested sludge

276

277 **Figure captions**

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

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

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

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

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

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

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

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

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

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

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