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Accepted Manuscript

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PII: S0043-1354(13)00518-6
DOI: 10.1016/j.watres.2013.06.027
Reference: WR 10027

To appear in: Water Research

Received Date: 10 February 2013
Revised Date: 5 June 2013
Accepted Date: 15 June 2013

Please cite this article as: Eshtiaghi, N., Markis, F., Baudez, J.-C., Slatter, P., Proxy model materials to simulate the elastic properties of digested municipal sludge, Water Research (2013), doi: 10.1016/j.watres.2013.06.027.

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Graphical abstract:

Similarities and differences between digested municipal sludge and the mixture of carbopol gel and glass bead suspension
Proxy model materials to simulate the elastic properties of digested municipal sludge

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The elastic rheological properties of sludge are complex and evolve with time as a result of aging and microbial activity. Due to the peculiar nature of sludge, this makes the measurement of physical parameters difficult. The challenge is to identify a reference material that can be used as a proxy for industrial process design or optimization.

In this study, respectively the mixtures of 0.5%, 0.7% and 1% glass beads suspension in water have been added to 0.5%, 0.7% and 1% carbopol dissolved in water and neutralized with NaOH to prepare gel, at different ratios. Elastic and loss moduli have been determined for different glass bead suspension ratios in the range of 0% to 80%.

The results showed that there is a critical glass bead suspension / carbopol ratio at which the elastic properties of the mixture changes dramatically. The elastic properties of these model mixtures of different glass bead/carbopol ratio suspensions are compared with the elastic property of municipal sludge sampled from a Melbourne Waste Water Treatment Plant, and similarity established.

Keywords: Elastic moduli, Loss moduli, Digested municipal sludge, carbopol gel, glass bead
1. Introduction

Digested sludge is sewage sludge after anaerobic digestion. It is mainly made of colloids and non-digested organic matter. Digested sludge contains 95% water and the remaining part is made of organic matter and bacteria which tend to aggregate forming flocs. Sludge can be visualised as interacting particles in a suspending medium which bacteria form extra polymeric substances (EPS), finally presenting a three-dimensional gel-like matrix (Wingender et al., 1999, Baudez et al. 2013a). Viscoelastic property of sludge is an important in particular in anaerobic digesters. If mixing systems fail for any reason in anaerobic digesters, the stored potential energy in material due to elastic property of sludge can return material to non-deformed state which is unmixed state. This situation causes the formation of a solids bed at the bottom of the digester which often hardly fluidised again when mixing systems restored (Eshtiaghi, Markis et al., accepted 2013).

Although it has been known for many years that wastewater sludge exhibits non-Newtonian behaviour, this behaviour has not been a major concern in the engineering of sludge digestion facilities until recently because most facilities were designed and operated at relatively low solids concentrations (4 percent feed and 2 percent in the digester). Growing population and financial constraints compels wastewater treatment plants to increase throughput of their digesters (Eshtiaghi et. al., 2012a). The trend towards digesting thicker sludge requires pumping and mixing even more difficult fluids. This problem is amplified by the limitations of existing methods for predicting sludge rheology.

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. 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 (Eshtiaghi et al. 2012b). 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 (Spinosa 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 the 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. Baudez et al. (2007) 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.

The idea of a model fluid for sludge is to find a material that is less complicated but at the same time still be able to simulate the sludge behaviour. There is no study to find a representative, safe and non-odourous fluid which would mimic the rheological behaviour of sludge. Baudez et al. (2013a) recently examined the similarity between soft glassy material and sludge, based on its dynamic visco-elastic behaviour, i.e. the storage modulus ($G'$) and loss modulus ($G''$), in the temperature range of 10-60°C using strain and stress sweep tests. Therefore, carbopol gel, a soft glassy material, was chosen as a medium. To study the impact of suspended solids on the elastic properties of sludge, non-interactive particles such as glass beads have been chosen in the first stage of this study. This work will be a preliminary attempt at developing a model fluid, which provides the basis for future work in sludge.
characterisation utilizing the known characteristic of soft glassy materials. This paper aims to identify model fluids which will mimic the elastic behaviour of digested municipal sludge.

2. Material and Method

2.1) Sample preparation

Carbopol gel, which is a soft glassy material with no thixotropic behaviour, was chosen for this study. Carbopol gel at three different concentrations (0.5, 0.7, and 1 Wt. %) was prepared by dissolving the required amount of Carbopol ®940 (ACROS ORGANICS) for each concentration 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 whilst the pH was simultaneously measured until a PH of 4.8-5 was reached. This pH was chosen because deionised water has this pH value. Different ratio (0% to 80%) of different carbopol gel concentration (0.5, 0.7, and 1Wt. %) was added to different concentrations (0.5, 0.7, and 1Wt. %) of 115.95 µm glass bead suspension with similar concentrations. Glass beads are the non-interactive solids used in this experiment and were supplied from Potters Industries Pty Ltd. with a density of 2640 kg/m³ and a mean particle size of 115.95µm, measured with a Mastersizer X (from Malvern Instruments). Different concentrations of glass bead suspension (0.5, 0.7, and 1 Wt. %) were prepared and then carbopol gel added, as explained previously.

Digested sludge was obtained from the Mount Martha wastewater treatment plant (Melbourne, Victoria, Australia). The initial concentration of the digested sludge was found to be 18.5 g L⁻¹. This digested sludge was further concentrated using a Buchner funnel under vacuum.
2.2) **Rheological measurements:**

The elastic modulus $G'$ is determined using oscillatory shear experiments in a controlled strain rheometer (ARES) using 25 mm parallel plates with 1 mm gap size at 25°C. Strain sweep test at 25°C were done at different constant frequencies of 0.1 rad/s to 50 rad/s for different concentrations of pure carbopol gel. The result for 0.1 rad/s was scattered indicating that the transducer detectable range limit had been reached. Since there was no difference between other frequencies results; therefore, 10 rad/s was chosen for the rest of measurement. Furthermore, at low strain amplitudes (i.e. 0.1%), all $G''$ measurements show a measure of scatter. A very low torque level might reach the transducer’s detection limit hence there were scattered measurements at the low strain range. This basically means that the data points at low strain are not reliable (i.e. <1%) for the $G''$ measurement.

Dynamic frequency sweep tests were carried out for different ratios of the mixture of 0.5, 0.7 and 1.0% glass bead suspension in 0.5, 0.7, and 1.0%, respectively, carbopol gel with the strain amplitude was changing from 0.1% to 10%. With low strain amplitude, the data points are very scattered at low frequency amplitude. Therefore higher strain amplitude will be used for future investigations.

The procedure for strain and frequency sweep measurements of digested sludge is documented in previous publications (Baudez et al. 2013a, Baudez et al., 2011).

3. **Result and Discussion**

Figure 1 shows the storage modulus ($G'$) and loss modulus ($G''$) as a function of strain for the different mixtures of 0.5% glass bead suspension and 0.5% carbopol gel. In this figure and the following figures, the mixture of, for example, 80% carbopol gel and 20% glass bead suspension will be shown named as “8c2s”.
Figure 1: Strain dependence of storage modulus (G', Pa, solid symbol) and loss modulus (G'', Pa, hollow symbol) of the different mixture of 0.5% glass bead suspension/0.5% carbopol gel at 25°C at 10 rad/s

Figure 1 shows that G' decreases with increasing amount of suspension in the sample for all the carbopol/glass bead mixtures. At low strain amplitudes, the mixtures behave as a solid as the elastic modulus is almost independent of strain. By increasing the ratio of glass bead suspension, the elastic modulus of the mixture decreases, which is an indication of breaking the carbopol gel network. Carbopol gel structure consists of the highly cross-linked dense core part and dangling free end which interacts strongly with the other free ends of the surrounding dense core (Oppong et al., 2006). Baek and Kim (2011) studied carbopol gel network loaded with particles through Cryo-SEM and depicted that the repulsive interactions between free-ends is affected by the particles which act as obstacles. Since they loaded particles directly to the gel network, at the start they observed an increase in elastic modulus. Then, when particle loading is over the maximum point, the storage modulus decreases steeply. In our study, we observed a continuous decrease in the storage modulus by increasing suspension loading, which indicates that there is another phenomenon in place which could be forming new hydrogen bonds. Since carbopol gel structure is sensitive to ion presence and hydrogen bonding, electrostatic interaction could be responsible for this gradual decrease in elastic modulus (Fernandez et al., 2011). This may also simply relate to the dilution effect that added water causes. As carbopol concentration decreases, the mechanical strength of the carbopol/suspension decreases as well. This may explain why we observe a decrease in the mechanical strength while others observe an increase. Clearly, the dramatic change in gel structure that we observed is not solely a dilution effect, but a particle
contribution as well. The quantitative contribution from each part requires further investigation.

Baek and Kim (2011) observed that by loading particles, particles attach to the wall of irregular fibrous network structure of gel which at low particle loading the fibrous network structure is sustained due to existence of enough space for deformation. But above a critical concentration, gel characteristic changes due to change in the network structure and the loss of connecting network (Daniel-da-Silva et al. 2008) which then result in sudden decrease in $G'$ which in our case occurs when the suspension ratio increases to 80%. This is related to formation of a particle-polymer network which reduces the polymer-polymer network (Baek and Kim, 2011). Although Daniel-da-Silva et al. (2008) and Baek and Kim (2011) indicated that this phenomenon is exclusive to nano-particles, our observation shows that this phenomenon also occurs for larger particle sizes, since our glass bead particles have a size of around 100 µm.

Although there is a sudden change in microstructure after adding the 80% glass bead suspension, the change in the elastic modulus with deformation follows the same pattern as pure carbopol gel. This indicates that even with an 80% glass bead suspension, the gel network still exists in the mixture.

The difference between this study and other studies that have been done on adding particles to a gel structure (Kim and Baek, 2011; Mahaut et al., 2008; Grand and Petekidis, 2008) is that they have added particles directly to gel structure. Here, we have added a suspension of particles to the gel structure. For this reason, we have observed the opposite result in comparison to their results. In all the abovementioned studies, by increasing particle loading, yield stress, storage modulus and loss modulus all increase.
As it can be seen in Figure 1, at low strain, the carbopol network is only slightly distorted and stress remains proportional to the applied strain. As the strain increases toward the cross-over strain \((G''=G')\), the particles start bumping into each other, producing a temporary “shear thickening” which brings a distortion of the stress and \(G''\) reaches a peak at the cross-over strain then exceeds \(G'\) \((G''>G')\). At this point, the carbopol network is being broken. At higher strains, strain softening-induced distortion becomes dominant as the sample behaves like liquid (Grand and Petekidis, 2008).

At high strain amplitude, the viscous modulus also decreases by increasing the ratio of glass bead suspension in the mixture suggesting that the carbopol becomes easily deformed due to network structure rupture.

The arrow in Figure 1 shows the cross-over point where \(G''\) surpasses \(G'\) decreases by increasing the ratio of glass bead suspension in the mixture. This means that the carbopol network structure is breaking down more readily at higher Wt.% of suspension. There is no error bar presented, as the results are reproducible.

As it can be seen in Figure 1, at low strain amplitude the material behaves as a solid because \(G'\) is much greater than \(G''\). In this region, both \(G'\) and \(G''\) for these mixtures are almost independent of strain. The sample starts yielding as the strain increases to higher levels, \(G''\) becomes larger than \(G'\) and a peak forms where there is a “cross-over” of \(G'\) and \(G''\). The point of cross-over is the “cross-over strain” and the material behaves as a liquid after this cross-over. This is because \(G''\) (viscous modulus) is greater than \(G'\) (storage modulus) in the region past the cross-over strain. After that, both moduli decrease with increasing strain after the cross-over point. This behaviour is a characteristic trend of soft glassy materials.
3.1) Similarities and differences between digested municipal sludge and the mixture of carbopol gel and glass bead suspension

Baudez et al. (2013a) investigated the soft glassy behaviour of digested sludge. Contrary to most colloidal suspensions for which both $G'$ and $G''$ decrease monotonically as the strain increases (Macosko, 1994), digested sludge presents a different behaviour. At low strain amplitudes, $G'$ is nearly constant, suggesting a linear viscoelastic regime (LVE). Then, both moduli become strain dependent with $G'$ decreasing and $G''$ passing through a peak before decreasing as well (Figure 2). More interesting is the strain-dependence of $G'$ and $G''$: when $G''>G'$, they both follow a power-law model $G'' \propto \gamma^{-n}, G' \propto \gamma^{-2n}$ (Figure 2). In comparison, the elastic modulus of different mixtures of 0.5 % glass bead suspension/0.5% carbopol gel follow the same power law relationship of digested sludge ($G'' \propto \gamma^{-n}$, Figure 1) but the loss modulus of the mixture follows a power relationship with a different coefficient. The loss modulus in the mixture is less dependent on strain ($G' \propto \gamma^{-\alpha}$) as compared with sludge. Figure 3 shows the same characteristic soft glassy behaviour of digested sludge that exists for higher concentrations of sludge.

Figure 2: Strain-dependence of $G'$ and $G''$, at 25°C and 1Hz, for the sludge concentrated at 3.2% (Baudez et al., 2013a)
Figure 3: Strain dependence of the storage modulus ($G'$, Pa, solid symbol) and loss modulus ($G''$, Pa, hollow symbol) of the different concentrations of digested sludge at 25°C and 0.2Hz.

As can be seen in Figure 3, when the concentration of the sludge increases the $G'$ - $G''$ crossover point moves toward lower strain. This is similar to the trend of increasing the ratio of the glass bead suspension in carbopol (Figure 1) which decreases the cross-over point strains. This implies that by increasing sludge concentration or suspension ratios, the mixture forms a weaker gel as the cross-over strain decreases. Therefore, it can be deduced that increasing suspension ratios has the same effect as increasing the total suspended solids in a sludge.

Figures 4 and 5 portray the elastic modulus and loss modulus of 3.23% digested sludge compared to different concentrations and ratios of glass bead suspension/carbopol gel. It can be seen from these figures that the critical ratio for adding glass bead suspensions is 80%, regardless of the original carbopol concentration as a dramatic change - or in other words, the structural change occurs at this ratio.

Figure 4: Strain dependence of storage modulus ($G'$, Pa, solid symbol) and loss modulus ($G''$, Pa, hollow symbol) of the different mixtures of 0.7% glass bead suspension/0.7% carbopol gel at 10 rad/s and 3.23% digested sludge at 25°C and 1Hz.

Figure 5: Strain dependence of storage modulus ($G'$, Pa, solid symbol) and loss modulus ($G''$, Pa, hollow symbol) of the different mixture of 1% glass bead suspension/1% carbopol gel at 10 rad/s and 3.23% digested sludge at 25°C and 1Hz.

If cross-over strains ($G'=G''$) for different concentrations of suspension/carbopol are extracted and plotted against increasing suspension ratios (Figure 6), all the mixtures follow
an exponential relationship. This trend is similar to the exponential change of cross-over
strain point for different concentrations of sludge (Figure 7).

Figure 6: The crossover strain (G’=G’’) as a function of glass bead suspension ratio for
0.5 % glass bead suspension/0.5% carbopol gel

Figure 7: The crossover strain (G’=G’’) as a function of concentration for digested
sludge

Similar to dependence of crossover strain to sludge concentration of suspension ratio, critical
strain is also concentration dependent. Critical strain (change from linear viscoelastic region to
non linear viscoelastic) of digested sludge decreases as sludge concentration increases
(Table 1). This means that by increasing sludge concentration, sludge will be a weaker gel
and its structure breaking easier. This is similar to increasing the concentration of glass bead
suspension which by increasing the suspension ratio, the mixture forms a weaker gel (Table
1). This may also highlight the fact that the suspended solids concentration plays a strong role
in the structure of the suspension.

Although both sludge and the mixture of suspension and carbopol gel gets weaker by
increasing the concentration of sludge or the ratio of suspension, the elasticity of sludge
increases with increasing solids concentration (Figure 3) but the elasticity of the mixture
decreases with increasing suspension ratio (Figure 1). This is a difference between sludge and
the prepared mixture - their elasticity does not follow the same relationship
Table 1: Critical strain dependence to concentration/ratio for digested sludge and the mixture of 0.5 %glass bead suspension/0.5% carbopol gel

<table>
<thead>
<tr>
<th>Glass bead suspension ratio</th>
<th>Sludge concentration</th>
<th>Critical strain (%)</th>
<th>Sludge Critical strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.85</td>
<td>143.9</td>
<td>8.7</td>
</tr>
<tr>
<td>20</td>
<td>3.17</td>
<td>131.4</td>
<td>5.6</td>
</tr>
<tr>
<td>30</td>
<td>4.89</td>
<td>119.7</td>
<td>5.3</td>
</tr>
<tr>
<td>50</td>
<td>4.89</td>
<td>99.6</td>
<td></td>
</tr>
<tr>
<td>60</td>
<td>90.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>70</td>
<td>68.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>80</td>
<td>68.9</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

It is shown in Figure 8 that $G'$ is always higher than $G''$ suggesting that the 0.5% carbopol-suspension mixture has more solid than viscous properties. The elastic modulus dominates across all frequencies, which reflects the dominant elastic property of the gel below the yield stress level (Tamburic and Craig 1995; A-sasutjarit et al., 2005, Kim and Baek, 2011). While $G'$ is monotonically increasing with frequency, $G''$ has a minima. This behaviour was observed for carbopol containing nano-particles (Kim and Baek, 2011) and digested sludge (Baudez et. al.2013a, Fig.9). By increasing the suspension ratio, $G'$ and $G''$ also decrease until a critical suspension ratio of 80% is reached, where $G'$ decreases dramatically by a factor of 3. This is also evident in the case of $G''$. This implies a change in the equilibrium gel structure as observed by other researchers as well (Kim and Baek, 2011). Even though there is a change in gel structure, $G''$ does not become larger than $G'$, meaning that it still remains as a gel.
Figure 8: Frequency dependence of 0.5 % glass bead suspension/0.5% carbopol gel at a constant strain of 10%

It seems there is no specific relationship between $G'$ and $G''$ and frequency. This behaviour is different from the behaviour of a 3.2% digested sludge, as there is a critical frequency (0.03 Hz) at which solid-like behaviour is gradually replaced by liquid-like behaviour. This demonstrates that the current mixture cannot mimic the low frequency or long time scale response of real sludge. Increasing liquid-like behaviour of sludge in long time scale processes could be related to a continuation of microbiological activities in the sludge which digests more solids and converts them to liquid (Baudez et al., 2013b). But this mixture could be a good representation of short time processes such as mixing as at higher frequency, the solid-like behaviour of the mixture dominates the liquid-like behaviour - similar to digested sludge and the response of both sludge and model fluid is entirely elastic.

Figure 9: Frequency-dependence of $G'$ and $G''$ at 25°C for 3.23% digested sludge (Baudez et al., 2013a).

4. Conclusion:

This paper is a preliminary result of an investigation of the viscoelastic behaviour of a mixture of three different concentrations of carbopol gel and glass bead suspensions at different ratios of suspension-to-gel with similar concentrations through oscillatory measurements. A ratio of addition between 0% and 80% of suspension was analysed. We found that adding 80% glass bead suspension across all carbopol concentration dramatically changes the gel network due to obstacles that particles cause in the dangling free end of the carbopol gel molecule. In comparison with digested municipal sludge, increasing glass bead suspension ratio has a similar effect to increasing the concentration of digested sludge - as in...
both cases the critical strain as well as the cross-over strain decreases exponentially with increasing either suspension ratio or sludge concentration. Also, the dependency of \( G' \), and \( G'' \) with strain after the cross-over point in both cases, follows a power-law relationship. It seems that particle loading can mimic the effect of increasing sludge concentration. The difference between increasing sludge concentration and increasing suspension ratio is the effect of these parameters on elasticity, as they show the opposite trend compared to each other. Another difference of prepared model fluid with sludge is its response to the low frequency or long time scale process is not the same as for digested sludge. But this mixture could be a good representation of short time process such as mixing as, at higher frequency, the solid-like behaviour of the mixture dominates the liquid like-behaviour - similar to digested sludge; and the response of both sludge and model fluid is entirely elastic. It is required to conduct creep and creep recovery test and other measurements before making any definite conclusion of suitability of prepared mixture as a representative of digested sludge.

**Acknowledgment**

The authors are very grateful for financial support of Melbourne Water and South East Water (Ltd.) as well as the Research and Innovation Office of RMIT University for an Emerging Researcher Industry grant.

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Figure 1. Strain dependence of storage modulus ($G'$, Pa, solid symbol) and loss modulus ($G''$, Pa, hollow symbol) of the different mixture of 0.5% glass bead suspension/0.5% carbopol gel at 25°C and 10 rad/s frequency.

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Figure 4: Strain dependence of storage modulus ($G'$, Pa, solid symbol) and loss modulus ($G''$, Pa, hollow symbol) of the different mixture of 0.7% glass bead suspension/0.7% carbopol gel at 10 rad/s and 3.23% digested sludge at 25°C and 1Hz.
Figure 5: Strain dependence of storage modulus (G’, Pa, solid symbol) and loss modulus (G”, Pa, hollow symbol) of the different mixture of 1% glass bead suspension/1% carbopol gel at 10 rad/s and 3.23% digested sludge at 25°C and 1HZ.

Figure 6: The cross-over strain (G’=G”) as a function of glass bead suspension ratio for 0.5% glass bead suspension/0.5% carbopol gel.
Figure 7: The cross-over strain ($G' = G''$) as a function of concentration for digested sludge.

\[ y = 61.425e^{-0.41x} \]
\[ R^2 = 0.9841 \]

Figure 8: Frequency dependence of 0.5% glass bead suspension/0.5% carbopol gel at a constant strain of 10%.
Figure 9: Frequency-dependence of $G'$ and $G''$ at 25°C for 3.23% digested sludge (Baudez et al., 2013a).
Research Highlights

- Developing a proxy model material for Elastic properties of digested sludge
- Similar effect between increasing glass bead suspension and sludge concentration
- Existence of critical glass bead ratio for dramatic change of carbopol gel network
- Glass bead/carbopol mixture is a good proxy only for short time process