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Influence of gas injection on viscous and viscoelastic properties of Xanthan gum

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Abstract

Xanthan gum is widely used as a model fluid for sludge to mimic the rheological behaviour under various conditions including impact of gas injection in sludge. However, there is no study to show the influence of gas injection on rheological properties of xanthan gum specifically at the concentrations at which it is used as a model fluid for sludge with solids concentration above 2%.

In this paper, the rheological properties of aqueous xanthan gum solutions at different concentrations were measured over a range of gas injection flow rates. The effect of gas injection on both the flow and viscoelastic behaviour of Xanthan gum (using two different methods - a creep test and a time sweep test) was evaluated. The viscosity curve of different solid concentrations of digested sludge and waste activated sludge

25 were compared with different solid concentrations of Xanthan
26 gum and the results showed that Xanthan gum can mimic the
27 flow behaviour of sludge in flow regime.

28 The results in linear viscoelastic regime showed that increasing
29 gas flow rate increases storage modulus (G'), indicating an
30 increase in the intermolecular associations within the material
31 structure leading to an increase in material strength and solid
32 behavior. Similarly, in creep test an increase in the gas flow
33 rate decreased strain%, signifying that the material has become
34 more resistant to flow. Both observed behaviour is opposite to
35 what occurs in sludge under similar conditions.

36
37 The results of both the creep test and the time sweep test
38 indicated that choosing Xanthan gum aqueous solution as a
39 transparent model fluid for sludge in viscoelastic regime under
40 similar conditions involving gas injection in a concentration
41 range studied is not feasible. However Xanthan gum can be
42 used as a model material for sludge in flow regime; because it
43 shows a similar behaviour to sludge.

44 Key Words:

45 Gas injection; Xanthan gum; Viscoelastic Properties; Flow
46 behaviour; Herschel-Bulkley Model

47 1. Introduction

48 Xanthan gum is a naturally occurring polysaccharide, produced
49 by fermenting glucose with the bacteria *Xanthomonas*
50 campestris, with a backbone of β -(1,4)-D-glucose (Kennedy et
51 al. 2015). The primary structure of the material is shown in
52 Figure 1. The structure consists of repetitive pentasaccharide
53 units formed with two glucose units, two mannose units, and
54 one glucuronic acid unit, with the molar ratio being 2.8:2.0:2.0
55 (García-Ochoa et al. 2000)

56 When mixed in aqueous solution, xanthan gum exists in an
57 ordered helical conformation, either single or double stranded.
58 The molecular structure of the material actively contributes to
59 its rheological properties, with the structuration pattern of the
60 solutions being related to hydrogen-bridging between lateral
61 chains and binding networks formed by molecular
62 entanglement (Laneuville et al. 2013). Additionally, Figure 2
63 shows that dehydration can affect the conformation of
64 molecules, in which the intra and intermolecular ester bonds
65 cause crosslinking with an extended polymer structure (Bueno
66 et al. 2013). Xanthan solutions are known to have a non-
67 Newtonian rheology, with a shear-thinning behaviour under
68 increasing shear rate. It has been widely reported that an initial
69 yield stress is exhibited by xanthan solutions must be overcome
70 for the solution to start flowing (García-Ochoa et al. 2000,
71 Marcotte et al. 2001, Song et al. 2006). Only when the

72 magnitude of stress reaches above the yield stress, the structure
73 is broken down, orienting the polymer chains to align with flow
74 stream. The yield stress is attributed to the molecular structure
75 of the material and a large number of hydrogen bonds which
76 exist in the solution (Bradshaw et al. 1983).

77 Xanthan gum is widely used as a model fluid for sludge to
78 mimic sludge shear thinning behaviour (García-Ochoa et al.
79 2000, Kennedy et al. 2015, Saha and Bhattacharya 2010).

80 Sludge is the residual, semi-solid slurry produced from waste
81 water treatment process. It is also well known that sludge is a
82 mixture of complex biological material and difficult to
83 characterize (Eshtiaghi et al. 2013, Ratkovich et al. 2013,
84 Seyssiecq. et al. 2003). Moreover, the opaque nature of sludge
85 makes it difficult to estimate the accurate bubble behaviour and
86 impacts on hydrodynamics of the process. Changes in complex
87 rheological behaviour of sludge over time because of aging and
88 microbial activity cause variations in sludge viscosity making it
89 difficult to optimize the process performance (Bajón Fernández
90 et al. 2015, Baudez and Coussot 2001). Since sludge
91 rheological properties have a significant impact on the design
92 parameters of the equipment used in the process, which
93 potentially can affect energy consumption, and the cost of
94 operation (Yang et al. 2009), Xanthan gum is used as a model
95 fluid for sludge under gas injection for optimising and
96 modelling of process equipment in sludge treatment plant

97 (Bhattacharjee et al. 2015, Cao et al. 2016). Gas injection in
98 sludge is recognized to play a significant role in oxygen
99 transfer, mixing efficiency and energy consumption in
100 membrane bioreactor and waste activated sludge process
101 (Åmand and Carlsson 2012, Bobade et al. 2017, Ratkovich et
102 al. 2013, Seyssiecq et al. 2008). The gas injection also has a
103 major impact on sludge physical properties like extra cellular
104 polymeric substances (EPS), soluble COD, particle size, etc.,
105 and changes its rheological properties influencing the
106 efficiency of the process (Drews 2010, Meng et al. 2006).

107 Although, the rheological properties of xanthan gum have been
108 extensively studied under varying conditions of pressure
109 (Laneuville et al. 2013), temperature (Marcotte et al. 2001),
110 gum concentration and ionic strength (Vega et al. 2015); there
111 is no study reporting the impact of gas injection on rheological
112 properties of xanthan gum. The objective of this work is to
113 investigate the effect of gas injection on the rheological
114 properties (apparent viscosity and viscoelastic modulus) of
115 xanthan gum at different solids concentrations and gas flow
116 rates by using dynamic time sweep test and creep test. As well
117 as to compare it with the sludge behaviour under similar
118 experimental condition (Bobade et al. 2017).

119

120 2. Materials and Methods

121 *2.1 Sample preparation*

122 Xanthan gum solutions of 0.3wt%, 0.4wt%, 0.5% and 0.6wt%
123 were prepared by mixing xanthan gum powder (supplied by
124 Sigma Aldrich) in deionized water to form a homogenous
125 solution. Solutions were mixed using a stirrer at approximately
126 700 rpm until the solution became homogenous. The solution
127 was allowed to rest for one day, to remove air bubbles from the
128 solution.

129 *2.2 Apparatus*

130 Rheological measurements were performed using a
131 commercially available hybrid stress controlled (HR3)
132 rheometer from TA Instruments equipped with Grooved bob
133 geometry with an outer diameter of 0.0149 m, and 0.042m
134 length. A custom designed plexi glass cup (inner diameter:
135 0.1m, length: 0.1m) was used. A stainless steel porous disk
136 (outer diameter: 0.1 mm, thickness: 0.0016 m, porosity: 40%,
137 from SINTEC Australia) was used at the bottom for the gas
138 sparging. The gas flow rate was varied from 0.5 litres per
139 minute (LPM) to 2 LPM i.e., 8.33×10^{-6} (m³/s) to 3.33×10^{-5}
140 (m³/s) using a gas mass flow meter from AALBORG at a
141 pressure of 10 Psi. All measurements were carried out at room
142 temperature.

143 *2.3 Rheological measurements*

144 To understand the impact of gas injection on apparent viscosity
 145 of Xanthan gum, a flow curve measurement was carried out
 146 using following procedure. The sample was pre sheared at high
 147 shear rate “310 s⁻¹” [the maximum shear rate without
 148 turbulence in this cup with grooved bob geometry] for 300 s
 149 and then allowed to rest for 120 s, to obtain an identical sample
 150 before each flow curve measurement. The viscosity of the
 151 sample was then measured at the shear rate from 0.001s⁻¹ to
 152 100s⁻¹. Further, the preshearing stage was repeated and the gas
 153 was injected with 0.5 LPM for 1200 s, and then the flow curve
 154 was measure. The above procedure is repeated for all the gas
 155 flow rates and all concentrations in duplicate.
 156 Since grooved bob geometry with a wide gap (0.042m) was
 157 used, the flow curves were recalculated using equations (1) and
 158 (2) (Estellé et al. 2008)

$$159 \quad \tau_{Ri} = \frac{M}{(2\pi H R_i^2)} \quad (1)$$

$$160 \quad \dot{\gamma} = 2M \frac{d\Omega}{dM}, \tau_c \leq \tau_y \leq \tau_b \quad (2)$$

161 , Where,

162 M = Torque (N.m),

163 H = Height of the bob (m),

164 R_i = Radius of the bob,

165 dΩ/dM = (Ω_j - Ω_{j-1}) / (M_j - M_{j-1}),

166 τ_y; τ_c ; τ_b = yield stress, stress at the cup and stress at
 167 the bob, respectively (Pa).

168 To understand the macro or micro structural changes occurring
169 in Xanthan gum due to gas dispersion, a time sweep
170 measurement was performed using following pattern: Preshear
171 the Xanthan gum at a high shear rate of 310s^{-1} and allow the
172 short rest period of 120s to remove the history of the sample
173 and obtain the identical sample for each test. Afterward,
174 oscillation time sweep test at 0.15% strain and 1Hz frequency
175 for 1500 seconds is carried out and preshearing step is repeated.
176 After repeating the preshearing step, the gas is injected for 20
177 mins and time sweep test is carried out again. This procedure is
178 repeated for all the four concentrations and 4 gas flow rates.
179 Similarly, to understand the impact of gas injection on elastic
180 deformation of xanthan gum, a creep test at very low stress of
181 3pa was carried out using the same procedure. This procedure
182 is similar to gas injection procedure into sludge which was
183 done by Bobade et al. (2017)

184 3. Result and discussion

185 *3.1 Impact of gas injection on flow behaviour*

186 *(apparent viscosity) of Xanthan gum*

187 The stress response over a range of shear rates was measured to
188 understand the flow behaviour of Xanthan gum. For different
189 solids concentration viscosity curves were plotted at different
190 gas flow rates. One sample graph is presented in Fig 3 (Veena
191 and Nicky 2017). The figure shows that at 0.3wt% xanthan

192 gum for different gas flow rates there is negligible change in
193 viscosity at given shear rate. Similar trend in viscosity curve
194 was observed for all the 4 concentrations at 4 different gas flow
195 rates (See supplementary Fig. S1). The uncertainty of change in
196 this observation is $\pm 5\%$ if the instrument is not calibrated
197 properly.

198 It is interesting to note that similar negligible change in
199 viscosity of sludge with gas injection was also observed by
200 Bobade et al. (2017). The reason for negligible change in
201 viscosity can be bubble coalescence occurring in Xanthan gum
202 because of its high viscosity (Bobade et al. 2017, Fransolet et
203 al. 2005). Thus the results clearly indicated that gas injection
204 has no impact on viscosity of xanthan gum.

205 In addition, to show which solids concentration of xanthan gum
206 can simulate 2 and 3.6wt % digested and 3wt% waste activated
207 sludge, the flow behaviour of xanthan gum was also compared
208 with the waste activate sludge (WAS) and digested sludge flow
209 behaviour as shown in Fig.4. The digested sludge data in
210 Figure 4 was extracted from Eshtiaghi et al. (2016). The Figure
211 clearly shows that 2wt % digested sludge is close to 0.3 wt%
212 and 0.4wt% Xanthan gum. However, there was a significant
213 difference between the viscosity curves for both 3wt% WAS
214 and 3.6wt% Digested sludge with even highest solid
215 concentration (0.6wt%) of xanthan gum. The Herschel Bulkley
216 parameters for 3wt% WAS and 3.5wt% digested sludge were

217 much greater than xanthan gum for all the concentrations as
218 shown in Fig 5.

219 The value of Herschel Bulkley parameters for each
220 concentration of Xanthan gum; WAS and digested sludge is
221 presented in Table 1. It is clear from Table 1 yield stress value
222 and flow consistency index of 3.6wt% digested sludge and
223 3wt% WAS is much higher than equivalent properties of the
224 highest concentration of xanthan gum. So much higher solid
225 concentrations of xanthan gum as a simulant is needed to be
226 used in order to mimic flow behaviour of 3.6wt% digested
227 sludge and 3wt% WAS in liquid regime.

228 *3.2 Influence of gas injection on viscoelastic*
229 *property of Xanthan gum in linear viscoelastic*
230 *region (Solid regime)*

231 To investigate the impact of gas injection on the viscoelastic
232 property of xanthan gum in the linear region, the influence of
233 gas was studied by both (1) creep test, and (2) time sweep test
234 as shown in Fig. 6. (Veena and Nicky 2017) Fig. 6A
235 demonstrates that, during creep test, the strain % for 0.3wt%
236 xanthan gum decreased by increasing the gas flow rate. The
237 decreasing values for strain % indicate more resistance to the
238 constant load and a lower degree of deformation of material.
239 The less strain % values means more solid behaviour and
240 difficult to deform as a result of being stronger material (Saha

241 and Bhattacharya 2010). Thus, xanthan gum solidifies and
242 becomes harder to deform as the gas injection rate increases.
243 Similarly, Fig. 6B reveals that, as the gas flow rate increased
244 both the viscous (loss) modulus (G'') and elastic (storage)
245 modulus (G') were increased while G' was larger than G'' at all
246 times. It means that that xanthan gum solidified in the linear
247 viscoelastic regime as the gas injection rate was increased
248 (Mezger 2011). Furthermore, the solid behavior of xanthan
249 gum has dominant impact compared to the liquid behaviour of
250 xanthan gum, indicating that material was still in solid regime.
251 It was also observed that an increase in G'' at different gas flow
252 rates is negligible in comparison to the impact of gas injection
253 on G' . A similar observation was observed for all the four
254 solid concentrations at four different gas flow rates in both
255 creep test and time sweep test (see supplementary Fig. S2 and
256 Fig. S3). The uncertainty of change in this observation is $\pm 5\%$
257 if the instrument is not calibrated properly.
258
259 The main possible reason behind xanthan gum solidifying
260 behaviour can be, change in molecular structure at a micro or
261 macro level due to the injection of nitrogen gas, which results
262 in a more entangled structure by altering crosslinking. In the
263 same way, the thickening behaviour has been observed in
264 similar polysaccharide materials such as mamaku gum
265 (Jaishankar et al. 2015), whereby the material exhibits

thickening behaviour when sheared below yield stress. Jaishankar et al. 2015 explained that thickening behaviour of polysaccharide at low shear rate occurs because of an interaction between intra and intermolecular associations. It means at low shear rates the molecule remains in equilibrium due to the disentanglement time being longer, because of which molecule gets elongated and results into thickening behaviour. As most of the characteristics of all types of polysaccharides are same (Lapasin and Prici 1995), when the gas is injected in the xanthan gum solution, the stress developed by gas injection is minimal and not sufficient to disentangle the molecular structure. Hence, the polymer chains became extensionally deformed and partially elongated, increasing exposure for local molecular interactions and physical crosslinking to form helices. The presence of helix structure and hydrogen bonds in xanthan gum show resistance to stress and flow is suggested by Bradshaw et al. (1983). Furthermore, as the gas velocity increases with increased flow rate, there is an increase in the shear stress between the bubble-liquid interface (Majumder et al. 2007). The increase in shear stress increases molecular elongation and thus the exposure for further molecular interactions, therefore contributing to an increased material strength, solution viscosity, and solid behaviour. Surprisingly, this behaviour of xanthan gum was not consistent with the sludge behaviour under similar conditions i.e., at low strain and

291 stress corresponding to linear region. Bobade et al. (2017)
292 have shown that increase in gas injection rate reduces the
293 viscoelastic properties of sludge using same experimental setup
294 and at strain and stress corresponding to linear region. This
295 reduction of viscoelastic properties of sludge is due to
296 breakdown of floc structure as a result of imposed shear by gas
297 injection. Moreover, the creep tests done under similar
298 condition showed that sludge deforms more by increasing gas
299 injection flow rate. This means weaker structure (see insets in
300 Fig 6). Bobade et al. (2017) proved weakening of sludge
301 structure through environmental scanning electron microscopic
302 analysis by observing more porous structure.

303 4. Conclusion

304 In this present study, we investigated how the rheological
305 properties of xanthan gum at different solids concentration
306 changes due to gas injection.
307 In flow region, the flow curve of xanthan gum showed
308 negligible change in the apparent viscosity of in xanthan gum
309 solution as gas injection flow rate increased. However, in linear
310 viscoelastic region, the creep test and time sweep test proved
311 that gas injection increased the storage and loss modulus which
312 is an indication of strengthening of molecular structure. This
313 could be due to deformation of the molecular structure of
314 xanthan gum and increasing the crosslinking within an

315 extended structure which will result in higher resistance to
316 stress and showing more solid like behaviour in the linear
317 viscoelastic region. Thus although xanthan gum behaves
318 similar to the sludge in the liquid regime, the behaviour of
319 xanthan gum contradicts with the sludge behaviour in the solid
320 regime which means Xanthan gum is not suitable as a model
321 fluid for sludge under gas injection below yield stress point.

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326 6. References

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