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

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

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

Key Words:

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

1. Introduction

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

When mixed in aqueous solution, xanthan gum exists in an ordered helical conformation, either single or double stranded. The molecular structure of the material actively contributes to its rheological properties, with the structuration pattern of the solutions being related to hydrogen-bridging between lateral chains and binding networks formed by molecular entanglement (Laneuville et al. 2013). Additionally, Figure 2 shows that dehydration can affect the conformation of molecules, in which the intra and intermolecular ester bonds cause crosslinking with an extended polymer structure (Bueno et al. 2013).Xanthan solutions are known to have a non-Newtonian rheology, with a shear-thinning behaviour under increasing shear rate. It has been widely reported that an initial yield stress is exhibited by xanthan solutions must be overcome for the solution to start flowing (García-Ochoa et al. 2000, Marcotte et al. 2001, Song et al. 2006). Only when the magnitude of stress reaches above the yield stress, the structure is broken down, orienting the polymer chains to align with flow stream. The yield stress is attributed to the molecular structure of the material and a large number of hydrogen bonds which exist in the solution (Bradshaw et al. 1983).

Xanthan gum is widely used as a model fluid for sludge to mimic sludge shear thinning behaviour (García-Ochoa et al. 2000, Kennedy et al. 2015, Saha and Bhattacharya 2010). Sludge is the residual, semi-solid slurry produced from waste water treatment process. It is also well known that sludge is a mixture of complex biological material and difficult to characterize (Eshtiaghi et al. 2013, Ratkovich et al. 2013, Seyssiecq. et al. 2003). Moreover, the opaque nature of sludge makes it difficult to estimate the accurate bubble behaviour and impacts on hydrodynamics of the process. Changes in complex rheological behaviour of sludge over time because of aging and microbial activity cause variations in sludge viscosity making it difficult to optimize the process performance (Bajón Fernández et al. 2015, Baudez and Coussot 2001). Since sludge rheological properties have a significant impact on the design parameters of the equipment used in the process, which potentially can affect energy consumption, and the cost of operation (Yang et al. 2009), Xanthan gum is used as a model fluid for sludge under gas injection for optimising and modelling of process equipment in sludge treatment plant

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

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

2. Materials and Methods

2.1Sample preparation

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

2.2Apparatus

Rheological measurements were performed using a commercially available hybrid stress controlled (HR3) rheometer from TA Instruments equipped with Grooved bob geometry with an outer diameter of 0.0149 m, and 0.042m length. A custom designed plexi glass cup (inner diameter: 0.1m, length: 0.1m) was used. A stainless steel porous disk (outer diameter: 0.1 mm, thickness: 0.0016 m, porosity: 40%, from SINTEC Australia) was used at the bottom for the gas sparging. The gas flow rate was varied from 0.5 litres per minute (LPM) to 2 LPM i.e., 8.33×10^{-6} (m^3/s) to 3.33×10^{-5} (m^3/s) using a gas mass flow meter from AALBORG at a pressure of 10 Psi. All measurements were carried out at room temperature.

2.3Rheological measurements

To understand the impact of gas injection on apparent viscosity of Xanthan gum, a flow curve measurement was carried out using following procedure. The sample was pre sheared at high shear rate “310 s^{-1}” [the maximum shear rate without turbulence in this cup with grooved bob geometry] for 300 s and then allowed to rest for 120 s, to obtain an identical sample before each flow curve measurement. The viscosity of the sample was then measured at the shear rate from $0.001s^{-1}$ to $100s^{-1}$. Further, the preshearing stage was repeated and the gas was injected with 0.5 LPM for 1200 s, and then the flow curve was measure. The above procedure is repeated for all the gas flow rates and all concentrations in duplicate.

Since grooved bob geometry with a wide gap (0.042m) was used, the flow curves were recalculated using equations (1) and (2) (Estellé et al. 2008)

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

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

, Where,

M = Torque (N.m),

H = Height of the bob (m),

R_i = Radius of the bob,

$d\Omega/dM = (\Omega_j - \Omega_{j-1}) / (M_j - M_{j-1})$,

$\tau_{y;}$ τ_c ; τ_b = yield stress, stress at the cup and stress at the bob, respectively (Pa).

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

3. Result and discussion

3.1 Impact of gas injection on flow behaviour (apparent viscosity) of Xanthan gum

The stress response over a range of shear rates was measured to understand the flow behaviour of Xanthan gum. For different solids concentration viscosity curves were plotted at different gas flow rates. One sample graph is presented in Fig 3 (Veena and Nicky 2017). The figure shows that at 0.3wt% xanthan gum for different gas flow rates there is negligible change in viscosity at given shear rate. Similar trend in viscosity curve was observed for all the 4 concentrations at 4 different gas flow rates (See supplementary Fig. S1). The uncertainty of change in this observation is ±5% if the instrument is not calibrated properly.

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

In addition, to show which solids concentration of xanthan gum can simulate 2 and 3.6wt % digested and 3wt% waste activated sludge, the flow behaviour of xanthan gum was also compared with the waste activate sludge (WAS) and digested sludge flow behaviour as shown in Fig.4. The digested sludge data in Figure 4 was extracted from Eshtiaghi et al. (2016). The Figure clearly shows that 2wt % digested sludge is close to 0.3 wt% and 0.4wt% Xanthan gum. However, there was a significant difference between the viscosity curves for both 3wt% WAS and 3.6wt% Digested sludge with even highest solid concentration (0.6wt%) of xanthan gum. The Herschel Bulkley parameters for 3wt% WAS and 3.5wt% digested sludge were much greater than xanthan gum for all the concentrations as shown in Fig 5.

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

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

To investigate the impact of gas injection on the viscoelastic property of xanthan gum in the linear region, the influence of gas was studied by both (1) creep test, and (2) time sweep test as shown in Fig. 6. (Veena and Nicky 2017) Fig. 6A demonstrates that, during creep test, the strain % for 0.3wt% xanthan gum decreased by increasing the gas flow rate. The decreasing values for strain % indicate more resistance to the constant load and a lower degree of deformation of material. The less strain % values means more solid behaviour and difficult to deform as a result of being stronger material (Saha and Bhattacharya 2010). Thus, xanthan gum solidifies and becomes harder to deform as the gas injection rate increases. Similarly, Fig. 6B reveals that, as the gas flow rate increased both the viscous (loss) modulus (G”) and elastic (storage) modulus (G’) were increased while G’ was larger than G” at all times. It means that that xanthan gum solidified in the linear viscoelastic regime as the gas injection rate was increased (Mezger 2011). Furthermore, the solid behavior of xanthan gum has dominant impact compared to the liquid behaviour of xanthan gum, indicating that material was still in solid regime. It was also observed that an increase in G” at different gas flow rates is negligible in comparison to the impact of gas injection on G’. A similar observation was observed for all the four solid concentrations at four different gas flow rates in both creep test and time sweep test (see supplementary Fig. S2 and Fig. S3). The uncertainty of change in this observation is ±5% if the instrument is not calibrated properly.

The main possible reason behind xanthan gum solidifying behaviour can be, change in molecular structure at a micro or macro level due to the injection of nitrogen gas, which results in a more entangled structure by altering crosslinking. In the same way, the thickening behaviour has been observed in similar polysaccharide materials such as mamaku gum (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 Pricl 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 stress corresponding to linear region. Bobade et al. (2017) have shown that increase in gas injection rate reduces the viscoelastic properties of sludge using same experimental setup and at strain and stress corresponding to linear region. This reduction of viscoelastic properties of sludge is due to breakdown of floc structure as a result of imposed shear by gas injection. Moreover, the creep tests done under similar condition showed that sludge deforms more by increasing gas injection flow rate. This means weaker structure (see insets in Fig 6). Bobade et al. (2017) proved weakening of sludge structure through environmental scanning electron microscopic analysis by observing more porous structure.

4. Conclusion

In this present study, we investigated how the rheological properties of xanthan gum at different solids concentration changes due to gas injection.

In flow region, the flow curve of xanthan gum showed negligible change in the apparent viscosity of in xanthan gum solution as gas injection flow rate increased. However, in linear viscoelastic region, the creep test and time sweep test proved that gas injection increased the storage and loss modulus which is an indication of strengthening of molecular structure. This could be due to deformation of the molecular structure of xanthan gum and increasing the crosslinking within an extended structure which will result in higher resistance to stress and showing more solid like behaviour in the linear viscoelastic region. Thus although xanthan gum behaves similar to the sludge in the liquid regime, the behaviour of xanthan gum contradicts with the sludge behaviour in the solid regime which means Xanthan gum is not suitable as a model fluid for sludge under gas injection below yield stress point.

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