

Evaluation of Some Rheological Properties of Xanthan Gum

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Abstract—The squeeze flow behavior of a shear thinning fluid was studied in this paper using squeeze tack technique. The test material was firstly squeezed out between two parallel circular plates, then it was relaxed for 1.5min and finally was separated at a predefined pulling velocity. From the flow curves obtained in the squeeze tack experiments, the rheological parameters, including yield stress in tension and in squeeze, have been calculated. The results show that the squeeze-tack test can be used for the analysis and evaluation of shear thinning fluids. Although the experimental results may not be considered complete, it can be seen that the stresses and tensile stresses have a linear relationship with goes through the origin.

Keywords—squeeze flow technique; xanthan; yield stress; shear thinning fluid

I. INTRODUCTION

Xanthan Gum (also called Xanthan) is a naturally occurring powdered polysaccharide, created through the fermentation of sugars (glucose or sucrose) by the bacteria *Xanthomonas campestris*. It is often used as an additive in foods and as a thickener and emulsifier in cosmetics. Xanthan has been studied for use in construction materials as a binder for soil stabilization when constructing roads in Sri Lanka [1], but these studies have not fully evaluated the applicability of Xanthan in construction, especially its rheological properties. Xanthan additives have been used in food and cosmetics, as thickeners, emulsifiers, etc. In construction, Xanthan has been studied and applied in bridge construction materials as adhesive binder to stabilize the ground when constructing roads in Sri Lanka [1], viscosity control additives in self-compacting concrete [2], etc.. In general building material technology, Xanthan is used as viscosity control admixture, thickener, etc. in concrete. However, in order to master the application of Xanthan additives in construction, the study of Xanthan's rheology and the proposition of specific application directions for construction is very necessary and highly practical.

II. EXPERIMENTAL SETUP

Squeeze test has been widely used to determine the flow properties of highly viscous pastes [3-5], as it overcomes some of the common problems of conventional rheometry such as slip, disruption of plastic materials, and the difficulty to load very thick and fiber-containing fluids in rotational devices [6].

Squeezing technique can characterize cementitious materials by compressing a cylindrical specimen between two parallel surfaces with controlled force or displacement rate. This method has been used by various researchers for characterizing the rheological behavior of cement pastes [4], Herschel Bulkley fluid [5], Bingham plastic [7], etc.. The typical load vs. displacement profile of a constant velocity squeeze flow experiment was determined in [8]. It can be used to obtain the rheological parameters of testing materials, including yield stress and viscosity. Direct measurements of yield stress are uniquely performed by stress-controlled rheometry [9-11]. Squeeze flow tests carried out with constant displacement velocity do not allow such direct measurements [6, 11] since the material flow occurs regardless of the existence of the materials' yield stress, unless the force required to overcome this value exceeds the load limit of the testing device.

In this work, the testing material (Figure 1) is firstly compressed/squeezed between two parallel surfaces until a predefined thickness. Then a relaxation period of 1.5min follows and it is finally separated with a predefined tack velocity. A typical curve of squeeze-tack experiment is presented in Figure 2, in which three periods can be observed, including squeeze, relaxation and tack.



Fig. 1. Xanthan in (a) powder and (b) gel form.

It is possible to conduct indirect yield stress determination by the extrapolation of the flow curves in the squeeze stage of the experiment. The yield stress is calculated by dividing the maximum force recorded by the area at that time:

$$\sigma_s = \frac{F_s^{\max}}{A_s} \quad (1)$$

where A_s is the average area of the testing sample at the moment that the recorded load is max and:

$$A_s = \frac{V}{h'} = \frac{\pi R^2 h}{h'} \quad (2)$$

where h is the predefine thickness of the test mortar, R is the radius of the sample and h' is the mortar's thickness at the moment of the maximum squeeze force.

Similar calculations with the yield stress at tensile stage can be performed in order to investigate the variation of the material's resistance in tack measurement.

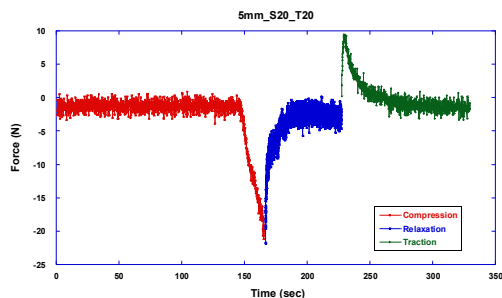


Fig. 2. A typical curve obtained in a squeeze tack test.

III. RESULTS AND DISCUSSION

Firstly, a controlled stress experiment was performed in order to plot the relationship between shear rate and shear stress. The applied stress was gradually increased until the appearance of material flow inside the vane. The result of this part of the experiment is the loading curve as can be seen in Figure 4.

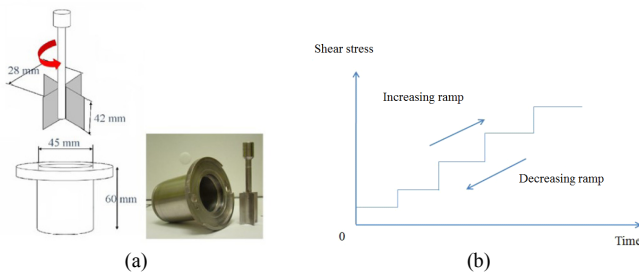


Fig. 3. Stress controlled experiment.

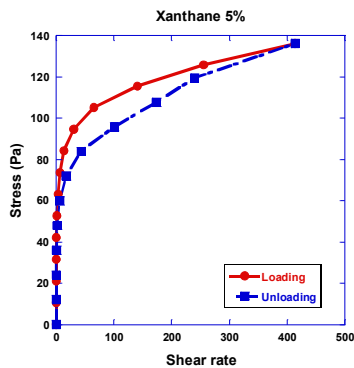


Fig. 4. Flow curves experimental results determine the testing material is shear thinning.

Then the applied stress was gradually decreased until the recorded shear rates approach 0. The result of this part is the unloading curve in Figure 4. The test procedure may be seen in Figure 3. The result in Figure 4 indicates that the test material is shear thinning, a non-Newtonian behavior of fluids whose viscosity decreases under shear strain. The flow curves obtained in the squeeze step can be seen in Figure 5. The subfigures correspond to initial material thicknesses of 1mm, 2mm, 3.5mm, and 5mm. During the squeeze step, the test material was squeezed to the previous set-up thickness and the normal forces were recorded as a function of displacement/time. The squeeze stress was then calculated. From the obtained graphs, it is possible to evaluate the role of the sample thickness to the rheological properties of the material by displaying the force relational graphs over time on the same general chart. From each of the graphs, the maximum tensile force value, corresponding to the yield tensile stress, can be calculated.

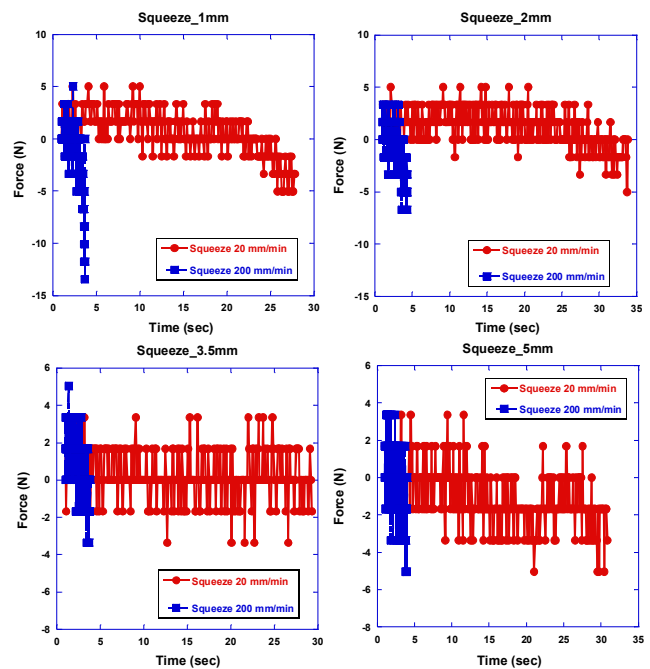


Fig. 5. Flow curves obtained in the squeeze experiments.

At the gap of 1mm, we can see a big difference between the recorded squeeze forces at different squeeze velocities. The recorded values are much higher in the case of high squeeze velocity (200mm/s) compared with those of the case of low squeeze velocity (20mm/s). These two sets of recorded forces come close as the predefined thickness of the material increases. It can be easily seen in Figure 5 that at the gaps of 3.5mm and 5mm, the squeeze speed does not affect to the squeeze forces. Figures 6 and 7 show the variation of normal force values over time for different sample thicknesses. It can be noticed that these measured force values fluctuate greatly around certain equilibrium values. The amplitude of oscillation is increasing at the end of the tensile test.

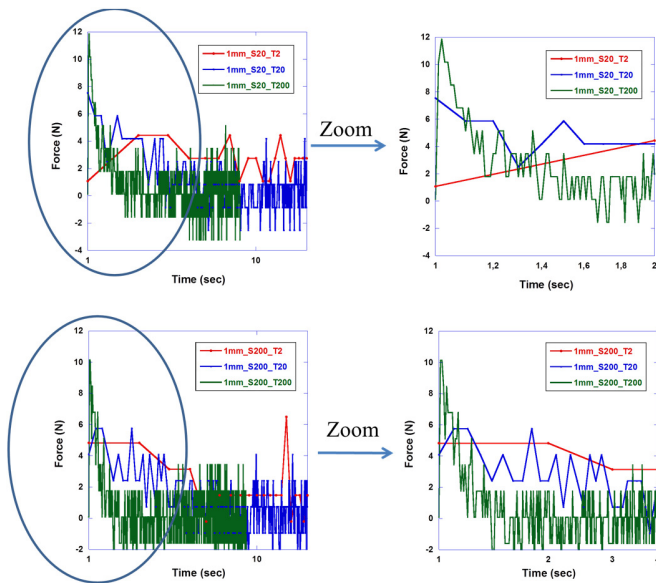


Fig. 6. Flow curves obtained in the tack measurements, for material thickness $h=1\text{mm}$.

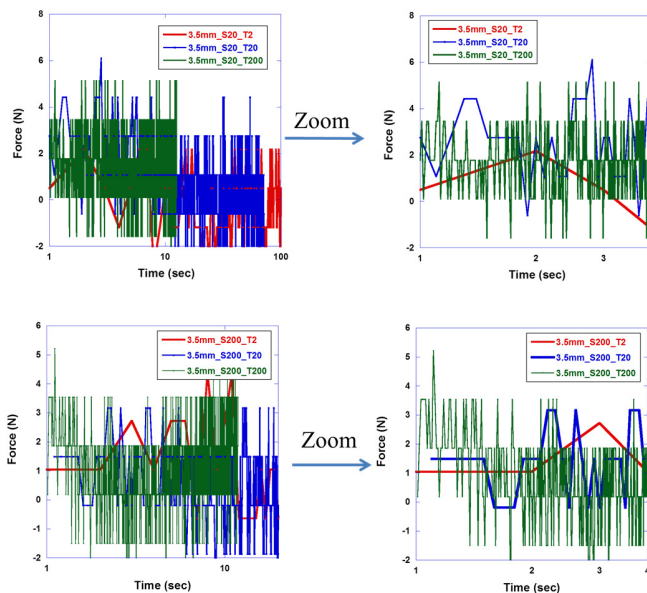


Fig. 7. Flow curves obtained in the tack measurements, for material thickness $h=3.5\text{mm}$.

It can be seen that the obtained magnified graphs in Figures 6 and 7 are almost similar to the typical graph in Figure 2, especially at low material thickness. The maximum force value recorded will be used to calculate the tensile stress of the test material. The fluctuation with large amplitudes of recorded normal forces also shows that later calculated stress values will also fluctuate. This should be noted when commenting or giving future trends. Figure 8 shows a visual image after the end of the experiment. As it can be seen, Xanthan gel is almost concentrated in the center of the lower plate. Almost no gel remains stick on the upper plate and the rest of the lower plate. This shows that the force obtained is mainly the adhesive force between the gel and the surface.

Xanthan gum can be used in self-consolidating concrete. This result is consistent with xanthan applied research in road construction [1], in which Xanthan was used as soil-stabilization binder. Furthermore, this proves that Xanthan gel can be used in concrete to increase the possibility of mold peeling. Figure 9 represents the relationship between tack stress and squeeze stress of the Xanthan gel. Individual areas that are related to each sample thickness value can be easily distinguished. This proves that the smaller the sample thickness, the greater the squeeze stress. Also, the tack stress obtained in the tack measurement significantly increases. As the thickness increases, these individual zones come closer to each other and progress toward the origin. This result can be explained by the arrangement of material particles when the thickness of the layer is high.

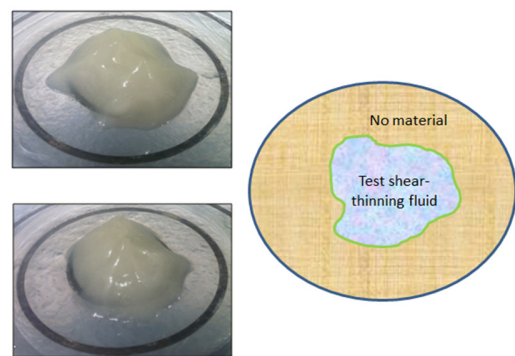


Fig. 8. The rest of materials after tack measurement. The photos were taken after the end of the tack process. The fluid was concentrated in the middle zone of the plate.

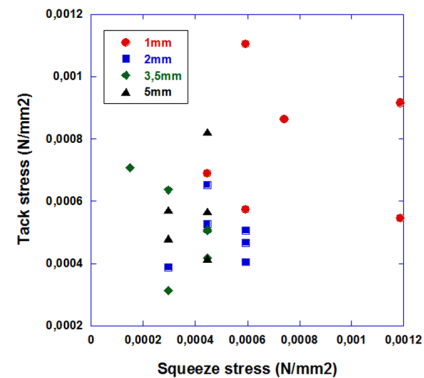


Fig. 9. Tack stress in relations with squeeze stress.

Considering the relationship between the two types of stresses when fixing squeeze and tack speed (Figure 10), one can see a messy behavior when observing Figure 10(a). This can be explained as follows: in the compression and tensile tests, from the recorded force value, shown in Figures 5-7, these values are unstable and fluctuate around an average value. This oscillation is large and noticeable. Thus, the calculated stress also oscillates, leading to the seemingly unordered picture in Figure 10. Because of the chaos of the data in Figure 10(a), especially when the material thickness is high (3.5mm and 5mm), splitting into two parts was attempted, each part corresponding to a different squeeze

speed as in Figure 10(b). With the data fluctuations as analyzed above, it is difficult to comment on the relationship between the two types of stresses. However, in most cases it can be seen that the stresses and tensile stresses have a linear relationship and go through the origin.

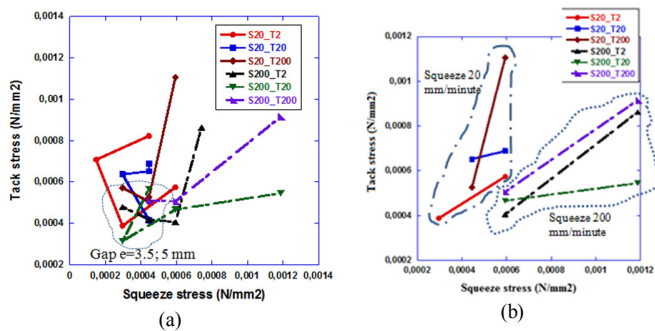


Fig. 10. Tack stress versus squeeze stress at constant test speeds.

Furthermore, the recorded yield stresses in both cases are relatively small, as can be easily seen in Figure 10. That proves that the squeeze-tack experiment is not sufficient to assess the change in stress of Xanthan under squeezing and tacking deformation. The obtained stress value is very small, showing the ability to apply Xanthan in construction to adjust the ability of formwork peeling.

IV. CONCLUSIONS

Squeeze flow behavior of a shear thinning fluid was studied using the squeeze tack technique. The test material was firstly squeezed between two parallel and circular plates, then it was relaxed for 1.5 minutes, and finally it was separated at a predefined pulling velocity. From the flow curves obtained in the squeeze tack experiments, the rheological parameters, including yield stress in tension and in squeeze, have been calculated. The results show that the squeeze-tack test can be used for the analysis and evaluation of shear thinning fluids. Although the experimental results are far from complete, in most cases it can be seen that stress and tensile stress have a linear relationship and go through the origin. The recorded normal forces also indicate that the squeeze flow experiment is not really suitable to study large sample thicknesses. When the thickness is greater than 2mm, the recorded force values fluctuate vividly around a certain equilibrium value. Therefore, the results of stress calculation are for reference and forecast only.

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