Cementbaserade injekteringsmedels reogram: instabilt flöde och inverkan på injektering

Cement grout rheological flow curves: unsteady flow and impacts on grouting

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Sammanfattning

Cementbaserade injekteringsmedels reologiska egenskaper har en viktig inverkan på dess inträngningsförmåga i bergssprickor. Medlen klassificeras ofta som Bingham vätskor, med en karakteristisk flytgräns och viskositet. Egenskaperna bestäms utifrån reogram som tagits fram med hjälp av en rotationsviskosimeter. Mätdata har potentiella felkällar såsom glidning vid en fast begränsningsyta samt inneboende egenskaper hos suspensioner, som tixotropi, separation och ett instabilt flöde vid låga deformationshastigheter. Dessa effekter leder ofta till felaktiga tolkningar av reogramen när tex Binghammodellen används. I föreliggande artikel diskuteras reogram från mätningar på injekteringsmedel med vattencementtal 0,6 och 0,8, med rotationsviskosimeter, olika geometrier och kontrollerade deformationshastigheter. Vi visar effekterna av varaktigheten mellan stegen av ändrad deformationshastighet och hur bra de olika geometrierna är på att förhindra glidning. Påverkan på de reologiska egenskaperna diskuteras slutligen inom ramen för RTGC (Real Time Grouting Control) teorin.

Summary

The rheological properties of cement grouts play a key role in determining the final spread in rock formations. In terms of flow properties, cement grouts are normally classified as non-Newtonian Bingham fluids with a characteristic yield stress value (cohesion) and an apparent viscosity. Grout rheology is usually determined from flow curve data using a rotational rheometer with a concentric cylinder geometry. This data is prone to measurement artifacts such as wall slip coupled to the intrinsic characteristics of cement grouts: thixotropy, sedimentation and unstable flow at low shear rates. These effects often lead to incorrect interpretations of the flow curves when using constitutive models such as the Bingham

model. Within this paper, we discuss flow curves obtained from rotational tests in controlled shear rate mode, carried out on cement grouts with water to cement (w/c) ratios 0.6 and 0.8 using different concentric cylinder geometries. We then describe the effect of duration during the shear rate steps in the flow curve measurement and how effective the different geometries are in eliminating slip. The impacts of these flow effects on the rheology and consequently the estimated grout spread are then discussed in line with the existing Real Time Grouting Control (RTGC) theory.

1 Introduction

The rheological properties of cement grouts determine to a large degree the flow behavior and the final spread that is achieved in grouted rock formations [1]–[4]. Without significant contributions from hydration, cement grouts' rheological behavior are known to be thixotropic (reversible shear thinning viscosity) yield stress fluids. This rheological behavior is often simplified by the use of flow models e.g., the Herschel-Bulkely and the more commonly used, two parameter Bingham model i.e., $\tau = \tau_B + \mu_B\dot{\gamma}$; where τ_B is the Bingham yield stress, μ_{pl} the Bingham or plastic viscosity, $\dot{\gamma}$ the shear rate and τ the yield shear stress [5]. The fitted Bingham properties are then used in analytical solutions to estimate the maximum grout spread, I_{max} and the corresponding characteristic time of grout spread, t_0 [6], [7], [3]: $I_{max} = \Delta p \cdot b/2\tau_0$; $t_0 = 6\Delta p\mu_g/{\tau_0}^2$. Where, Δp is the effective grouting pressure (difference between imposed grouting pressure p_g and opposing ground water pressure p_w in the rock); $\mu_g$is the grout viscosity (Bingham), b the fracture aperture and τ_0 the yield stress (Bingham) of the cement grout (see [2], [8], [9]).

Rheometric data to describe the fresh flow properties of cement grouts is often obtained from rotational shear flow tests, and it is to this data that the Bingham model is fitted [5]. However, the Bingham model like most constitutive flow equations assumes ideal 'simple' yield stress behavior in which all shear rates are attainable under steady state conditions. With the Bingham model, yielding of the cement grout is marked by a sharp transition from 'solid-like' to 'liquid-like' behavior upon increasing shear stress/shear rate. On the contrary, yielding in complex fluid is not the case especially for complex thixotropic materials e.g. cement grouts, bentonite drilling muds [10]–[13]. For these fluids it has been shown that there exists a certain range of shear rates for which no-steady, homogenous flow is attainable. This range of shear rates that corresponds to the yielding stress is marked by the critical shear rate ($\dot{\gamma}_c$), and applied shear rates ($\dot{\gamma} < \dot{\gamma}_c$) below the critical value result in flow stoppage (shear banding). Shear banding has been attributed mainly to the intrinsic nature of thixotropic yield stress fluids, where in thixotropic build-up of the materials microstructure competes with the mechanism of structural breakdown due to shear [14]. A flow phenomenon such as shear banding complicates the interpretation of flow curve measurements since the flow behavior within the shear banding regime is unstable and does not correspond to steady flow.

In addition to shear banding inherent in the material, shear localization due to the rheometer geometry e.g., in the concentric cylinder (Couette) needs to be noted for the correct use of flow curve data. With shear localization the fluid material within the rheometer gap is not completely sheared at low shear rates at radial positions furthest away from the rotating bob i.e., if the stress is not high enough to overcome the yield stress within the entire gap [13]. Other, flow phenomena such as sedimentation and wall slip have been described in literature as having a significant influence on the flow curve data. Wall slip is normally reduced by the use of roughened geometry walls, and for suspensions such as cement grouts the vane tool is often used. However, recent studies with the vane have shown that this geometry is also prone to significant sedimentation, secondary flows and non-cylindrical flow streamlines. Sedimentation is known to increase the measured stresses, whereas wall slip results in lowered stress values especially in the low shear rate range (i.e., for grouts typically below ~10 1/s). Such artefacts must be taken into consideration when using the vane [15]–[17].

In this paper, we present rotational flow tests carried out on typical cement grouts. For our tests the focus is on showing the influence of rheometer geometry and measurement interval on the measured flow curves, particularly below the critical shear rate where negatively sloped branches in the flow curves were observed, as evidence of unstable flow. We also describe the effects of slip and the unstable flow region on the Bingham fitting procedure. Lastly, we then recommend some reasonable test procedures and also consider some possible impacts of this unstable flow in grouted rock formations.

2 Experimental methods

2.1 Materials and equipment

Cementa Injektering 30 (CEM I 52,5 N - SR 3 LA) was used for the tests. Two cement grout mixes were prepared at water-to-cement (w/c) ratios of 0.6 and 0.8. A high shear mixer (VMA, Dispermat CV30, and D-51580) was used for dispersing the cement in water for ~4 minutes at ~10 000 rpm. This type of mixing resulted in fully dispersed and homogenous grouts.

A TA AR-2000ex rheometer with 3 different rotors (smooth, grooved, 4-blade vane) and 2 different cups (smooth, grooved) was used (i.e., 3 different setups). The effective shear gap for all geometries was 1 mm (see Figure 1).

2.2 Measurement of flow curves

Once the grout was mixed it was transferred (~2 minutes) and loaded into the rheometer geometry. The grout was then pre-sheared for 20 s, and immediately subjected to a rest period of 30 s to eliminate loading stresses and to establish a common reference state for all grout mixes. The flow curve measurement was carried out by applying increasing and decreasing flow sweeps in controlled shear rate (CSR) mode. Only the down curves are considered here for analysis, since the data obtained from down curves is more representative of the grouting condition i.e. starting from higher flow rates and also this data is more repeatable within ~10%. The applied shear rates ranged from (300 down to 0.001) 1/s, with 10 points per decade. Different measurement intervals (t_w) of 4 s, 24 s and 40 s were used per shear rate point. These measurement intervals were chosen based on different results that we had seen in some flow curves, especially at low shear rates whilst testing at our laboratories. All tests were then carried out at a set temperature of 20 °C. A fresh batch was prepared for each test, resulting in a total of 18 tests i.e., 3 geometries and 3 test times per cement grout mix.

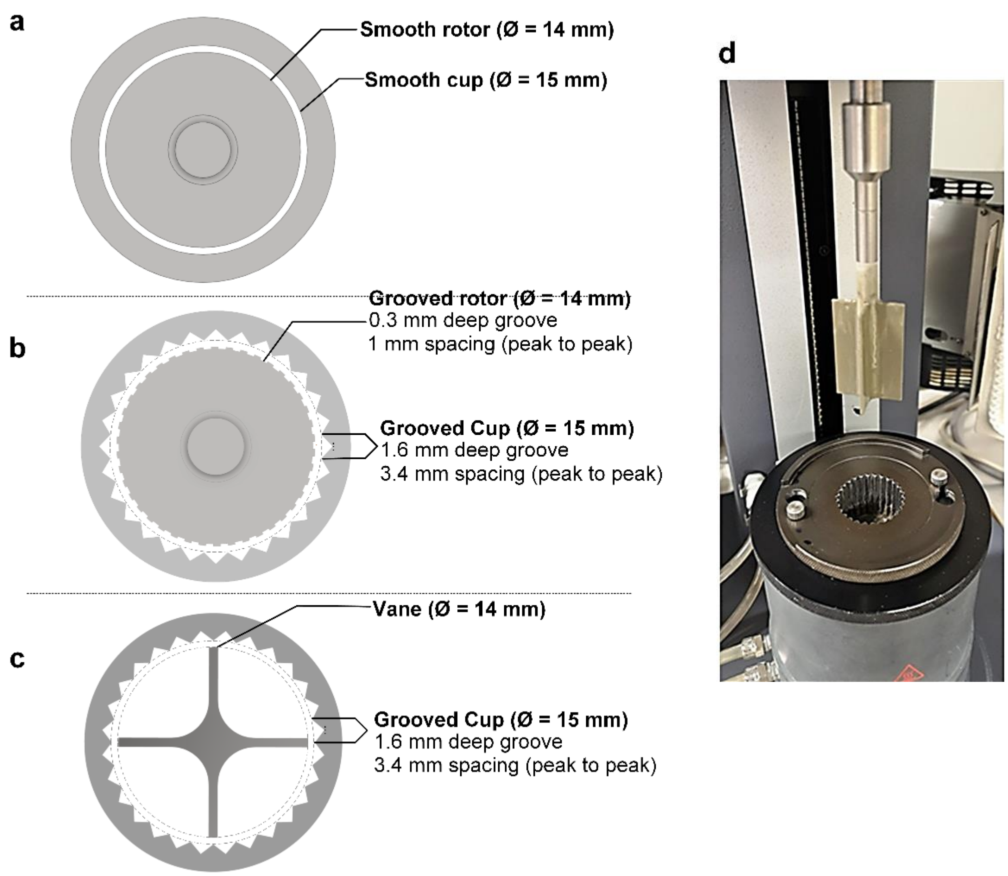

Figure 1. Geometries used for the flow curve measurements (a) Smooth rotor and smooth cup (b) Grooved rotor and grooved cup (c) 4-blade vane and grooved cup, all with a 1 mm measurement gap (d) Example of Vane setup on a rheometer.

3 Results

All our flow curve results are presented and analyzed in linear-logarithmic format in order to clearly show the interesting flow behavior at low shear rates, that is often compressed against the vertical shear stress axis in a linear-linear plot (Figure 2).

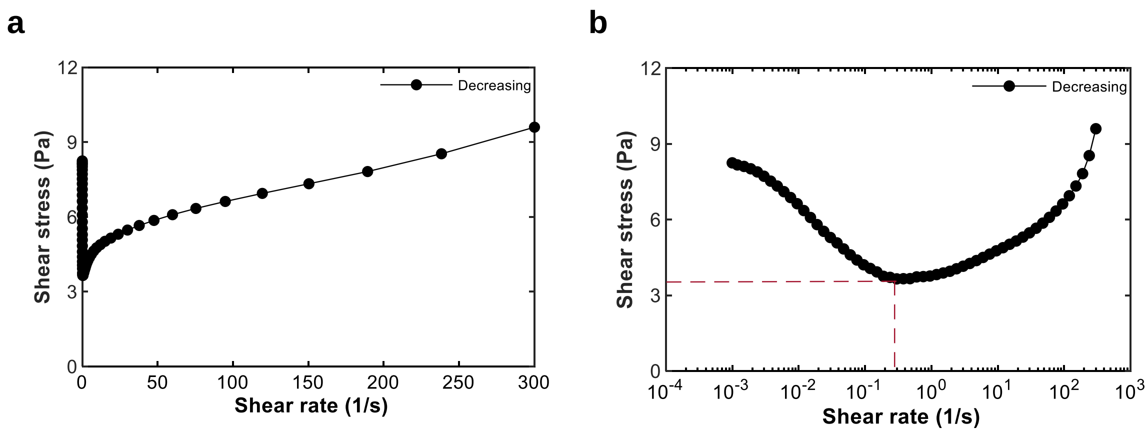

Figure 2. Down curve of cement grout (w/c = 0.8) measured in the vane setup at t_w =24 s measurement interval (a) in linear axis plot (b) linear-log format.

All flow curves showed an unstable flow branch below a critical shear rate $\dot{\gamma}_c$. The slope of this branch was more pronounced in tests carried out at long time intervals, and was noted to increase with time interval t_w. Figure 3 illustrates the principle observations.

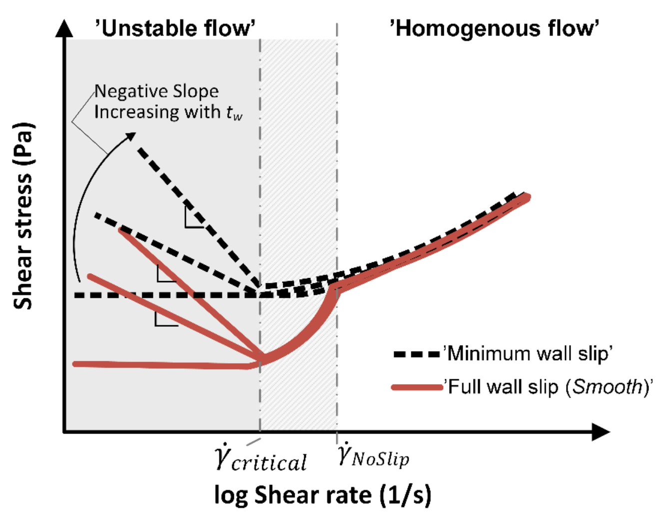

Figure 3. Schematic showing the unstable flow region, slip and homogenous flow region

The unstable branch below the critical shear rate was more visible in the vane tests (Figure 4). This could also be an effect of sedimentation and structural build-up that have been reported to be more significant in this geometry. The vane has the largest space and volume of grout particles that are unsheared and remain stationery between the rotor blades. At low shear rates structural build up dominates over breakdown due to shear; also, with flow localization at low shear rates, the combined effect is that of increased sedimentation due to thixotropic build-up of flocs in the grout material. Slip was observed in the smooth-smooth and grooved-grooved geometries. A surprising result was that slip (lowered shear stress values especially below ~10 1/s) was also observed in measurements carried out with the grooved geometry. This shows that such a magnitude of roughness (~300 µm) is not sufficient for eliminating slip with such particles. Slip elimination was noted from the vane measurements at $t_w = 4$ s.

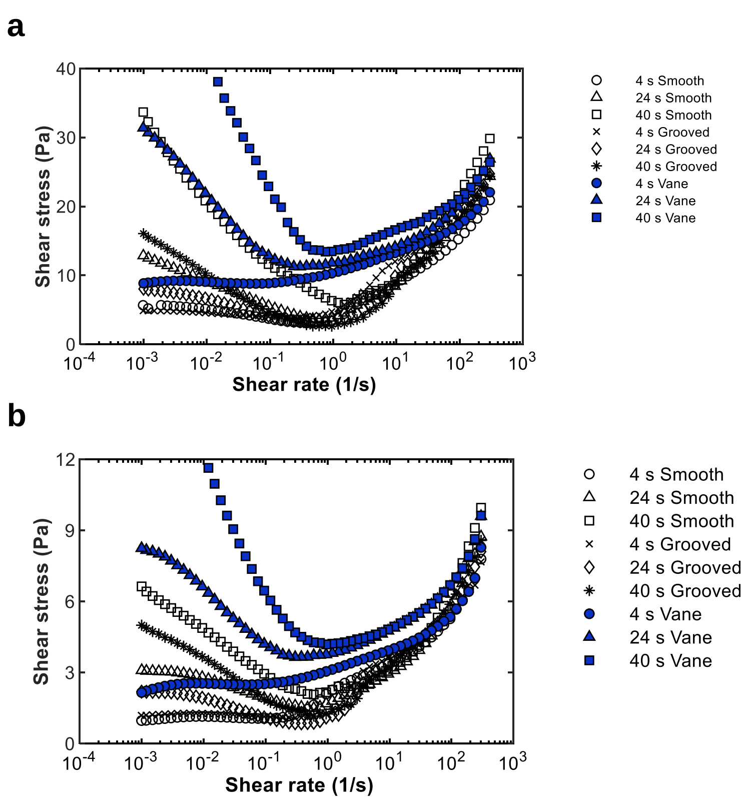

Figure 4 Flow curves for cement grouts at (a) w/c = 0.6 (b) w/c = 0.8

To analyze the effects of flow artefacts such as slip and the unstable flow below the critical shear rate on the fitted Bingham properties, we then carried out Bingham fitting within two shear rate ranges The first range for fitting was from the critical shear rate up to the maximum measured shear rate ($\dot{\gamma}_c$ to $\dot{\gamma}_{max}$); whereas, the second shear rate range was from the lowest point on the homogenous branch where no slip was observed to the maximum shear rate ($\dot{\gamma}_{NS}$ to $\dot{\gamma}_{max}$). The no-slip point ($\dot{\gamma}_{NS}$) was marked by the characteristic lowering of the flow curve as it departed from the Bingham curve fit in the first range ($\dot{\gamma}_c$ to $\dot{\gamma}_{max}$) (see Figure 3 and 5).

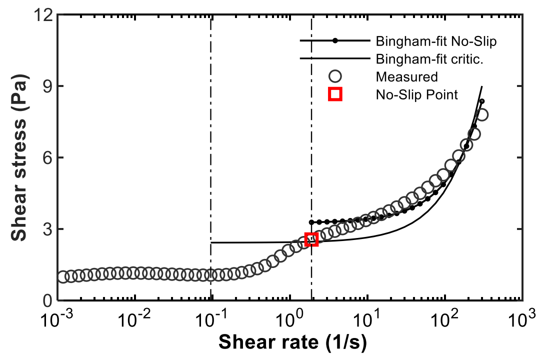

Figure 5. Example flow curve of cement grout (w/c = 0.8) showing the No-slip point and Bingham fits over the two shear rate ranges.

The results of the fitting for both cement grouts showed that the critical shear rate was within the range ~(0.1 – 1) 1/s and always lower than the lowest no-slip shear rate that reached a maximum of ~10 1/s (Figure 6).

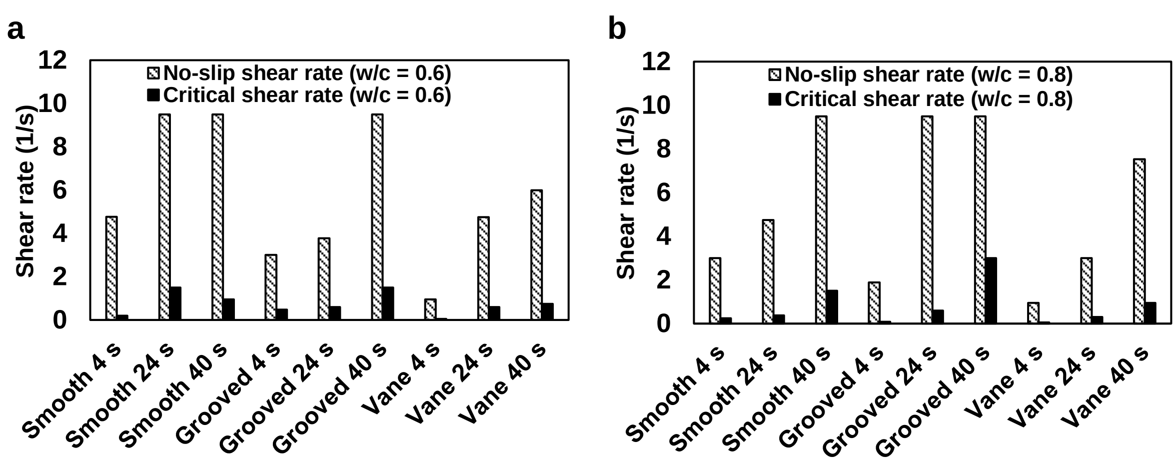

Figure 6. Values of the critical and No-slip shear rates

As expected, the fitted Bingham yield stress within the range ($\dot{\gamma}_{NS}$ to $\dot{\gamma}_{max}$) was consistently lower than that in the wider range ($\dot{\gamma}_c$ to $\dot{\gamma}_{max}$). The difference was close to ~%50 for the smooth geometry, mainly due to slip that lowers the lower shear rate range data (Figure 7). The vane data had the least difference in the two values (~10%), due to less slip effects.

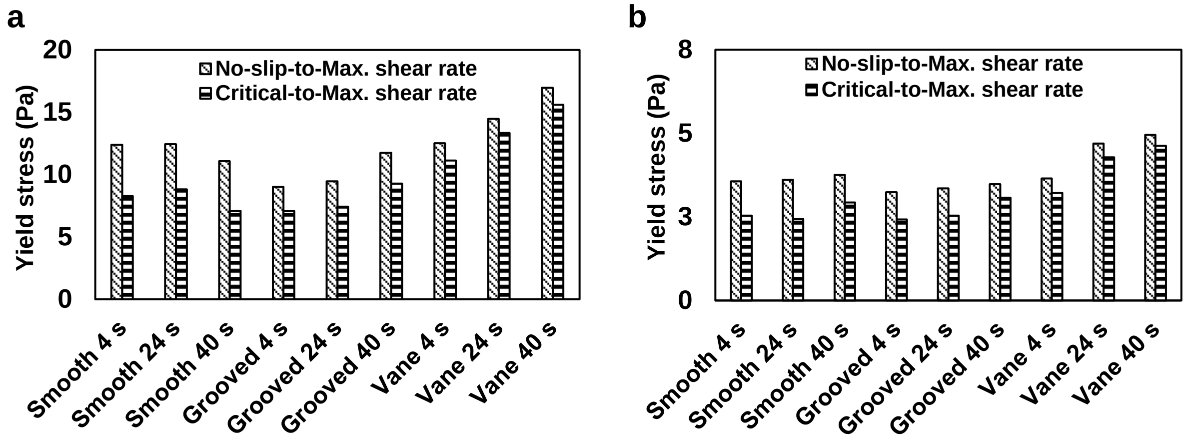

Figure 7. Comparison between yield stresses determined in the two shear rate ranges (a) w/c = 0.6, (b) w/c = 0.8.

As a final step in our analysis we assessed how well the Bingham model fitted the data based on the Normalized Root Mean Square Error (NRMSE). Generally, the Bingham model was a better fit (lower NRMSE Figure 8) for the less concentrated grout (w/c = 0.6 , suggesting that the model is not suitable for effectively describing the highly shear thinning behavior of concentrated suspensions. Furthermore, the vane data also fitted the vane data due to less slip variations in the lower shear rate range data that affected the other two geometries (Figure 8).

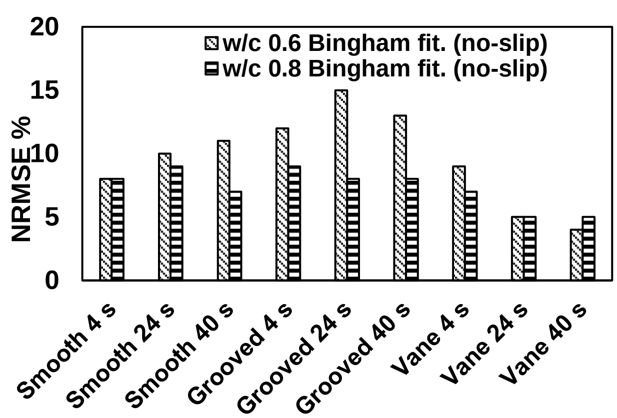

Figure 8. Evaluation (NRMSE) of the Bingham model fit over the two shear rate ranges

4 Conclusions and recommendations

In summary, we showed that for the cement grouts there exists some critical shear rate associated with the yielding transition below which no stable flow can be achieved.

By conducting tests in the CSR mode of the rheometer, we showed that an unstable flow branch can be observed, with a slope that increases with the measurement interval of the flow sweep. The vane data had at longer measurement intervals (t_w 24s; 40 s) the largest measured stress values, possibly due to the combined effect of sedimentation and structural build-up that are more likely to occur in this geometry due to the large amount of trapped particles that remain unsheared (Figure 4). Moreover, flow localization at low shear rates increases the extent of the unsheared region in the gap at low shear rates (below $\dot{\gamma}_c$). Wall slip mainly affected flow data in the smooth geometry and unexpectedly that from the grooved rotor, showing that the roughness value required to eliminate slip needs to be dimensioned appropriately to the particle size distribution of the suspended particles.

By considering the case of grouting in real fractures, the observed flow localization suggests that it might be the case that flow stoppage occurs faster than predicted by assuming simple yield stress fluid behavior due to the critical shear rate. Such conditions might occur at longer penetration distances when the grouting pressure is low, or when grouting is momentarily stopped and started after thixotropic build-up in the grout forms a material structure that is stronger than the initial one. As a next step in grouting research, the overall result of such flows in large fracture networks therefore needs to be assessed by incorporating thixotropic flow models e.g., in simulations, for improved grouting design and execution.

4.1 Recommendations

Based on our tests we recommend the following procedures for reasonable determination of grout properties using the Couette geometry for rotational flow tests:

1. Bingham fitting be carried out only within the homogenous flow branch after slip has been eliminated from flow curve data. It must be noted that, the use of much higher values of the no-slip shear rate in order to avoid the unstable region and slip, has the consequent result of an increased fitted Bingham yield stress and lower plastic viscosity. Thus, it advisable to consider the characteristic shear rates that are relevant to the application in order to appropriately determine the required shear rate range for Bingham fitting.
2. Our results show that in the absence of slip, the grooved and smooth geometries may be more suitable for flow-curve measurements compared to the vane geometry since the vane results in much higher yield stress value especially for long duration measurements where sedimentation and thixotropic build up are more significant.
3. To use the vane geometry for direct yield stress measurements (e.g. stress response), and not for long duration flow curve tests.

Acknowledgement: We are grateful to SBUF, the Development Fund of the Swedish Construction Industry for funding this work.

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