PARTICULATE REFERENCE MATERIALS FOR RHEOMETERS

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ABSTRACT

Rheometers are typically calibrated against a standard oil of known viscosity. This is a valid procedure when the geometry of the rheometer is simple, since the viscosity can then be calculated using validated analytical formulas. In principle, this procedure could be applied to measure rheological properties of a granular material such as cement paste using a parallel plate or a coaxial geometry. Unfortunately, if the rheometer geometry is not a standard geometry, and further, if the material is non-Newtonian, then the usual analytical formulas no longer applicable leading to an inaccurate instrument calibration. In this case, a different calibration strategy must be employed. We propose a strategy that utilizes a combination of a granular reference material and computational simulation of the flow in the rheometer. The granular reference material should have a similar particle size distribution, about the same rheological properties (e.g., viscosity and yield stress) as the material to be tested, and these properties should be temporally stable. The computational simulation will help determine the flow pattern and the shear rate in the non-standard rheometer. This paper presents a test case for development of a methodology for calibration of non-standard rheometers.

INTRODUCTION

Cement paste, mortar, or concrete are considered fluids and therefore, their fresh properties should be determined using rheological measurements. Unfortunately, due to the size of the particles, especially for mortar and concrete, traditional parallel plate or coaxial cylinder rheometer geometries are not appropriate, as the gap between the shearing planes is usually less than 1 mm while sand particles can have a maximum effective diameter up to 5 mm and the coarse aggregates are larger than 5 mm. The gap between the shearing planes should be at least 3 to 5 times larger than the largest particles. In recent years, several new rheometer configurations have been marketed to accommodate these materials (1). It was determined that all the rotational rheometers consistently measured the flow behavior of concretes but it was also shown that (2, 3) the absolute values for yield stress and plastic viscosity differed. In the ACI report (1), other rheological techniques for cement paste, mortar, and concrete were described. Again, the rheological properties measured were method dependent. This lack of standard methodology affects the sharing of data among laboratories and the determination of the intrinsic properties of a material. The solution would be to have a reference material whose rheological properties such as yield stress and plastic viscosity are well-characterized. Rheometers could then be calibrated against this well defined material. ACI 238 has opened the discussion for sharing potential candidate particle. This paper will examine the characteristics required by such a material and present a potential candidate. The determination of the fundamental rheological properties of such a material will only be possible by a synergy between experimental data and computer simulation.

REFERENCE MATERIAL BACKGROUND

Reference materials to be used in calibrating rheometers for cement paste, mortar, or concrete having the following attributes:

- Cement paste is a Bingham liquid with particles in the size range from 1 μm to 100 μm.
- Mortar consists of cement paste with the addition of sand or particles that range in size up to a few millimeters.
- Concrete consists of mortar with the addition of coarse aggregates or particles that range in size up to tens of millimeters.

All these materials show a yield stress and a plastic viscosity that vary with the particle concentration. Therefore, ideally the reference material should be composed of a Newtonian liquid and particles of various sizes to replicate the range from cement paste to concrete, and whose properties do not change with time, i.e., no hydration like cement paste.

In practice, rheometers are usually calibrated using a standard oil. The oil is Newtonian and contains no particles. Ferraris et al. (4) did an extensive study using a standard oil and two rheometer geometries designed for cement paste and mortar. The geometry for the cement paste was a parallel plate (diameter 35 mm) with serrated surfaces. The mortar was also tested by a parallel plate geometry but with a different design of the plates and a confinement ring [4]. They found that a correction factor should be applied to the nominal gap to take into account the serration. Only the serrated parallel plate will be used in this paper.

Another commonly used geometry is the vane rheometer or coaxial rheometer. The gap in a coaxial rheometer is narrow as the ratio of the diameter of the container and the inner cylinder is supposed to be close to 1 to ensure that the shear rate is linear in the gap. This restriction obviously makes such a rheometer not applicable for measuring mortar or concrete unless the gap is increased significantly. In this case, the shear rate is not linear and the shear stress is no longer calculable using the known equation for such a rheometer. The vane rheometer does not have a gap restriction but the shear rate and shear stress are not known. This geometry is often used to avoid slippage on the shear plane. Only the rotational speed and the torque can be measured and used to compare different materials. To transform the results in fundamental units, a reference material is needed. Tests were performed using a vane rheometer with a container with a inner diameter of 43 mm and a vane with a diameter of 22 mm and a length of 16 mm. The torque resulting from the material resistance was measured at the central rotating tools.

MATERIAL USED

Silica fume in water was considered as a candidate reference material to simulate cement paste. The silica fume used has a density of 2.5 g/mL and mean particle size of 0.6 μm as per the manufacturer. Ultrasound was used to disperse the material. These particles are much finer than cement but they do not react with water. The material was prepared by mixing water and silica fume in a high shear blender in mass ratio of 1.5 (water/silica fume). High range water-reducing admixture (polycarboxylate based) was also added at a dosage of 0.2 % by mass of silica fume. This would correspond to a 21 % silica fume volume concentration. Care was taken that no calcium contamination occurred to avoid agglomeration of the silica fume.

RESULTS

The shear stress and shear rate relationship of silica fume paste was measured using a parallel plate rheometer with serrated plates 35 mm in diameter [5], shown in Figure 1. The gaps between the plates were 0.5 mm. If the correction of the zero gap of 0.27 mm is used, as described by Ferraris et al. [4], no significant modification of the curves is seen. Therefore, it is assumed that the gap correction here is not necessary. Also, it can be noticed that the behavior seen is not Bingham. This could create problems as a reference material because if the torque measured depends on the shear rate at least at the lower values, calibration of a rheometer with an unknown shear rate would lead to an unknown torque. Figure 2 shows the results obtained with the same material but tested using a vane rheometer. In this rheometer, the shear rate and the shear stress cannot be calculated as the geometry is not standard. Modeling would be the only method to be able to extract the material properties from these data.

MODELLING BY SMOOTH PARTICLE HYDRODYNAMICS (SPH) METHOD

The interpretation of the experimental results is not always easy due to the lack of an analytical methodology to extract shear rates and shear stresses in fundamental units. Therefore, a numerical simulation, predicting the bulk and interfacial properties of complex fluids, has been used to complement and guide experimentation. Numerical simulations of non-Newtonian flows in complex geometries have often been based on a macroscopic approach where one numerically solves the conservation laws together with a suitable rheological constitutive equation. In this area, many numerical schemes have been proposed, generally based on finite difference, finite element (FEM), finite volume

(FVM) or boundary element methods (BEM). All of these numerical methods are, in essence, Eulerian schemes. As an alternative to classical Eulerian methods, Lagrangian based approaches avoid the complicated evaluation of advective terms and allow for tracing the motion of solid-fluid interfaces and simulating free surface flows without additional difficulty. Since our long term goal is to simulate multiphase complex fluids such as suspensions, a Lagrangian scheme was adopted in the numerical computation. The SPH method [6-7] has been applied to flow of non-Newtonian fluids [8-9].

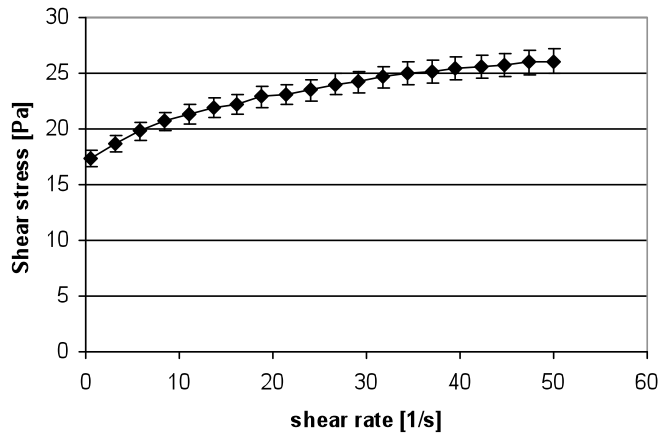

Figure 1: Shear stress vs. shear rate (down curve) of silica fume paste determined in a parallel plate rheometer with a gap of 0.5 mm. The error bars represent one standard deviation.

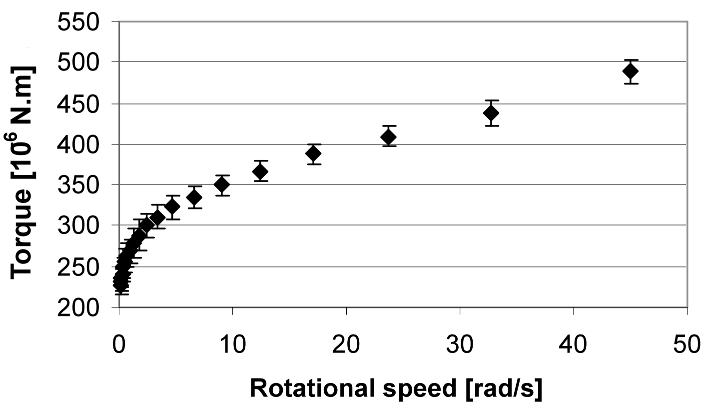

Figure 2: Torque vs. rotational speed of silica fume paste determined in a vane rheometer. The error bars represent one standard deviation.

The tensorial continuum and momentum equations have the form

$$\frac{d\rho}{dt} = -\rho \nabla \cdot \boldsymbol{v}, \tag{1}$$

$$\frac{d\boldsymbol{v}}{dt} = \nabla \cdot \boldsymbol{P} + \boldsymbol{b}\,, \qquad (2)$$

where ρ is the density, $\boldsymbol{v}$ is the velocity, $\boldsymbol{b}$ is the body force, and $\boldsymbol{P} = -p\boldsymbol{I} + \boldsymbol{\tau}$, where p is the hydrostatic pressure at equilibrium and $\boldsymbol{\tau}$ is the extra stress tensor. It is difficult to apply the Bingham model directly in numerical simulations, especially in complex geometries. The difficulty is mainly due to the discontinuity in the constitutive relations; specifically, as the yield point is approached, the presence of the shear rate in the denominator of the Bingham model makes the apparent viscosity diverge. Therefore, a regularized Bingham model will be used [10]:

$$\boldsymbol{\tau} = \left(\eta + \tau_0 \frac{\left[1 - e^{-m\dot{\gamma}}\right]}{\dot{\gamma}} \right) \dot{\boldsymbol{\gamma}}\,, \qquad (3)$$

where m is a parameter related to the transition between the solid and fluid regimes. The higher the value of m, the sharper the shape of the transition.

Figure 3 shows the numerical results for the flow of Bingham fluids in a 2D vane rheometer. The yield stress parameter τ_0 is chosen to be 137.5 Pa and the plastic viscocity 27.5 Pa.s. It can be observed that there are stress concentrations at the tips of the vane blades, as a result of non-uniform flow around the vane blade tips. Also, it was observed that a rotational flow developed between the blades. As a result, relations between stress and shear rate can not be obtained analytically, therefore numerical simulation is need to interpret experimental data. A parallel plate rheometer was not analyzed because it cannot be done in 2D. Note that simulation in the present study only qualitatively shows the potential of the application of SPH method in rheometric flow. The authors plan in the future to generalize the model to 3D and then simulate any rheometer geometry by using material parameters with physical basis.

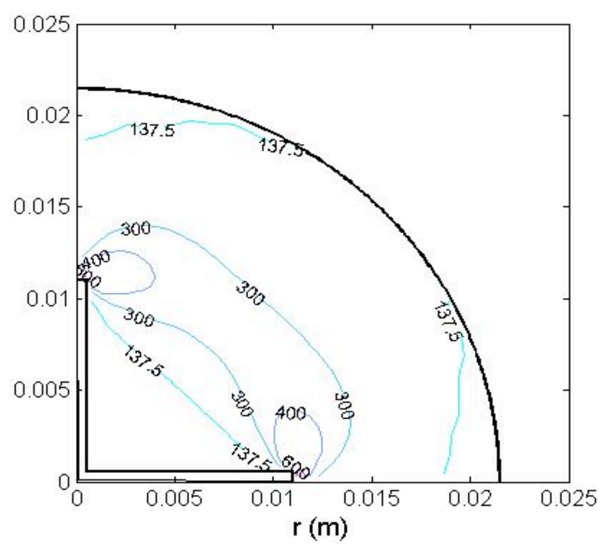

Figure 3. Contour plot of the stress field (Pa): flow of Bingham fluid with a yield stress of 137.5 Pa in vane rheometer (angular velocity: 7.5 rad/s (1.20 rps)). The blades are in the left bottom corner, due to symmetry only one quadrant is shown.

CONCLUSION

This paper presented a study of how to combine modeling and tests to characterize the flow of a particulate material. This method can be used to evaluate reference materials to simulate cement paste. A reference material should have properties that are not changing with time due to hydration or chemical reaction. Silica fume by its particle size and non-reactivity with water, provided that calcium is not present, would seem a promising candidate but has the disadvantage of being an non-Bingham fluid. It seems that a more sophisticated approach including numerical simulation is need for silica fume paste to be used as a reference material for a rheometer with an unknown shear rate. In the future, sand and coarse aggregates will be added to simulate mortar and concrete. Other simulated materials will be investigated by the authors and the American Concrete Institute (ACI) 238.

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