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Development of standard reference materials for rheological measurements of cement-based materials

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ABSTRACT

Rotational rheometers are routinely used for homogeneous materials, but their usage for characterization of a granular fluid like concrete is a relatively new phenomenon. As measurements with rheometers can involve flow in a complex geometry, it is important that they are calibrated with a reference material or an SRM. NIST has produced an SRM for cement paste (SRM 2492) as the first step for the development of a reference material for concrete rheometers. The second step is the development of a reference material for mortar, composed of the SRM 2492 with added spherical beads. Here, material properties, such as viscosity, cannot be measured in fundamental units with certainty, thus modeling was used to determine the plastic viscosity of the SRM mortar which in turn could be compared with experiments. This paper will present the process used to develop the mortar reference material. Measurements and modeling will be presented.

1. Introduction

Self-consolidating concrete (SCC) is defined by American Concrete Institute (ACI) terminology for its ability to flow and consolidate under its own weight without vibration. Therefore, the rheological properties of SCC are the most essential parameters that determine successful placement and consolidation. In the past 10–15 years, concrete rheometers were built to measure such properties. It was found by two international round-robin tests [1,2] that different concrete rheometers do not provide the same viscosity or yield stress value while measuring the same concrete batch. Nevertheless, they rank different concrete mixtures in the same order. Thus, the rheological properties of a concrete can only be specified relative to the rheometer used. This is not an optimal approach for developing specifications and codes that require viscosity or yield stress for general usage instead of the slump or slump flow that is currently used.

Thus, discussion in ACI 238 committee, *Workability of Fresh Concrete*, led to the consensus that a reference material needs to be developed. This material must be similar to concrete (e.g., containing aggregates). The NIST approach, presented here, was to develop a reference material having similar rheological properties as cement paste and then add fine aggregates to simulate mortar and then add coarse aggregates to approximate concrete. The Standard Reference Material (SRM) for paste rheology was developed and it is composed of a solution of corn syrup and limestone fine powder (SRM 2492) [3]. The next step, presented here, is the development of the SRM for mortar rheology. While the rheological properties of a SRM paste can be measured accurately using a laboratory rheometer, the mortar SRM properties cannot be measured with certainty because mortar rheometers cannot be properly calibrated with oil. Therefore, a model was developed to determine the rheological properties to be assigned to the mortar SRM. The goal is that these SRMs will be used to calibrate rheometers to be used to measure mortar or concrete, allowing all rheometers to report data normalized to the same material values.

2. Background

2.1. Rheological measurements

Rheological properties are typically measured using rotational rheometers that essentially shear a material between two surfaces. Usually one surface is stationary and the other is rotating. Various geometries could be used. For this report, we used a vane and a coaxial configuration. Rheological measurements typically produce a shear stress vs. shear rate plot. In cases where the geometry of the rheometer does not allow a direct calculation of the shear stress and shear rate in fundamental units, as for a vane, the rotational speeds and the resulting torques are plotted [10].

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Viscosity [4] is defined as the ratio of the shear stress to the shear rate at a given shear rate. For a Newtonian fluid, it is also equal to the slope of the fitted line of the shear stress–shear rate plot, going through zero, as the relationship is linear. But most granular fluids, such as mortar and concrete, are non-Newtonian. Their most notable characteristic is that they exhibit a yield stress, which is the stress needed to initiate deformation or flow of the material. The two most common methods for measuring this behavior are the stress growth method and the extrapolation from the Bingham test method [5,6].

Most researchers use the method based on the Bingham equation (Eq. (1)) to determine the plastic viscosity and the yield stress. This procedure implies that the plastic viscosity is defined as the slope of the shear stress–shear rate curve and the yield stress is the intercept of the curve at zero shear rate. This point is generally not directly measured, so this constitutes an extrapolation (Fig. 1). The Bingham rheological parameters, yield stress and plastic viscosity, will characterize the flow curve within a range of shear rates, as shown in Fig. 1 and Eq. (1) [4].

$$\tau = \tau_B + \mu_{pl}\dot{\gamma} \quad (1)$$

where τ = shear stress, τ_B = Bingham yield stress, μ_{pl} = plastic viscosity, and $\dot{\gamma}$ = shear rate.

2.2. Model overview

The computational model of fluid flow utilized for this work is based on a Smoothed Particle Hydrodynamics approach [7,8], and Lucy [9,10] that is a Lagrangian formulation of the Generalized Navier–Stokes equations. The Lagrangian formulation is preferred because this approach can give us more flexibility in handling moving boundaries. This approach can account for a spatially varying viscosity that is, for example, dependent on the local shear rates. While details of the model are beyond the scope of this paper, we briefly describe some of its features.

The time evolution of a fluid is represented as a set of particles, located at r_p, carrying local flow information (velocity, density). These particles undergo motion in response to effective "interparticle" forces that are defined by a discretized version of an integral representation of the general Navier–Stokes equations [11]:

$$\frac{\partial \rho}{\partial t} = -\rho \nabla v \quad (2)$$

and

$$\rho \frac{\partial v_i}{\partial t} = -\frac{\partial P}{\partial x_i} + \frac{\partial}{\partial x_k}\left[\mu\left(\frac{\partial v_i}{\partial x_k} + \frac{\partial v_k}{\partial x_i} - \frac{2}{3}\delta_{ik}\nabla v\right)\right] + \frac{\partial}{\partial x_i}(\zeta \nabla v). \quad (3)$$

Here, ρ is the fluid density, P is pressure, v is velocity, μ and ζ are the shear and bulk viscosities respectively.

In these equations the bulk and shear viscosities cannot be taken outside the gradient operator because they can vary in space. The discretization form is very similar in structure to a molecular dynamics simulation and indeed can be related to other approaches like dissipative particle dynamics. Lubrication forces are also included in this model to account for behavior of dense suspensions where there are many solid inclusions in close contact. The lubrication forces have been modified such that the viscosity is dependent on the local shear rate between neighboring sphere surfaces. This approach has been demonstrated to recover simple analytic solutions of flow fields of pipe flow for non-Newtonian fluids and agrees well with experimental data of relative viscosities of suspensions having a Newtonian and non-Newtonian fluid matrix. The full details of this model are given elsewhere [7].

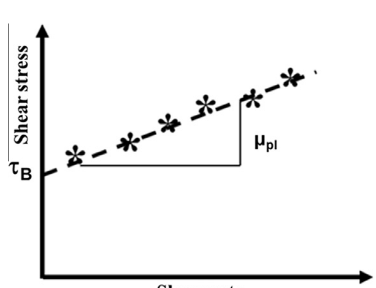

Fig. 1. Bingham model and calculation of the plastic viscosity and yield stress.

3. Experimental work

Reference materials were prepared as stated in the SRM certificate [12] and beads were added to simulate mortar reference material.

3.1. Materials and mix proportions

SRM 2492 is composed of pure corn syrup, water and limestone. The corn syrup is used as supplied by the manufacturer. Its density measured at NIST is 1427 kg/m^3 ± 5 kg/m^3, with a water content of 18.6% ± 0.2% by mass fraction, and its chemical composition is 100% glucose.

The limestone powders were analyzed to determine mineralogical, chemical, and physical characteristics. Table 1 and Fig. 2 show some physical properties and the particle size distributions, respectively. The particle size distribution (PSD) was measured using laser diffraction with either water or isopropanol (IPA) as the suspension media. It should be noted that there is little difference, suggesting that the particles are well dispersed in either medium.

The proportions of the SRM 2492 are shown in the certificate [12] and are:

- Corn syrup: 200 g.
- Distilled water: 63.16 g.
- Limestone: 458.1 g.

The rheological values associated with this material were determined at NIST after a statistical experimental design and are described in a report [13].

Glass beads with diameters, reported by their manufacturer, of 0.50–0.60 mm were used as aggregates for creating the mortar reference material. Fig. 3 shows the distribution of the size of the beads, which was measured using laser diffraction assuming an index of refraction of 1.52.

The SRM 2492 is prepared by the operator using the procedure described in ASTM C1738 and it is designed to have stable rheological properties for 7 days. The mortar is prepared by adding the beads to the SRM 2492 in the proportions desired. In this paper, the volume fractions used were 0%, 20%, 30%, and 40%.

3.2. Experimental set-up

The rheological measurements were performed using a coaxial rheometer that was shear rate (rotational speed) controlled. The speed ranges were (a) 0.12–5.2 rad/s with 10 points measured, and (b) 5.2–0.1 rad/s with 15 points measured. At each point the rotational speed was maintained for 20 s to ensure equilibrium. In the future other shear rate range should be investigated.

The coaxial rheometer was composed of a bob manufactured at NIST that rotates within a container of 43 mm in diameter. The bob is shown in Fig. 4 [14]. The gap between the bob and the container