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The rheology of cementitious suspensions: A closer look at experimental parameters and property determination using common rheological models

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ABSTRACT

This paper investigates the influence of gap between parallel plates, surface texture of the bottom plate, and mixing intensity on the yield stress and plastic viscosity of cementitious suspensions extracted using the Bingham model. Special emphasis is paid toward understanding the effects of shear rate range and different rheological models on the flow parameters. It is shown that the use of a wider shear rate range (0.1–100/s), can be beneficial in obtaining a reasonable portion of the stress plateau in the shear stress–shear rate relationship, which facilitates a model-less, yet accurate extraction of yield stress. The Bingham model that considers only the linear region (i.e. ~5–100/s) overestimates the yield stress as indicated by the stress asymptote while the Herschel–Bulkley (H–B) equation applied in the 0.1–100/s shear rate range underestimates the yield stress. Further lowering the evaluated shear rate range (i.e. 0.005–100/s) does substantially improve the H–B prediction of yield stress.

1. Introduction and background

Rheological studies of concentrated suspensions of solid particles in a continuous liquid medium are commonly used to assess the characteristics of materials in industries ranging from food to pharmaceuticals to construction materials. The flow behavior of these concentrated suspensions is influenced by surface contacts between solid particles and interparticle forces such as van Der Waals and steric forces [1]. Rheological studies of cementitious suspensions provide an understanding of how these materials behave in the fresh state and serve to monitor structure development that dictates the development of the mechanical properties [2]. However, in order to apply rheological experiments to cementitious suspensions, it is important to clearly understand the influences of experimental parameters and the selected rheological model on the measured and predicted characteristics of flow to establish their relevance and applicability.

Rheological experiments are typically carried out using a rotational rheometer, which monitors the change in torque required to change the shear rate (constant strain) or the change in strain required to change the torque (constant stress). There exist several experimental parameters of significance, including but not limited to: testing geometry (parallel plate, coaxial cylinder, cup, vane, etc.), the gap between shearing surfaces, roughness of the shearing surfaces, testing temperature, and the state of dispersion determined by the particle characteristics and the mixing method. Bingham, Herschel–Bulkley, and/or Casson models [3–6] are commonly applied to the shear stress–shear rate response to extract the rheological parameters (mainly yield stress and plastic viscosity) that describe flow.

Previous studies [3,7,8] have reported the influence of the experimental setup on the yield stress and plastic viscosity of cementitious suspensions extracted using a Bingham model. The plastic viscosity is a measure of the rate of increase in shear stress with increasing strain, and is thus a measure of the flowability of a fluid. The plastic viscosity of fluid suspensions is thought to be primarily influenced by interparticle friction and surface contacts [9], wherein decreasing the interparticle (friction) forces by increasing particle spacing (or by decreasing surface contacts) results in a decrease in plastic viscosity. The yield stress is a more complex parameter, defined as the non-zero (finite) stress at a “zero” strain rate.[1] Several methods

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[1] It should be noted that a “zero” strain rate does not practically exist. A non-zero strain rate (even if infinitesimally small) needs to be applied in order to obtain a resistance to flow (shear stress). The apparent “zero” strain rate is thus a mathematical simplification through which a value for the yield stress is obtained.

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have been proposed to determine the yield stress, including: an extended duration constant stress experiment [10], stress growth and strain reduction experiments [11], and oscillatory experiments [12]. Yield stress is typically determined in cementitious suspensions using strain reduction experiments with reverse extrapolation to a zero strain rate using a rheological model fit to the measured shear strain rate–shear stress dataset [9].

Yield stress has been attributed to the effects of both the surface contacts between particles which prevent flow (jamming) below a certain applied stress, as well as interparticle attractive forces [1,13,14]. Yield stress is commonly reported as the stress required to initiate flow of a fluid [9]; however some authors distinguish between a dynamic yield stress and static yield stress [15]. The dynamic yield stress is the stress required to maintain flow once it has commenced, while the static yield stress is the stress required to initiate flow from rest. This indicates that the dynamic yield stress is a model-dependent parameter, i.e. the choice of model can have a significant influence on the calculated yield stress, as can be seen from the data presented in [3]. This distinction is significant when comparative rheological studies of cementitious suspensions are to be performed, as selection of different shear rate ranges or models will result in substantial variations in the calculated yield stress, indicating that the dynamic yield stress is not a material parameter in the truest sense. This paper explores this idea in some detail in the context of cementitious suspensions. Furthermore, an understanding of the influence of the shear history of the suspension, as represented using different mixing procedures is developed in addition to new evaluations of the influences of experimental parameters including: the gap between shearing surfaces and the surface roughness of the bottom plate on the measured rheological response.

2. Experimental program

2.1. Materials

The materials used in this study are a commercially available Type I/II ordinary portland cement (OPC) conforming to ASTM C 150 [16], Class F fly ash conforming to ASTM C 618 [17], and limestone powder of 3 μm median particle size, conforming to ASTM C 568 [18]. The particle size distributions of these materials are presented in Fig. 1 and their compositions in Table 1.

For all the cementitious suspensions considered, cement was replaced by either limestone or fly ash on a volumetric basis to ensure that the comparisons are consistent. The suspensions were prepared at a constant volumetric water-to-solid ratios, $(w/s)_v$, of 1.42, equivalent to mass-based water-to-solid ratio, $(w/s)_m$, of approximately 0.45. No chemical admixtures were used.

2.2. Experimental parameters and suspensions

The rheological response of the suspensions is considered in the context of four distinct parameters: (i) gap between the top and bottom plates in a parallel plate configuration (top plate diameter of 50 mm, serrated to a depth of 1.0 mm), (ii) roughness and surface treatment of the bottom plate[2] (serrated to a depth of 0.15 mm, or resin-coated sandpaper[3] of mean surface roughness, MSR = 0.12 mm or 0.017 mm), (iii) type and speed of mixing of the suspension, and (iv) range of shear rates considered. Further details regarding the parameters are provided in Table 2. The suspensions consisted of: (i) OPC + water (referred to as OPC) and (ii) OPC + limestone + water (referred to as LS) where the fine limestone ($d_{50} \approx 3$ μm) replaced 10% of OPC by volume, and (iii) OPC + fly ash + water (referred to as FA) where fly ash replaced 10% OPC by volume. A gap of 2.0 mm, a bottom plate with 0.15 mm deep serrations, a shear rate of 5-to-100/s, and high-shear mixing corresponding to ASTM C 1738 [20] were used as the general "default" evaluation parameters. Three replicate samples were produced and tested for each mixture and experimental condition.

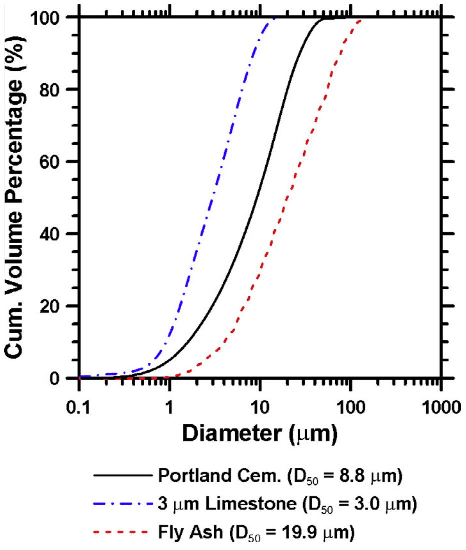

Fig. 1. The particle size distributions of OPC, 3 μm limestone, and fly ash, as measured using a light scattering analyzer.

2.3. Mixing and testing procedure

All powders were dry blended prior to the addition of water. To investigate the effects of mixing on the rheological response, four different mixing procedures were used – three involving a high shear mixer and the fourth using a hand-held kitchen mixer. All the mixing procedures consisted of an initial powder addition phase, followed by initial mixing, a covered rest period, and final mixing. Table 3 illustrates the four different mixing procedures along with the mixing speeds, time, and rest durations. For all the three high shear mixing procedures, the powder blending speeds and the rest durations are the same. The differences lie in the initial mixing speed after adding water, and in the final mixing speed and its duration. The mixing condition of the highest intensity, both with respect to speed and duration is the one similar to ASTM C1738 (but differing in the initial speed and rest period), and is described as 12-30-12-90. The first and third numbers represent the initial mixing speed after powder blending and the final mixing speed after the rest period, in 1000× rpm (i.e. the number 12 in first and third positions in the above sequence indicates 12,000 rpm), and the second and fourth numbers represent the duration (in seconds) of initial and final mixing steps respectively.

In addition to the effects of mixing procedure, gap, and surface condition, the influence of the selected shear rate range on the rheological properties was also investigated. These experiments were of three different types: (i) a "normal" shear rate range between 5 and 100/s, typical of the range used in typical rheological studies of cement pastes [3,7], (ii) a "low" shear rate range, between 0.1 and 10/s, and (iii) a "wide" shear rate range, from 0.1 to 100/s, which encompasses both prior ranges. All rheological sequences consisted of a ramp-up pre-shear phase lasting approximately 80 s to homogenize the paste, an instantaneous ramp-down

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[2] The influence of the surface condition of the Peltier plate on rheological measurements including the effects of slippage and plug flow have been reported elsewhere [3,19].

[3] Care should be taken to ensure that the sandpaper is non-absorbent as otherwise it will result in changes in water availability and thus the rheological parameters.