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Flow onset of fresh mortars in rheometers: Contribution of paste deflocculation and sand particle migration

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ABSTRACT

Shear-induced particle migration is widely recognized to be a challenge in characterizing the rheological properties of fresh cement-based mortars. In this study, we aim to quantify shear-induced particle migration by characterizing the stress decay process during constant shear flow with the aid of a modified thixotropy/migration model. It is found that a conventionally used single exponential model is not sufficient to fit the stress decay and describe the destructuration and sand migration of mortar under shear. Instead, a two exponential model is needed to capture the interaction of sand particles and the suspending cement paste phase. Model parameters are used to quantify the effect of sand volume fraction, clay addition, and applied shear rate on the kinetics and intensity of colloidal deflocculation and sand migration. Results provide evidence that the colloidal and granular contributions to the overall stress decay of mortars can be represented by each of the two exponentials.

1. Introduction and background

Most studies on the rheology of cement-based materials thus far have focused on the paste phase and the concrete phase [1]. Paste is investigated because it is the phase that embodies the colloidal and hydration effects. However, the rheology of concrete is heavily influenced by the characteristics of the granular phase. Many experimental rheological studies investigate concrete systems directly to capture the critical aspects of particle packing and grain-to-grain contact. Sophisticated measurements are challenging, though, due to the limited sensitivity of large-scale viscometers and increased likeliness of inhomogeneity within the suspension, although some modified geometries have been proposed [1–3]. Mortar is an intermediate scale that exhibits both colloidal and granular behavior. They can be prepared in relatively small batches, and tested on rotational rheometers with precise shear and measurement control, allowing for more complex flow situations. Serving as the suspending phase for the coarse aggregates, the rheological properties of the mortar are important in regards to the stability of fresh concrete systems [4]. Particularly for self-consolidating concrete (SCC), the mortar phase makes up a greater part of its composition compared to conventional concrete. Thus testing the rheological properties of mortar is an integral part of SCC design [5].

However, a widely recognized challenge in characterizing fresh cement mortar through shear rheological methods is shear-induced particle migration [6–8]. This has invoked studies that explore the influence of various parameters, e.g. setup geometries and solid inclusions, on sedimentation and migration under rotational shear [9]. However, more investigation is needed. In particular, it would be useful to develop a method to quantify sand particle migration in fresh mortars to help guide the design of protocols for dynamic rheological characterization. Further, it can help determine the range of shear rates within which migration can be minimized or held constant. It can also be used to evaluate the effect of various mix parameters, e.g. mix proportioning and use of mineral/chemical admixtures, on dynamic segregation. As part of a wider investigation on the thixotropy of fresh mortar systems, in the present paper we discuss the potential of a thixotropy/migration model to quantify shear-induced particle migration in fresh mortars.

The idealized shear stress response of a fresh cement-based suspension under a constant intermediate shear rate can be described as follows: an initial increase to a peak value, considered to be the static yield stress as measured by the stress growth protocol, followed by a decay until steady-state is reached. The shear stress decay from the peak value to the equilibrium value captures the process of structural breakdown and is related to the thixotropy of the material. Thixotropy is defined as a decrease in viscosity under shear, followed by an increase upon removal of shear. From a microstructural point of view, it can be described as paste deflocculation and breakage of CSH bridges under flow, and reflocculation and formation of CSH bridges over time at rest [10]. Meanwhile, sand migration induces structural heterogeneity. In investigating stress decay, we focus on paste deflocculation and sand migration.

The stress decay curve of cement paste is found to be fitted well by an exponential curve. Empirically, Tattersall [11], Papo [12] and Lapasin et al. [13] measured the difference between the maximum shear stress, τ_{max}, needed to initiate flow and the steady-state equilibrium value, τ_e, at constant shear rate, then proposed simple thixotropy models that

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predict an exponential decrease of the shear stress. A logarithmic evolution of the shear stress was obtained, as follows:

$$\tau = \tau_e + (\tau_{max} - \tau_e) \exp(-Bt) \quad (1)$$

where B is a constant depending on the shear rate. The characteristic time of destructuration is $t_c = 1/B$, i.e. when $\exp(-Bt) = e^{-1}$.

Several thixotropic models have been proposed to quantify the structural breakdown process of cementitious materials. Roussel has proposed models for cement paste [14] and concrete [15]. For fresh mortar under shear, the measured decay can be expected to be a function of both deflocculation of the colloidal suspension and sand particle migration. Concerning the latter, in a rotational shear setup consisting of a concentrically oriented vane rotor and a cylindrical container, as the rotor introduces shear to a fresh mortar suspension sand particles will tend to migrate from the inner rotor to the wall of the cylinder. A void area with a smaller volume fraction will form in the suspension near the inner rotor, thus making the apparent viscosity and shear stress decrease. So modelling the shear stress decay provides a promising way to quantify the sand migration process.

Shear-induced particle migration has been explored in other system types, and the influence of different parameters has been measured by direct methods. It has been observed through magnetic resonance imaging (MRI) that shear-induced particle migration of beads in yield stress fluids is not apparent when the particle volume fraction is below 20%, while it becomes more apparent at higher particle volume fractions [6].

Ovarlez et al. investigated the effect of shear rate on noncolloidal rigid particles suspended in a Newtonian fluid [16]. They found very similar concentration profiles in a 58% suspension under constant rotational velocities ranging from 0.06 to 25 rpm with preshears of 9-rpm and 100-rpm. At all rotational velocities with each preshear, the concentration was found to be lower near the inner cylinder where the shear rate is highest. This may be attributed to the concentration profile that is irreversibly established by the preshear. Ovarlez et al. [6] also found that the ratio of normal stress difference from the inner rotor to the wall, which is the force of shear-induced particle migration, is proportional to shear stress but not dependent on the shear rate.

In addition to particle volume fraction and shear rate, it is expected that mix constituents, namely the presence of chemical and mineral admixtures, will change the rheological properties of the suspending paste system and thereby change the kinetics of shear-induced sand particle migration.

In this study, we propose a two exponential model to fit the torque/stress decay of fresh cement mortar systems subjected to constant angular velocity in a rotational rheometer. Results provide evidence that the colloidal and granular contributions to the overall destructuration and sand migration of mortars can be represented by each of the two exponentials. Model parameters are used to quantify the effect of sand volume fraction, clay addition, and applied shear rate on the kinetics and intensity of thixotropic deflocculation and particle migration. We find the influence of sand volume fraction and rotational velocity on shear-induced particle migration, as described by the model parameters, agree well with the findings of other studies as presented in the literature review above.

2. Experimental methods and materials

2.1. Materials

All mixes are prepared with tap water and Type I Portland cement. According to ASTM C150 [17], its compressive strength at 28 days is 44.8 MPa, the Blaine fineness is 420 m^2/kg and the chemical constituents are summarized in Table 1. The sand used in this study is silica-quartz. It is oven-dried for 24 h and sieved between sieve #16 and #30, yielding diameters between 0.6 and 1.18 mm.

Table 1
Cement chemical constituents.

Constituents	% by mass
SiO_2	19.22
Al_2O_3	4.98
Fe_2O_3	3.42
CaO	62.42
MgO	3.87
SO_3	2.72

Highly purified attapulgite clay, or magnesium aluminosilicate, is also used. It is a commercially available clay that is chemically exfoliated from bulk attapulgite to remove all impurities. When dispersed, it is needle-like with an average length of 1.75 μm and diameter of 3 nm, which gives rise to a high aspect ratio and high specific surface area. Given its nanoscale dimension, it will be referred to as nanoclay herein.

2.2. Mix proportion

All mortar mixes have a water-to-cement (w/c) ratio of 0.5 by mass. Water absorption of the sand is considered when proportioning to achieve the desired w/c ratio.

To explore the effect of sand volume, we test mortars with sand-to-cement (s/c) ratios of 1.5, 1.75, 2, and 2.25, yielding sand volume fractions of 41%, 45%, 48%, and 51%, respectively. In this case, nanoclay addition is held constant at 0.5% by mass of cement, which is found to be sufficient to achieve stable mixes that exhibit no visible signs of static bleeding or sand sedimentation. To explore the effect of nanoclay, we test mortars with nanoclay additions of 0, 0.25 and 0.5% by mass of cement. In this case, s/c ratio is held constant at 2 by mass. Finally, to explore the effect of angular velocity all mixes have s/c ratio of 2 and 0.5% nanoclay addition.

2.3. Mortar preparation

In this study, steps are taken to ensure that the cement paste phase is mixed at the same state for various s/c ratios. First, only the fresh cement paste phase is prepared in a medium upright planetary mixer. Then, to ensure that the sand is mixed evenly in the cement paste, sand is poured into the cement paste and hand-mixed randomly at high intensity. The details of the protocol are given here.

Nanoclay powder is blended with the mixing water in a Waring blender for 2 min to produce a nanoclay suspension, which remains stable for at least 6 h. The clay suspension and remaining mix water to reach the target w/c ratio is poured into the mixing bowl. Cement powder is slowly poured into the wet ingredients and mixed at a speed of 136 rpm for 1 min, then at a higher speed of 281 rpm for an additional 4 min. We occasionally scrape the bowl to ensure the cement paste is mixed evenly. Then, sand is slowly poured into the cement paste and hand-mixed in a random manner for 4 min. Finally the mortar is poured into the construction cell of the rheometer for testing.

2.4. Rheometer and construction cell

The rheometer in this study is a HAAKE MARS III rheometer. A drawing of the setup is shown in Fig. 1. The construction cell is a 74 mm diameter and 150 mm height cylinder, with 24 profiles of 2 mm evenly distributed at the wall to prevent wall slip. The rotor is a two-bladed vane with a diameter of 52 mm and height of 50 mm. The coaxial gap between rotor and construction cell is 11 mm, which is much larger than the size of sand. For all the tests in this study, the vane rotor is placed at the height where the gap between the bottom of the rotor and bottom of the construction cell is 20 mm.