Investigating Shear-Induced Particle Migration in Fresh Cement Mortars

Ye Qian, Shiho Kawashima

Civil Engineering and Engineering Mechanics, Columbia University, New York, New York, USA

Abstract: Shear-induced particle migration is widely recognized to be a challenge in characterizing the shear rheological properties of fresh cement-based mortar systems. In this study, we aim to quantify particle migration by characterizing the de-structuration process during steady-state flow with the aid of a thixotropic model. The interaction of sand particles and the cement paste is explored through varying the sand volume and resting time.

Keywords: Cement mortar; Sand; Rheology; Thixotropy

Introduction and Background

Most studies on the rheology of cement-based materials thus far have focused on the paste phase and the concrete phase. 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. Mortar is an intermediate scale that exhibits both colloidal and granular behaviour. They can be prepared in relatively small batches, like pastes, and tested on rotational rheometers with precise shear and measurement control, allowing for more complex flow situations to be simulated.

However, a widely recognized challenge in characterizing fresh cement mortar through shear rheological methods is shear-induced particle migration [1-3]. Therefore it would be useful to develop a method to quantify sand particle migration in fresh mortars. This can help determine the range of shear rates within which
migration can be minimized, to guide the design of protocols for dynamic rheological characterization. It can also be used to evaluate the effect of mix proportioning, use of mineral/chemical admixtures, etc. on dynamic segregation. As part of a wider investigation on the thixotropy of fresh mortar systems, in the present manuscript we discuss the potential of a de-structuration model to quantify shear-induced particle migration in fresh mortars. To do so, we look at the effect of different sand-to-cement ratios and resting time on model parameters.

**Experimental Methods and Materials**

**Materials**

Type I Portland cement, tap water and silica-quartz sand are used in all mixes. The sand is oven-dried for 24 hours and sieved between sieves #16 and #30, yielding diameters between 0.6 mm and 1.18 mm. Water absorption is measured to be 3.10% according to ASTM C218-15. To mitigate static segregation, highly purified attapulgite clay is added to all mixes.

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. 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. Attapulgite clays are added at 0.5% by mass of cement to all mixes, which is found to be sufficient to achieve stable mixes that exhibit no visible signs of bleeding or sand sedimentation over 20 min.

**Methods**

All rheological tests are performed on a rotational rheometer in a construction cell. The dimensions and geometry of the cell and rotor are shown in Figure 1. For all tests in this study, the gap between the bottom of the rotor and bottom of the cell is set at 20 mm. In this paper, all values will be reported as angular velocity (rad/s) and torque (mNm) of the rotor.

For rheological characterization of thixotropic materials like cement-based materials, it is necessary to achieve a reference state between samples/test runs. Since the aim of the study is to quantify shear-induced particle migration, we were unable to take the standard approach of applying a pre-shear with the rheometer. Instead we adopt an alternative reference state based on the work of Mahaut et. al. [4], which is to introduce random mixing by hand for 60 sec after the fresh mortar is loaded into the cell. Once the mix is manually agitated the rotor is set and the sample is subjected to a constant rotational rate for 60 sec. From there the torque decay of each mix is recorded at a data acquisition rate of 4 data points per second.
Results and Discussion

De-structuration model for mortar

A number of thixotropy models exist, reviewed by Roussel [5], to describe flocculation and deflocculation behavior of cement-based materials. Although described in detail elsewhere, it will be briefly presented here.

The general form for existing models can be written as the following:

\[ \tau = (1 + \lambda)\tau_0 + k\dot{\gamma}^n \]  
\[ \frac{\partial \lambda}{\partial t} = \frac{1}{T\lambda^m} - \alpha\lambda\dot{\gamma} \]

where \( \lambda \) is the flocculation state of the material and \( T, m, \) and \( \alpha \) are thixotropy parameters. Two assumptions are applied: i) the steady-state flow can be described by the Bingham model and ii) the yield stress at rest increases linearly. It is reasonable to assume both for our cement mortars systems based on the results of other studies on concretes [5, 6] and mortars [7].
From there a simplified deflocculation model is found to be:

\[ \tau = (1 + \lambda \dot{\gamma}) \tau_0 + \mu_p \dot{\gamma} \]  \hspace{1cm} (3)

where \( \mu_p \) is plastic viscosity.

It has been found that shear stress decay under shear of pastes and concretes can be described by a single exponential [5, 6, 8]. However, we find that the destructuration of mortars cannot. Instead, it requires two exponentials, as shown in Figure 2. It is hypothesized that it has to do with the viscous and granular contributions in mortar systems that is not as present in paste or concrete systems. Thus it is proposed that the destructuration of mortars under shear be described by the following:

\[ \tau = \tau_0 + \tau_1 e^{-a_1 \gamma t} + \tau_2 e^{-a_2 \gamma t} \]  \hspace{1cm} (4)

where the colloidal and granular components are described by each exponential. We observe the effect of s/c ratio and resting time on the parameters and discuss in the following sections.

Figure 2. One versus two exponential model for capturing destructuration of fresh mortar (s/c = 2) under constant shear (10 s\(^{-1}\)).

Effect of s/c
We capture the torque decay of fresh mortars with s/c ratios ranging between 1.50 and 2.25 at a constant applied rotational rate of 10 s\(^{-1}\) over 60 seconds, then apply the two exponential model (Eqn. 2). The results of parameters \(\alpha_1\) and \(\alpha_2\), and \(\tau_0\), \(\tau_1\) and \(\tau_2\) are presented in Figures 3 and 4, respectively. First, with increasing s/c, all \(\alpha\) and \(\tau\) parameters increase, indicating higher degree of destructuration overall. Second, comparing \(\alpha_1\) and \(\alpha_2\), \(\alpha_1\) is more than 20 times greater than \(\alpha_2\) throughout while the difference between \(\tau_1\) and \(\tau_2\) is much smaller. This indicates there are two distinct destructuration modes at different rates yet similar intensities. Third, it is apparent that there is greater variability for \(\alpha_1\), especially for s/c = 2.25. This variability can be expected as it describes the instantaneous decay upon introduction of shear, which highly depends on the initial flocculation state.

Focusing on rate of decay, we observe an increase in \(\alpha_1\) and \(\alpha_2\) with increase in s/c ratio. This can be attributed to both deflocculation of the paste phase and shear particle migration of the sand. Increase in sand volume increases the local shear rate applied on the paste phase. Therefore it is expected that it would experience a higher rate of deflocculation and a higher rate of decay. And in magnetic resonance imaging (MRI) results by Ovarlez [1], it was observed that degree of shear-induced particle migration increases with particle volume fraction.

It could be reasoned that \(\alpha_1\) and \(\alpha_2\) correspond to cement particle deflocculation and sand particle migration, respectively. First, the critical strain to break down the paste structure is much smaller than that of sand migration. So at constant shear rate, the characteristic time for paste deflocculation is much smaller than that of sand migration. This means the parameter corresponding to paste deflocculation can be expected to be much bigger, which is \(\alpha_1\). Secondly, with increasing s/c from 1.5 to 2.25 \(\alpha_2\) increases much more rapidly than \(\alpha_1\), indicating \(\alpha_2\) is more sensitive to sand volume. More investigation is needed to distinguish each of these contributions. To continue to explore this further, evolution over time and effect of stabilizer (e.g. clay addition, viscosity modifying agents) on these parameters will be tested.
Effect of resting time

To better understand the physical meaning of the parameters, we also look at the effect of aging on mortar systems with s/c = 2 subjected to a constant shear rate of
20 \text{s}^{-1}. Up to 1200 \text{s}, \alpha_1 \text{ and } \alpha_2 \text{ remains stable at } 0.18 \pm 0.0052 \text{ and } 0.0055 \pm 0.00014, respectively, with no apparent trend with time. On the other hand, parameters \( \tau_0, \tau_1, \text{ and } \tau_2 \) are all found to increase with time. Figure 5 presents the normalized values of these parameters up to 1200 \text{s}. We observe that \( \tau_1 \) increases linearly over time while \( \tau_2 \) is non-linear. Previous work has shown that pastes modified with the attapulgite clay at 0.3\% addition by mass of cement, similar to the paste phase in the mortar systems studied here, exhibit a linear increase in structural rebuilding energy (based on dynamic yield stress) over 60 min [9]. This provides support that \( \tau_1 \) is associated with the paste phase.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figure5.png}
\caption{Effect of resting time on parameters \( \tau_0, \tau_1, \text{ and } \tau_2 \).}
\end{figure}

Conclusions

This paper presents the preliminary results of an investigation on shear-induced particle migration in fresh cement mortar systems. It is found that a single exponential is not sufficient in describing its destructuration under shear. Instead, a two exponential model is needed. Examining the effect of s/c ratio and resting time provides evidence that each exponential can describe the colloidal and granular components of destructuration. Investigation is ongoing to elucidate the physical meaning of the parameters and to develop it as a means to quantify particle migration.

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References