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Distinguishing dynamic and static yield stress of fresh cement mortars through thixotropy

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ABSTRACT

The dynamic and static yield stress of fresh cement mortar were measured in a rotational rheometer with a vane geometry using shear rate and shear stress-controlled protocols, respectively. Through a shear rate-controlled steady-state protocol, the equilibrium flow curve is measured and fitted with the Bingham model to obtain dynamic yield stress. A negative slope in the equilibrium flow curve, shear banding and stick-slip phenomena are observed and discussed. Through a stress-controlled creep-recovery protocol, viscosity bifurcation behavior is captured and static yield stress is marked as the creep stress when the bifurcation occurs. Finally, the discrepancy between dynamic and static yield stress is tied to thixotropy.

1. Introduction

Yield stress has been extensively studied in non-Newtonian fluids, including colloidal gel, bentonite, polymer suspension, food, paints, waxy crude oil, pastes, and foams [1–5]. Specifically in cementitious materials, yield stress is important in quantifying flowability [6] and is correlated to conventional field-friendly measurement methods such as the slump and slump flow test, both experimentally and in simulation [7–10].

As a common method to obtain yield stress, an equilibrium flow curve is plotted as the equilibrium shear stress – shear rate relationship. Since the publication of the Bingham model [11], many models have been proposed to describe the equilibrium flow curve. One of the most common models for cementitious materials is the Bingham model [12–14], $\sigma = \sigma_y + \eta_{pl}\dot{\gamma}$, where σ is the shear stress, $\dot{\gamma}$ is the shear rate, σ_y is the Bingham yield stress and η_{pl} is the plastic viscosity. As a linear model, it is unable to capture shear thinning and shear thickening behaviors that can be observed in cementitious materials. Thus, the Herschel-Bulkley model is also commonly used, $\sigma = \sigma_y + k\dot{\gamma}^n$, where k and n are constants describing material properties, and $n < 1$ for shear thinning and $n > 1$ for shear thickening [15,16]. Studies by Yahia and Khayat [17] show that a modified Bingham model, $\sigma = \sigma_y + \eta_{pl}\dot{\gamma} + c\dot{\gamma}^2$, also provides a good description of non-linear flow behavior. With each of these models, a measure of yield stress can be obtained.

The yield stress of cement paste originates from the microstructure of particle-particle network through colloidal interaction or direct contact. The microstructure sustains a certain amount of stress before it is broken down and starts to flow, which defines the yield stress. At rest, the microstructure builds up due to colloidal flocculation and cement hydration bonding, such as CSH bridges [18], thus resulting in higher yield stress. Under shear flow, the structure and bonding network is broken down, resulting in lower yield stress. This property of fresh cementitious materials, i.e. before setting, where the microstructure and yield stress changes with flow history is termed thixotropy [19–22]. Roussel [18] assumed that the hydration products, even at early ages, contributes to the structuration of fresh cement paste. Early hydration products on the surface of cement particles bridge these particles together to form a rigid network, which exhibits a relatively small critical strain. In comparison, colloidal forces of cement powders contribute to a softer network with a relatively higher critical strain. These two networks both act as the origin of thixotropy of cement paste. They also play a big role in structuration and yield stress. Thus, yield stress and thixotropy are closely related [23,24].

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It could be reasoned that because of thixotropy there should be more than one state of flocculation or microstructure when yield stress is measured, depending on the flow history of the material. Since generally the equilibrium shear stress increases with shear rate, the yield stress measured through equilibrium flow curve is regarded as the shear stress when shear rate is 0, so the minimum stress to sustain or terminate the flow of the material. This is commonly termed dynamic yield stress, and so is the yield stress that is obtained through the aforementioned steady-state flow models. Meanwhile, there exists a yield stress corresponding to a state before the structure is broken down, so the stress necessary to initiate flow, corresponding to a well-connected undisturbed microstructure [23,25]. This is considered to be the static yield stress. One method to measure the static yield stress is to apply creep-recovery, which was implemented by Struble and Schultz [15] and later by the authors [26]. This is a stress-controlled test where the material will not flow until the applied creep stress is higher than its static yield stress. The static yield stress is expected to be higher than dynamic yield stress considering the corresponding structural states. The existence of separate dynamic and static yield stresses in the same material is known in cement-based systems, as well as bentonite [27], paints [28] and waxy crude and fuel oils [29].

Dynamic yield stress is commonly measured and studied because the equilibrium flow curve is a convenient, well-established method of shear rheological characterization. Measurements taken in field tests [30], such as the slump flow test, are also tied to dynamic yield stress [7,8,31]. Meanwhile, static yield stress is also very important and critical for concrete rheology and SCC studies. For example, formwork pressure [13,32–37], stability [38], distinct layers casting [39] of SCC are closed related to structural rebuilding, which is directly related to static yield stress. Static yield stress is also of great interest for 3D concrete printing applications [39,40].

In this study, flow behavior of mortar under constant shear rate and constant stress is studied. Mortar systems are selected as they exhibit both viscous and granular behavior, similar to concrete, but can be probed with high precision rotational rheometers. Through constant applied shear rate, the equilibrium flow curve and dynamic yield stress are obtained. Under constant applied stress, performed in the creep-recovery protocol, static yield stress is measured. The discrepancy between static and dynamic yield stress is related to thixotropy and it is explicitly demonstrated and discussed that the dependence of measured yield stress on preshear condition is due to thixotropy.

2. Material and procedures

The materials, rheometer, and mixing and testing protocols are described in detail in another study [41] and only briefly explained here.

All mortars are prepared with Type I Portland cement, oven-dried sand between 0.6 and 1.18 mm in diameter, highly purified attapulgite clay, i.e. nanoclay, and water are used. No chemical admixtures are used. The water-to-cement ratio (W/C) is 0.5 by mass. The sand-to-cement ratio (S/C) is set to 2:1 by mass, yielding a sand volume fraction of approximately 48%. The sand is river sand, sieved through ASTM #30 and #50 sieves, thus the diameter is between 0.6 and 1.18 mm. The water absorption rate is 1.55%, which is considered when proportioning to achieve the desired w/c ratio. Nanoclay was incorporated into the mixes because previous studies by the authors showed that it alleviates sand migration and enhances static stability of fresh mortars [41]. Nanoclay is added at 0.5% by mass of cement, which is found to be sufficient in achieving statically stable mixes with no visible signs of bleeding.

Nanoclay powder is first blended with water for 2 min to produce a suspension. Cement powder is slowly poured into the solution and mixed at a speed of 136 rpm for 1 min, then at a higher speed of 281 rpm for an additional 4 min. Then, sand is slowly poured into the cement paste and hand-mixed in a random manner for 4 min. Immediately after mixing, the fresh mortar is poured into the construction cell. Before each test, the mortar is strongly tampered by hand mixing in a random manner for 1 min to bring the material to a destructed, homogeneous state. More detailed discussion on the hand-mixing approach are presented in Refs. [25,41].

The rheometer is a HAAKE MARS III rheometer. The construction cell is a cylinder with the diameter of 74 mm and the height of 150 mm, with 24 profiles of 2 mm evenly distributed along the wall to prevent wall slip. The rotor is a two blade vane with a diameter of 52 mm and height of 50 mm. More details are provided in Ref. [41].

In this study, torque (mNm), angular velocity (rad/s) and angular deformation (rad) are used. Other studies have focused on processing raw data, such as torque and angular velocity to shear stress and shear rate, respectively. The Reiner-Riwlin equation is commonly used for this transformation [42], which assumes Bingham behavior for flow. Because of the non-linear flow behavior of cementitious materials, Feys et al. [43] extended the Reiner-Riwlin equation and used a modified Bingham model to transform raw coaxial cylinder data. Other methods such as Estelle et al. [44] also use a linear model to process the transformation. However, due to the complexities of the flows observed (i.e. shear banding, stick slip, shear localization), results are presented in torque, angular velocity, and angular deformation. This is believed to be sufficient for discussion of the presented work.

In all tests, at least three samples were tested and the average was taken to be the representative value.

3. Measuring dynamic yield stress through shear rate-controlled protocol

3.1. Equilibrium flow curve: using constant angular velocity protocol

Here, dynamic yield stress is obtained by fitting the equilibrium flow curve with a steady-state flow model. For each step, the fresh mortar is subjected to 1 min of strong hand mixing, then a constant angular velocity is applied for 60 s during which the corresponding torque decay is recorded. For each run, the torque development with time is as follows: the torque increases from 0 to a peak value and then decreases to an equilibrium value over time, as shown in Fig. 1. Constant angular velocity is applied between 1 and 50 rad/s – it is verified that 60 s is sufficient to reach torque equilibrium within this range. It is observed that the material response differs between higher velocities, i.e. 7–50 rad/s, and intermediate velocities, i.e. 1–7 rad/s.

From 7 to 50 rad/s, the equilibrium torque increases with increasing angular velocity. The resultant equilibrium flow curve can be obtained by plotting the equilibrium torque and corresponding applied angular velocity, and is presented in Fig. 2. It is depicted that from 7 rad/s to 50 rad/s the flow curve can be fitted with the Bingham model:

$$T = 0.5033 \cdot \Omega + 41.72 \tag{1}$$

where T is torque in mNm and Ω is angular velocity in rad/s. According to the fitting, with a R^2 of 0.9741, 41.72 mNm is the dynamic yield stress.

It must be noted that shear-induced particle migration is a critical issue in fresh mortar systems under flow and has long been