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On the measurement of evolution of structural build-up of cement paste with time by static yield stress test vs. small amplitude oscillatory shear test

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ABSTRACT

The structural build-up of cement-based materials is often characterized by the determination of the evolution of static yield stress with time. This property is crucial to many applications of concrete. However, the measurement of static yield stress may disturb the structure of cement-based material. As a consequence, the true structural build-up of the materials may not be detected. In this paper, the evolution of static yield stress and storage modulus (G') determined by small amplitude oscillatory test was determined to characterize the evolution of the structural build-up of cement pastes. Results showed that G' and static yield stress developed similarly with time. When the tests were carried out on the same sample, the measurement of static yield stress disturbed the structure of cement paste and had a significant effect on the following measurement of G' , but a slight effect on the following measurement of static yield stress.

1. Introduction

Fresh cement-based materials are thixotropic and non-Newtonian fluids. With the growing use of self-consolidating concrete (SCC), more attention has been paid to the thixotropy of cement-based materials. Thixotropy is generally defined as the continuous decrease of viscosity with time when a shear rate is applied to a sample that has been previously at rest, and the subsequent recovery of viscosity in time when the shear rate is discontinued [1]. One important characteristic of thixotropy is reversibility. Once cement is in contact with water, both reversible and irreversible effects happen simultaneously. These are quite difficult to distinguish from each other. In fact, structural build-up is a more precise term for cement-based materials than thixotropy at rest. The former one is the sum of reversible and irreversible effects, while thixotropy is defined as a reversible process. Roussel et al. [2] reported that the reversible effect may be dominating during the first hour since the static yield stress of the paste increases linearly with time during this period. This was verified by magnetic resonance imaging (MRI) velocimetry [3]. Thus, the terminology of thixotropy and structural build-up are often interchangeable at early age of fresh cement-based materials. Structural build-up will dominate over thixotropy at later age due to cement hydration.

Thixotropy/structural build-up of cement-based materials at early age is an important property that can influence the formwork pressure

exerted by concrete, stability of concrete after casting, and the formation of lifeline in multi-layer casting [4–11].

In order to follow the thixotropy/structural build-up of cement-based materials at early age, the sample should not be disturbed during the test, there are four approaches that can be used to measure thixotropy of materials, i.e. hysteresis loop, stepwise changes in shear rate or shear stress, dynamic moduli, and start-up or creep tests [1]. The first two methods are destructive when the material is a fresh cement paste, and thus not suitable for continuously monitoring of the evolution of structural build-up of a cement paste sample with time.

Small amplitude oscillatory shear (SAOS) testing can be used to monitor the structural build-up of cement paste at rest. During the SAOS test, a sample is subjected to a continuous sinusoidal excitation of either a given deformation or a given shear stress. The response (either shear stress or deformation, respectively) of the material to the excitation is then measured, and used to evaluate the structural build-up of the material. When the applied excitation is very low, and the material is still within the linear viscoelastic range, the test will not interfere with the structure of the material, and can then be considered as a nondestructive method. SAOS has been successfully applied to test cement pastes [12–16], providing a rheometer with very high precision is employed. On the other hand, such approach necessitating very small amplitude oscillatory displacement is not available for the testing of concrete materials.

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Static yield stress test, or start-up test and shear growth test, is most often used to monitor the structural build-up of cement-based materials, including concrete [4,9]. By running a series of static yield stress tests, the development of static yield stress with time can be used to characterize the evolution of the structural build-up of cement-based materials with time at early age. The static yield testing involves incrementally increasing stress or strain until a critical value is identified at which the sample starts to flow. Most often, static yield test is performed by shearing the sample at a very low and constant shear rate, and a peak value or a plateau of stress is obtained before the flow, is used to calculate static yield stress. The value of the constant and low strain rate involved in the static yield test varies in literature. For example, Mahaut et al. [5] conducted static yield stress tests of cement paste at a constant shear rate of 0.01 s^{-1} . Perrot et al. [17] measured static yield stress of cement paste at a shear rate of 0.001 s^{-1} for 180 s. Roussel et al. [2] used a constant shear rate of 0.005 s^{-1} for the measurement of static yield stress of cement pastes. Mahaut et al. [7] compared the static yield stress of a suspension with $140 \mu\text{m}$ polystyrene beads in an emulsion at shear rate of 0.01 and 0.003 s^{-1} , and found there is a 10% difference between yield stress measured at 0.01 and 0.003 s^{-1} .

The static yield test only measures the minimum shear stress at which the solid-like property of fresh cement paste diminishes, which reflects the initiation of flow of the material. Although the flow is very slow and concentrated in a very narrow area, the structure is still considered to be disturbed [7], and the evolution of solid-like properties of fresh cement paste might not be fully detected. Since concrete materials contain large aggregate, rheometers performing SAOS on conventional concrete are not available, and the static yield test seems to be a suitable way for the measurement of the thixotropy of concrete. Hence, it is necessary to understand to what extent the structure of cement-based materials is disturbed when performing static yield tests on the material.

In this paper, the structural build-up of cement paste was measured by both static yield stress test, which might be a slightly destructive test, and SAOS test, which is considered to be a non-destructive test. The testing was carried out on the same sample over a period of 120 min. In order to understand how the structure of cement paste is disturbed when using a series of static yield tests to follow the structural build-up of cement-based materials, the structural build-up measured by static yield stress test was compared to that by SAOS test.

2. Experimental programs

2.1. Materials and specimen preparation

Type I/II portland cement and a polycarboxylate-based high-range water-reducing admixture (HRWRA) were used. The HRWRA was designed to promote long retention of fluidity. According to ASTM C 191, the initial setting time of the cement paste with w/c of 0.42 was 250 min. Water and HRWRA (if any) were first added into the Hobart mixer before cement was introduced. The paste was mixed at low speed for 30 s, and then the mixer was stopped for 60 s to manually homogenize the paste sample. This was followed by mixing at high speed for 90 s and low speed for another 30 s. The volume of each batch was about 400 ml. Cement pastes with w/c of 0.3, 0.35, 0.42 and 0.45, and HRWRA addition levels of 0%, 0.1%, 0.2% and 0.3% (by the mass of cement) were used.

2.2. Testing procedures

A high precision rheometer, Anton Paar MCR 302, was used for the rheological test. During the test, the temperature of paste was maintained at 20°C . The rheometer has concentric cylinder geometry with the inner and outer diameters of 26.661 and 28.913 mm, respectively. Although unpublished measurements have confirmed similar results

with smooth and sandblasted geometries as long as the paste sample is not too stiff or too viscous, the sandblasted surfaces in contact with paste samples were used to reduce wall slip. The gap between the two cylinders is small, thus a constant shear rate could be assumed across the testing sample.

Two types of rheological tests were conducted: static yield stress test and SAOS test. Different testing regimes were used for various purposes, as described below.

(1) Static yield test

In order to study the effect of shear rates, the static yield stress test was conducted on cement paste at different shear rates of 0.001, 0.003, 0.005, 0.008, 0.01, and 0.02 s^{-1} . However, the rheometer software calculates the shear rate from the rotational velocity using a linear function. Due to the yield stress of the cement paste, the flow is expected to be localized near the inner cylinder, especially for the low rotational velocities, and thus low shear stresses, were employed. As the flow domain is restricted (the gap is not fully sheared), a calculation of the shear rate based on the rheometer software may not be correct. Furthermore, the exact width of the flow domain is also unknown, thus making the calculation of the real shear rate range impossible. In order to compare with the published literature, shear rates were still used in the test. However, the corresponding rotational velocities to each of the six imposed shear rates are: 0.00077, 0.00231, 0.00385, 0.00616, 0.0077, and 0.0154 rpm for 0.001, 0.003, 0.005, 0.008, 0.01, and 0.02 s^{-1} , respectively.

Since the static yield test is quite sensitive to the shear history, the mixing regime and resting time, a comparison made between shear rates by using different batches of sample could be overshadowed by the difference between the samples. This is illustrated in the next section. Hence, the comparison of shear rates was conducted on the same sample. When cement paste is sheared at different shear rates, different time periods may be needed to reach the peak value or plateau shear stress. To assure that the shear tests can reach peak value or plateau, the static yield stress test was conducted by shearing a paste sample at a relatively long shearing time, i.e. 80 s. Before conducting the static yield shear test protocol for each of the six shear rates, the paste sample was sheared at 100 s^{-1} for 60 s to secure a reference state. The static yield stress tests were consecutively conducted on the same sample at the six shear rates, as shown in Table 1. The tests were finished within 30 min to minimize the effect of hydration of cement. By doing this, the sample measured at various shear rates was assumed to be the same initial state.

(2) Continuous SAOS test

The amplitude sweeps were first used to determine the critical strain of cement paste, which is defined as the cutoff point between linear and nonlinear regions of strain sweep. It is found by various authors [12–16] that the critical strain is on the order of 10^{-4} . Thus, the strain was increased from 10^{-5} to 10^{-1} incrementally, while the frequency was kept constant at 1 Hz until the structure broke down, and the storage modulus (G') starts to decrease, as shown in Fig. 1. The critical strain of the investigated cement paste was found to be within 10^{-4} – 10^{-5} . Hence, SAOS, which is considered not to interfere with

Table 1
Testing regime of effect of shear rates on static yield stress.

Step	Test	Duration
1	Shear constantly at 100 s^{-1}	60 s
2	Shear constantly at 0.001 s^{-1}	80 s
Repeat step 1 and step 2 at the shear speed sequence of 0.003, 0.005, 0.008, 0.01, and 0.02 s^{-1}		

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