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Parallel-plate rotational rheometry of cement paste: Influence of the squeeze velocity during gap positioning

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ABSTRACT

This paper reports an experimental investigation regarding the influence of the squeeze velocity, during positioning of the parallel-plate gap, on the rotational shear flow behaviour of a cement paste. The descent of the upper plate was performed using diverse speeds while the normal force generated due to the compression of the paste was recorded. The slower the plate speed, the higher the resulting normal force. This behaviour was caused by liquid–solid separation, which is more likely to occur at slow squeeze velocities. Phase separation was confirmed by assessing, via microwave drying, the water contents of the trimmed portion of the paste sample and of the portion actually subjected to rotational shear cycles. Owing to the variation of water/cement ratio induced by liquid radial migration, paste's Bingham yield stress and plastic viscosity were significantly affected by squeeze speed, and both rheological parameters presented an inversely proportional relationship with this experimental variable.

1. Introduction

Concentric cylinders, cone-plate, and parallel-plate geometries are normally used either in rotational flow or in oscillatory modes for the rheological evaluation of concentrated suspensions. The first is used for relatively low viscous suspensions as cement slurries, whereas the parallel plate geometries are more appropriate for assessing the rheological behaviour of pastes [1–3].

Cone-plate setup has the advantage of shearing the material at a constant rate along the sample radius, while in the plate–plate geometry the shear rate varies (being zero at the centre and maximum at the edge) [1–4]. However, the former geometry also presents a main drawback for the evaluation of granular suspensions, which is the jamming of particles under the apex of the cone [1–6]. The truncated cone minimizes this effect allowing for its utilisation for suspensions [1–6], though only for a limited maximum gap, which also restricts the maximum particle size present in the suspension (due to the minimum required gap/particle size ratio of 10). On the other hand, the parallel-plate geometry does not have the jamming problem; the gap can be adjusted from tens of microns to a few millimetres according to the material [1–3] or to the goal of the experiment [4,5,7,8], and it provides a large shear rate range that is adjustable by changing gap and plate diameter [1–4].

Because of its suitable features, the parallel-plate geometry has been extensively used for the rheometry of cement pastes in flow [4,6–18] and small amplitude oscillatory [4,16–22] modes. Most of the studies aimed to evaluate the effects of composition and admixtures [9,13–18, 20–22]; while others focused on the influence of processing parameters like mixing method [4] and temperature [17]; understanding the setting kinetics and microstructural development [11,16,17,19–22]; or the relationship between rheology of cement paste and that of concrete [7,15,18]. The technique was also utilised for the development of a reference material intended for calibration of cement paste rheometers [12]. Roughness [8] and gap distance [4,7,8] were the test parameters systematically investigated to date. Rough plates tend to increase the measured Bingham yield stress compared to the values obtained with smooth plates; on the other hand, plastic viscosity decreases with plate roughness [8]. The use of smaller gaps resulted in higher flow resistances [4,7,8], especially when the gap approached the size of the particles/agglomerates of the paste, which was considered as a limiting gap related to a sharp increase of flow resistance [4,7].

Despite the extensive employment of the parallel-plate technique for cement paste rheometry, there is no mention whatsoever in the consulted references on cement pastes about the procedure used to adjust the gap prior to the rotational or oscillatory measurements. As general rules for most materials, the documents from rheometer manufacturers suggest the use of excess material and, then, trimming the squeezed-out portion at a gap 5% higher than the measuring position to ensure a correct filling and total contact between sample and plates [1,23]. In addition, it is recommended to attempt disturbing the sample as little as possible when loading, in order to maintain its original structure [1,23–25]. This mainly consists of descending the upper plate slowly to keep low shear rate and to avoid excessive

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increase of the normal force and possible modification/destruction of the sample structure during squeezing [1,25]. This suggestion is worthy particularly for structured fluids, gels, and highly viscoelastic materials. Nevertheless, this may not be the best choice for concentrated suspensions of particles in the micron range like cement pastes.

Actually, the squeeze-flow is a widely used rheometry technique, which has been applied for food, pharmaceuticals, polymer composites, ceramic pastes, and other concentrated suspensions [26–30]. Its use is becoming more frequent as an alternative/complementary method for cement-based pastes [16,31,32] and mortars [33–37], especially to simulate flow situations associated to geometric restrictions (extrusion, spreading, brick laying, flow through a nozzle during pumping or spraying). The geometry change inherent to the method can induce liquid–solid phase separation, because the liquid may flow radially outwards through the porous structure of packed particles (filtration or drainage), thus having a substantial influence on the squeeze-flow behaviour of concentrated suspensions [27–30,32–36]. The occurrence and intensity of phase separation depend on material characteristics (liquid viscosity and permeability of packed particles) and test parameters, mainly speed and gap [27–30,32–36].

As the oscillatory and flow parallel-plate rheometry of pastes must be preceded by a squeeze-flow, it is important to understand how this pre-test stage may affect these measurements, thus providing useful information to the development of experimental procedures more suitable for concentrated suspensions. For this reason, the objective of this work is to determine the influence of the squeeze velocity, during positioning of the parallel-plate gap, on the rotational shear flow behaviour of a cement paste.

2. Experimental

2.1. Cement paste

Cement pastes with water/cement ratio of 0.40 were prepared with Portland cement type CEM I 52.5 N (Lafarge Ciments — Usine Du Havre, France) specified according to EN 197-1. Specific gravity (by Helium picnometry), BET specific surface area (by Nitrogen adsorption) and particle size distribution (by laser diffraction in deionized water) of the cement are shown in Table 1.

2.2. Mixing procedure

The paste batches were prepared with 50 g of cement and 20 g of water both at room temperature of 23 °C. The materials were manually mixed with a small metallic laboratory spoon in a 150 mL plastic container for 180 s. The cement and 10 g of water were mixed for 30 s and, then, the whole paste for additional 150 s. Immediately after mixing, apparent density was measured using a 20 mL cylindrical container. The average values of fresh apparent density and air content were 1.89 g/cm^3 and 2.4%, respectively. Solid concentration of the paste was 44.6 vol.% without considering the entrained air.

2.3. Rheometer

The rheological measurements were performed using a shear rheometer AR2000 Ex (TA Instruments) with stainless steel parallel-plate geometry with 40 mm in diameter and crosshatched surfaces. The Peltier plate maintained the temperature of the lower plate at 25 °C during the tests.

Table 1
Physical characteristics of the Portland cement: ρ = specific gravity; SSA = BET specific surface area; D10, D50, and D90 = particle diameters associated to values of 10%, 50%, and 90% of the cumulative size distribution curve.

Cement	ρ (g/cm^3)	SSA (m^2/g)	D10 (μm)	D50 (μm)	D90 (μm)
CEM I 52.5 N	3.10	1.05	4.1	21.2	55.0

2.4. Sample loading

The samples were moulded immediately before the tests. A metallic ring with 40 mm diameter and 2.8 mm height was used for moulding the cement paste on the lower plate (Fig. 1a). The paste was placed with a spoon and the excess material removed during levelling the sample with a spatula; the ring was then removed. The upper plate was lowered until it gently touched the flat surface of the sample at a gap distance of ≈2800 μm (Fig. 1b). This preparation procedure ensured that initial geometry and volume of the samples were constant for all tests.

Gap positioning was performed in two steps:

- 2800–1050 μm: four constant squeezing speeds (50, 100, 250 and 750 μm/s) and the automatic exponential decay mode (average speed of 24 μm/s) were employed (Fig. 2). The automatic exponential mode was adopted because it is the default mode of the equipment and was used in previous publications [14,16,17,20,21]. The normal force was measured at the lower plate. At 1050 μm (Fig. 1c), the portion of the sample squeezed out from the plates was removed and placed in a small silicone container for the phase separation test.
- 1050–1000 μm: only the automatic exponential decay mode (average speed of 1.4 μm/s) was used, as the displacement was small and the gap had to be reached very precisely. At this stage (Fig. 1d) no further manipulation was done in the sample and, after coupling the solvent trap device on the lower plate, the system was ready to start the rotational test.

2.5. Shear cycles

The rheological evaluation consisted of two consecutive shear cycles with no rotational pre-shear step. The shear rate varied in ramp mode from 0 to 300 s^{-1} in 60 s and then back to 0 s^{-1} [5]. The total testing time was 240 s. Tests were performed at 5 and 35 min after the beginning of the mixing and the duration of the routine — from squeeze to the end of second cycle — was approximately 8 min.

For each squeeze speed used for the positioning of the gap, all tests were performed three times. A different mixing batch was used for each repetition.

2.6. Phase separation evaluation

In order to assess the occurrence of liquid — solid phase separation induced by squeezing the sample during loading, the water contents of the trimmed portion of the paste sample and of the portion actually subjected to the rotational shear cycles were determined. For this purpose, microwave drying was employed due to its successful utilisation on mortar samples in previous studies [35,36].

A reference portion was also used in every test for control; a similar amount of material (≈4 g) was taken from the bulk paste in the flask and placed directly in the silicone container for drying. The pastes were spread all over the container to increase surface area and avoid spalling when drying, which could invalidate the measurements due to loss of material. This problem was worse with the tested samples, as some of them presented drier and more rigid lumps than the other portions. All three portions — trimmed, tested sample, and reference — were weighed before and after microwave drying for 7 min at maximum power of the oven (Micro-ondes 23 L 800 W, MS23F301EFS — Samsung France). The period of 7 min was more than enough to reach constant mass of the samples. Additional time just heated the oven excessively without drying the samples any further.