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A new methodology for characterizing segregation of cement grouts during rheological tests

Quoc Gia Hoang, Abdelhak Kaci *, El-Hadj Kadri, Jean-Louis Gallias

Université de Cergy-Pontoise, L2MGC, EA 4114, F-95000 Cergy-Pontoise, France

HIGHLIGHTS

- A new methodology for characterizing the segregation of cement grouts under shearing is proposed.
- The segregated grout presents particular rheological behavior which is not observed in case of stable grout.
- The reliability of rheological testing of grouts can be verified.

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ABSTRACT

This work proposes a new methodology for characterizing the segregation of cement grouts under shearing during rheological tests. This methodology uses a rotational rheometer with coaxial-cylinder geometry (Couette type) and a specific protocol composed of two shearing cycles with various steps of shear rate and two values of bottom gap. Density measurement of the upper and the lower part of the grout's sheared volume in the rheometer were carried out at different characteristic times of the protocol and compared with rheological behavior of grout during shearing. The methodology was validated on (i) a stable grout with low water/cement ratio, (ii) an unstable grout with high water/cement ratio and high amount of superplasticizer. It has been shown that the segregation of the unstable grout, characterized by the density decreasing in the upper part of the sheared volume, leads to a characteristic rheological behavior depending on the bottom gap of the rheometer and the shear rate. This specific rheological behavior is not observed in the case of stable grout or when viscosity modifying agent was added for stabilizing unstable grout.

1. Introduction

Cementitious grouts are widely used in various applications in the field of civil engineering as for filling post-tensioning ducts, soil stabilization by injection, reinforcement of cracked structures [1–6]. The quality of grout is judged by its ability to remain homogeneous under the action of its own weight (static stability) or when its subjected to high shear rates during processing, particularly in the case of injection (dynamic stability). Admixtures are generally used to improve the resistance to separation between the liquid and solid phase of the grout [7–9].

Stability of a cementitious grout depends on several factors related to the properties of the various constituents, mix design and resulting rheological properties including viscosity and thixotropy [9–13]. Various empirical tests are recommended for estimating the degree of static segregation of cementitious materials. They are generally based on following up segregation of particles over time by measuring different properties (density, distribution of coarse particles...) at different heights [14,15]. Other techniques based on evaluation of sedimentation of particles were also used by various authors to highlight the influence of mix design parameters on the stability of cementitious materials. The Zeta potential measurements based on DLVO theory are commonly used in the case of diluted cementitious mix [16,17] but it's not suitable for concentrated cement grout [18]. Mikanovic and Jolicoeur [19] used electrical conductivity measurements and admixture adsorption to estimate the influence of superplasticizers on the rheological properties and dynamic stability of cement and limestone pastes. In their investigation, they observed that the curves obtained for cement pastes are somewhat more complex to analyze since the electrolytes, which dissolve upon cement hydration, change conductivity of the solution by time. Bouras et al. [8] were interested in the influence of sedimentation on the

* Corresponding author.
E-mail address: abdelhak.kaci@u-cergy.fr (A. Kaci).

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flow curves obtained for cement pastes at 10 and 45 min after mixing for two different volume issued from two heights levels. They concluded that for their cement grout mix there is no difference between the two results at given times. Perrot et al. [20] proposed a correlation between yield point and bleeding instead of sedimentation to estimate the stability of a suspension. Recently, Peng and Jacobsen [21] have used hydrostatic pressure measurements to assess the sedimentation and bleeding of different cement grouts by varying the mix design parameters such us W/C ratio, admixtures and fillers proportion.

For our knowledge, contrary to the static segregation, studies or methods used to characterize segregation induced when the grout is subject to high shear stress are very scarce. This dynamic segregation is especially important to take count in injection process or during rheological measurements by rheometer for example. Indeed, by application of high shear rate during rheological measurements, a relative movement of the particles might be generated which increase the risk of migration and segregation. Therefore, the obtained measurements may be incorrect and by consequence identified rheological parameters such as yield stress and plastic viscosity were not intrinsic. Such initially stable cement grouts can become unstable under the effect of shear [22,23].

Bhatty and Banfill [24] were the first to focus on the risk of segregation during rheological measurements using rotating viscometers. They studied the experimental conditions needed to minimize the errors through the measurement of variation of solid particles fraction taken at three different heights levels and on both sides of used cylinders. They also highlighted the role of chosen system (coaxial cylinders and helical geometries) and the effect of horizontal gap (between cylinders) on the risk of occurred segregation under application for constant value of shear rate. Conclusions of this study did not take into account the effect of the level of applied shear rate which can cause migration especially for high values. Wallevik [25] proposed a methodology to check if there is any segregation occurred at the end of the rheological measurement by adding a "segregation point" in the measurement protocol. This methodology allows controlling the quality of the rheological measurements. However, it does not assess the impacts of segregation on the rheological behavior of grout and the potential segregation of grout at high shear rates. Another approach has also been proposed by Tregger [26] which is based on the measurement of the radial distribution of the grains during spreading tests of self-compacting concrete. Among inline measurements used we can mention Magnetic Resonance Imager (MRI) to measure during transient and steady state flows, the local velocity field and thus calculate the local shear rate [23] or an interesting technique based on the Ultrasound Velocity Profiling with Pressure Difference measurements proposed by Wiklund et al. [27] for process monitoring and control of grouting applications under realistic field conditions.

In this study, we developed a specific protocol with several shear rates steps to characterize the segregation of cement grout under shearing and its influence on rheological behavior. This protocol is used to understand the evolution of grouts during testing and to identify and quantify the risk of segregation of the cement grouts during the rheometric test.

2. Materials and experimental methods

2.1. Materials

All grouts of the study are prepared with a Portland cement CEM I 52.5N CP2 (from Villiers-au-Bouin, France) according to the European Standard EN 197.1 [28] and the French standard NF P 15318 [29]. The Blaine specific surface area is 3790 cm^2/g and density is 3.15 g/cm^3. The initial setting time of cement is 150 min and the final setting time 240 min.

A new generation modified polycarboxylate is used as superplasticizer with 30% mass fraction of active ingredients, allowing efficient dispersion of cement particles and improving grout's flowability for extend duration. The viscosity modifying admixture is also used. It's composed of hydroxyl-propyl-methyl cellulose solid particles with surface treated for delaying solubility and improving dispersion in water.

2.2. Rheometer

The rheological behavior of cement grouts was characterized by rheometer equipped with coaxial cylinders (Couette type). The outer cylinder, with 2Re = 43.40 mm diameter and He = 95 mm height, is fixed and serves as grout container (Fig. 1a). The inner cylinder, with 2Ri = 38.02 mm diameter and Hi = 55 mm height, is rotating. Its lateral surface is grooved for reducing slipping. Grout shearing occurs into the radial distance between the two cylinders, ΔR = Re − Ri = 2.69 mm, called *lateral gap*. This gap can be considered narrow compared to the cylinders diameters because the radius ratio Re/Ri is equal 1.14 (it is preferable to have radius ratio less than 1.2 [24]) ensuring full shearing of the grout and allowing hypothesis of quite constant shear stress and shear rate for the sheared volume (of homogenous and stable grout). The determination of the shear stress as a function of shear rate from the measurements of the torque for different angular velocities is consequently simplified.

The volume of sheared grout into the lateral gap is 18.91 ml. We divided this volume in two half parts (upper and lower) for the measurements of the density of the grouts (Fig. 1b). The distance between the bottom of the rotating cylinder and bottom of the fixed cylinder, called here *bottom gap* and designated "g" in Fig. 1a, was set at and 10 mm for tests. The grout volume in the bottom gap is respectively 1.48 ml and 14.79 ml. The grout filling the bottom gap is not sheared during tests. In the case of stable and homogeneous grout, the height of the bottom gap has no influence on the rheological measurements. We will show below that this is not verified in the case of unstable, segregative grouts during testing.

2.3. Rheological protocol and density measurements

The mixing of grouts was carried out on 800 ml mixture with a blender revolving at 700 rpm during 7 min. We verified that this duration was enough to guaranty full homogenization of the mixture without any segregation at the end of the mixing for all studied grouts. The rheometer is filled in 90 s after mixing and a specific protocol was used to characterize the rheological behavior and the potential segregation of cement grouts during measurements.

The protocol has been developed after several trials. It is composed of two identical cycles of 9.5 min with various steps of shear rates between 0 and 500 s^{-1} (Fig. 2). The protocol begins with a high-speed pre-shearing, composed of a steady rise of shear rate from 0 to 500 s^{-1} in 30 s, followed by a constant shear rate at 500 s^{-1} for 120 s and a coming back to 0 in 30 s. After 30 s at rest, a new step of constant shear rate at 300 s^{-1}, reached directly, is performed for 50 s, followed by 35 steps with decreasing shear rate till 0.1 s^{-1}. For shear rate range between 300 s^{-1} and 10 s^{-1} the duration of every step is 5 s and for shear rate range between 6 s^{-1} and 0.1 s^{-1} it is 10 s, allowing enough time for the stabilization of the value of torque and obtaining reliable measurements. After 30 s at rest, the same cycle is repeated for a second time. The total duration of the protocol is 19.5 min. Thus, at the end of the measures, the age of grout from the beginning of the mixing is 28 min. We consider that for this duration the hydration effect on rheology could be neglected [30,31].

For the characterization of each grout, the previous protocol was applied systematically twice: one time with a 1 mm bottom gap (g1) and one time with 10 mm bottom gap (g10). Each application of the protocol requests the mixing of a new quantity of grout under the same experimental conditions.

The segregation of cement grout during shearing tests was also characterized directly by five measurements of the grout density at five characteristic times during the protocol for both bottom gap values, listed from 0 to 4 on Fig. 2 and Table 1.

For a density measurement, *n* (from 1 to 4), the protocol ran till the chosen time. The rheometer was stopped and the upper of the sheared volume of grout in the rheometer and then the lower part (equal to 9.5 ml each) (Fig. 1b) was collected with a calibrated syringe. After that, each part of grout was weighted by an ultra-high precision balance. Taking in account that the accuracy of the measure of the collected volume was ±0.1 ml and these of weigning was ±0.01. The accuracy of the density measures was ±1.5%. Note that each measurement of density needed a new mixing of grout and requested an independent test. Thus, the characterization of the segregation of a given grout for 5 moments and for both gap values requires 4 × 2 = 8 independent ones. Despite its heavy procedure, this characterization is very effective for monitoring and quantifying all the phenomena that take place during the rheometric characterization.

We designate D1 the density of the upper part and D2 the density of the lower part of the sheared volume of grout.