Cement and Concrete Research 71 (2015) 29–35

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: http://ees.elsevier.com/CEMCON/default.asp

Assessment of potential concrete and mortar rheometry artifacts using magnetic resonance imaging

H. Hafid [a,b], G. Ovarlez [b], F. Toussaint [c], P.H. Jezequel [c], N. Roussel [a,*]

[a] Université Paris Est, IFSTTAR, Paris, France
[b] Université Paris Est, Laboratoire Navier (CNRS-IFSTTAR-ENPC), Champs sur Marne, France
[c] Lafarge Centre de Recherche, Saint Quentin Fallavier, France

ARTICLE INFO

Article history:
Received 28 September 2014
Accepted 13 January 2015
Available online 31 January 2015

Keywords:
Fresh concrete (A)
Rheology (A)
Aggregate (D)
Mortar (E)
Yield stress

ABSTRACT

In this paper, we compare macroscopic measurements and local MRI measurements of the behavior of mixtures of natural and crushed sand particles in model yield stress fluids. In doing so, we seek to assess the potential consequences of artifacts on the measurements of the flow curves of mortars and concretes in rheometers. We conclude that, once plug flow is taken into account and corrected, shear-induced particle migration is the dominating artifact for these systems. This shear-induced particle migration occurs for very low strains and is at the origin of a steady state inhomogeneous particle concentration profile. This induced inhomogeneity shall be therefore always present in most concrete rheometers. We extrapolate that shear-induced particle migration could be at the origin of the existing discrepancy between commercially available concrete rheometers. From a measurement point of view, it leads to an underestimation of yield stress and apparent viscosity at low shear rates.

1. Introduction

Fresh concrete is a dense suspension, the rheology of which dictates the success of the casting or molding process. Concrete rheometry has gone through major changes in the last decades and measuring the rheological properties (yield stress and plastic viscosity) of this dense suspension has become a major objective of the cement and concrete scientific community. Advanced concrete rheometers were developed [1–5] and the correlation between industrial test and fundamental rheological parameters has been thoroughly studied [6]. Because of the fundamental role of yield stress during concrete casting processes [6], most existing studies dealing with fresh cementitious materials properties mainly focus on yield stress. This parameter can be assessed through concrete rheometers but also through empirical test such as slump or slump flow [7–9], L-box [10] or channel flow [11]. Within the nowadays frame of work, even if marsh cone [12] or V-funnel test results can be roughly correlated to viscosity, only rheometers give theoretically access to the value of plastic viscosity.

In parallel, it has been shown recently in the field of general rheology that artifacts are likely to be involved during measurements of viscosity on dense systems containing both fluid and particles [13–15]. For example, shear-induced migration [13,15] or shear-induced settling [14] of particles may occur within the gap. Moreover, one frequently observes the presence of a dead zone in the gap: shear is localized and flow is inhomogeneous (as in plug flow) [13,16,17]. These artifacts generally induce an apparent non-Newtonian behavior that is not representative of the actual behavior of the material.

The above artifacts, which strongly depend on the rheometer used, could be (collectively or individually) at the origin of the discrepancy between the values of the rheological parameters (both yield stress and plastic viscosity) measured using the various commercially available concrete rheometers [18,19]. This discrepancy has been the topic of many debates in the last two decades. It was shown in [18,19] that this discrepancy can reach up to a factor 4 between two rheometers measuring the properties of the same material although the degree of correlation of both yield stress and plastic viscosity measurements between any pair of rheometers is reasonably high [18,19].

Because of the existence of these artifacts, specific protocols were developed for concrete testing. They mostly consist in carrying out tests as short as possible to limit shear-induced migration and shear-induced settling while long enough to allow the steady state properties of this thixotropic material to be measured [20–22]. Moreover, measurements carried out at the beginning and at the end of the testing procedure are often compared to check if particle migration occurred in the sample throughout the testing procedure and if it has induced a deviation in the measured rheological properties. Finally, a large proportion of analyses of concrete rheometer raw data includes a shear localization correction allowing for a decrease of the consequences of this artifact on the measured rheological behavior.

In this paper, we choose to go deeper in the study of these artifacts by getting access to the local and real rheological constitutive law of the studied material: we therefore measure local material velocities [13,16,17] and local concentrations of particles [13] through magnetic

* Corresponding author. Tel.: +33 180668605.
E-mail address: nicolas.roussel@ifsttar.fr (N. Roussel).

http://dx.doi.org/10.1016/j.cemconres.2015.01.009
0008-8846/

resonance imaging (MRI) of the flowing material. We compare macroscopic rheometer measurements and local MRI measurements of the behavior of mixtures of rounded and crushed sand particles in laboratory yield stress fluids. In doing so, we seek to assess the potential consequences of artifacts on the measurements of the flow curve of cementitious materials containing inclusions far larger than cement grains.

In the first part of this paper, we present the studied materials and experimental protocols. In the second part, we discuss migration and sedimentation of particles along with plug flow measured with MRI. Finally, we compare the flow parameters obtained from macroscopic measurements with the real local values obtained from MRI. We conclude that, once plug flow is taken into account and corrected, particle shear-induced migration seems to be the dominating artifact in our measurement. We extrapolate that it should also be the one most strongly effecting the measurements of mortar or concrete rheological parameters in concrete rheometers.

2. Materials and procedures

We study here the rheological behavior of mixtures of real sand particles suspended in model yield stress fluids. The behavior of this type of materials was investigated in detail in [23], and it was shown that suspensions of aggregates in cement pastes behave like suspensions of particles in any other yield stress fluid [24] from a purely rheological point of view. That is why we choose here to suspend our particles in water-in-oil emulsions, which have been shown to be simple yield stress fluids [25]: this allows us to avoid all the experimental and analytical complexity linked to nonreversible and reversible changes in cement paste rheology while allowing for a proper measurement of the variations of the rheological properties of model mortars as a function of the sand particle volume fraction [24]. It moreover greatly simplifies all MRI measurements as, compared to cement, these systems do not contain Fe paramagnetic oxides. These enhance relaxation kinetic and lead therefore to poor signal to noise ratio.

The emulsion yield stress was varied between 10 and 20 Pa. Three real sands were studied. The first one was natural rounded sand (S_1). The two other sands S_2 and S_3 were crushed sands. The sands differed by their shape as shown in Fig. 1. From a quantitative point of view, their convexities (*i.e.* defined as the average ratio between the perimeter of the grain projection and the Convex Hull perimeter) were respectively equal to 0.95, 0.91 and 0.85. Three sand grain fractions were studied (160–200 μm, 315–400 μm and 800–1000 μm). The maximum packing fractions ϕ_m of the dry sands were measured after compaction by vibration using the measurement protocol described in [26] and were respectively of 65%, 62% and 60% for sands S_1, S_2 and S_3 no matter the grain fraction.

The experiments were performed within a wide gap Couette type rheometer coupled with MRI [13,16,17,25]. MRI was used to measure the local velocity and concentration profiles of the material inside the gap of the geometry, while a Bohlin® C-VOR 200 rheometer was used to measure in parallel the torque for the exact same protocol, geometry and flow history. The inner cylinder radius R_i was 4.15 cm while the radius of the outer cylinder R_e was 6 cm. The resulting gap was therefore 1.85 cm. The two cylinders were covered with sandpaper to prevent any slippage (200 μm grade). The experimental protocol was designed to stay close to standard protocols used in fresh concrete rheology [20]; these experiments are usually short, which are supposed to help avoid shear-induced or gravity-induced inhomogeneities [27]. An increasing rotational velocity ramp from 4 to 100 rpm (each step lasting for 20 s) was therefore followed by a decreasing rotational velocity ramp from 100 to 5 rpm (*Cf.* Fig. 2). A local velocity profile within the gap was measured using MRI [16,17,25] for each rotational velocity. Moreover, concentration profiles along the radial and vertical direction were measured before and after each rotational velocity.

3. Experimental results

3.1. Artifact N°1: migration

For all mixtures with particle volume fraction higher than 20%, the volume fraction profiles measured after shear were found to be inhomogeneous. The volume fraction increases with the distance from the rotational axis (see Fig. 3): the rotation of the inner cylinder causes migration of the particles from the regions of high shear (near the inner cylinder) towards the zones of low shear (near the outer cylinder).

It is often considered in cementitious material literature that this migration is a slow phenomenon and that it is therefore possible to measure the behavior of the system before any migration of the particles effect the measured data. This common belief comes from the literature of migration in Newtonian suspensions (see [13] for a review), where it was indeed observed that it is usually a slow-diffusive-phenomenon. The typical strain scale for migration to occur is expected to be rate independent and to scale as $\propto \text{gap}^2/a^2$ where a is the particle size, *i.e.* the use of large gap to particle size ratio would likely prevent migration.

From a review of the literature [13], this would *e.g.* lead to an expected strain of order 5000 for migration to occur in our geometry for 200 μm particles in a Newtonian fluid.

However, it has been recently shown that, depending on the flow characteristics, at high particle volume fraction, migration may be very rapid and thus unavoidable. The strain to induce this migration could be smaller than 100 for the particles studied here in our geometry. This is more than 500 times faster than expected [13]. This new behavior was attributed to the emergence of significant direct interactions between the particles in a regime where grain inertia plays a major role [13].

Consistently with these last observations, in our experiments, we have observed (i) that there is no observable migration in dilute suspensions, and (ii) that migration occurs almost instantaneously when we shear our dense suspensions: at the end of the first ramp in Fig. 3, the material is already inhomogeneous, showing that migration occurs in less that 100 strain unities. This means that this phenomenon can basically not be avoided when performing rheological measurements on such system, and that it may thus occur in most fresh concrete rheology studies.

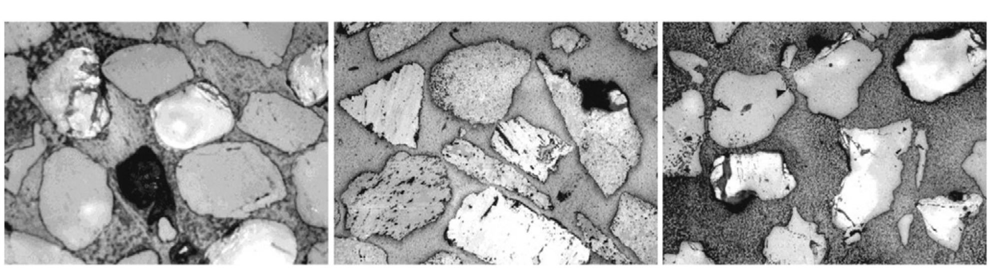

Fig. 1. Shapes of the grains studied in this paper (size 315–400 μm). From left to right, S_1 (natural rounded sand), S_2 (crushed sand) and S_3 (crushed sand).