Cement and Concrete Research 82 (2016) 130–141

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: www.elsevier.com/locate/cemconres

Homogeneous and heterogeneous rheology and flow-induced microstructures of a fresh fiber-reinforced mortar

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ARTICLE INFO

Article history:
Received 26 September 2014
Accepted 28 December 2015
Available online 29 January 2016

Keywords:
Mortar (E)
Fiber reinforcement (E)
Rheology (A)
Microstructure (B)
Workability (A)

ABSTRACT

External wall insulation (EWI) usually comprises a porous cement mortar used as protective external render into which short fibers are added to enhance its mechanical properties. The rheology of these porous, fibrous, and granular suspensions was investigated using lubricated compression tests in the fresh state, whereas flow-induced porous microstructures were studied using X-ray microtomography. We show that these suspensions exhibit a homogeneous isovolume flow regime and two heterogeneous flow regimes, *i.e.*, a consolidating regime, and a consolidating and segregating regime. A decrease in the compression strain rate and/or an increase in the number of fiber contacts in the entangled fibrous network induced flow heterogeneity accompanied by heterogeneous modifications of density, porosity, and pore size distribution of render. These undesirable microstructure changes are prone to occur during mortar processing and placement. They drastically affect the properties of renders such as the permeability that was calculated using X-ray microtomography images and pore scale numerical simulation.

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1. Introduction

External wall insulation (EWI) is an interesting technology for the thermal insulation of buildings. EWI limits thermal bridges compared to other insulation systems and can be used for aesthetic purposes. An EWI system generally comprises an insulating layer made up of polystyrene, polyurethane, wood, or mineral wool topped off with a protective render, *i.e.*, a highly porous cement mortar reinforced with glass or metal fiber meshes. The placement of the render involves at least four operations. A first layer of render is sprayed onto the insulating material. Then a glass fiber mesh is fixed in the wet render and a second layer of render is added. Finally, a third layer of finishing render is placed after one or two weeks. The cost induced by these numerous processing operations hinders the development of this insulation technique. However, the use of renders that are self-reinforced with discontinuous fibers constitutes an interesting alternative to the classical EWI systems [1]. Self-reinforced renders are sprayed onto the insulation material in one operation using specially designed apparatuses. Hence, these renders must exhibit a particular and complex rheological behavior, which leads to complicated formulation problems. Indeed, they have to undergo flow as they are conveyed through the pneumatic spraying apparatus, projected onto the insulation materials, and levelled. They must also stick onto the insulating material without flowing. Thus, during these stages, where fresh mortars are subjected to a wide range of strain rates (from very low to very high values), it is of great importance to control their flow-induced granular, fibrous, and porous microstructures. Such evolution of render structures drastically affect the mechanical and physical properties of mortars in their hardened state [15,16], such as their permeability, which plays an important role for the performance of the EWI systems.

Characterizing and controlling the rheology of these fiber-reinforced and porous renders is a complex problem. For instance, it is well known that adding fibers into fresh cements induces important variations in their rheological behavior, with complex thickening effects, evolution of fiber orientation and placement [2–9,57] that modify their microstructure [4,8,9,13,14]. The study of the links between the evolution of the microstructure of self-reinforced renders and the processing conditions involves the use of suitable experimental devices. In a previous study [17], lubricated compression experiments were performed using a rheometer having large dimensions. For these tests, a commercial organo-mineral render (ParexLanko, Maité monocomposant) was reinforced with commercial alkali-resistant glass fiber bundles (Owens Corning, Cemfil Anti-crack). Two types of fiber bundles were also used, *i.e.*, high performance (HP) or highly dispersive (HD) fiber bundles, with fiber contents and aspect ratios typically used for practical applications. Coupled with an analysis of the microstructure based on the use of three-dimensional X-ray microtomography images, these tests

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http://dx.doi.org/10.1016/j.cemconres.2015.12.012
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enabled the effects of fibers on the rheology and on the microstructure of the resulting fresh mortar to be estimated at a fixed strain rate. These tests also enabled a better understanding of the interactions between the rheology, the microstructure, and the physical properties of the hardened render. The main conclusions that have been reported by Chalencon *et al.* (2010) [17] are as follows:

(1) The initial porous microstructure and granulometry of the render did not depend on the fiber content, nor the fiber orientation.
(2) For moderate strain rates, *i.e.*, $D_{33} = 10^{-1}$ s^{-1}, compression had a weak influence on the evolution of the microstructure. The porous and fibrous phases did not evolve: the 2D planar random orientation of the fiber was maintained and the macro-pore size distribution ranged from 50 and 600 μm in diameter and was centered on 200 μm, before and after compression.
(3) The stress levels increased with the fiber volume fraction f and the fiber aspect ratio r. However, the influence of the fiber reinforcement on the axial compression stress did not depend on the axial compression strain. For the HP fiber bundles (aspect ratios that ranged from 40 to 455), this evolution could be modelled by adopting a theoretical framework developed for incompressible semi-dilute Newtonian suspension of well dispersed and straight fibers [19,20]. On the contrary, this approach did not work for the HD fiber bundles (aspect ratios of 400 and 793). The potential origin of this behavior could be (i) the limited bending stiffness of the fibers (the HD fiber bundles disaggregated which resulted in nearly completely individualized glass filaments that could easily bend during flow [17], and thus limit the increase in the stress level compared to perfectly straight fibers) and (ii) the clusters of fibers that remained after processing.

In this previous study only one strain rate was tested. However, renders are subjected to high strain rates during the spraying stage: $D_{33} \approx 10$ s^{-1}. In some "dead zones" of the processing or spraying apparatuses, the strain rate can be very small values. The strain rate can also be very small if the renders slide on their substrate ($D_{33} \approx 10^{-5}$ s^{-1}). Hence, in this study, new rheometry experiments were performed using the same render in a wider range of applied strain rates for several fiber fractions to better investigate the coupling between these parameters and the flow-induced microstructures. For that purpose, the porous and fibrous microstructure of renders was studied using X-ray microtomography. X-ray micrographies were also used to estimate the permeability of renders by direct pore scale flow numerical simulations, which allowed us to illustrate the important coupling between the flow-induced microstructure of renders and one of their main end-use properties. These experiments also enabled a workability domain to be defined for renders based on the microstructure of their fibrous reinforcement and the imposed flow conditions.

2. Compression rheometry

2.1. Materials

The self-reinforced render was made up of a dry pre-mix composed of more or less spherical grains of various sizes and natures (polymers, cement, silica fillers, and sand limestone) and glass fiber bundles [17]. After pouring water in the granular pre-mix, the polymer was dispersed, air bubbles formed, and the as-obtained mixture formed a porous and concentrated granular suspension with a porosity of 17% in the fresh state [17]. For all rheological experiments, time t_0 was the initial time of the sample after 3-min mixing of the mortar constituents and 10-min rest.

The two types of fiber bundles were different because of their sizing, which enabled the HP fiber bundles not to disintegrate during both the processing and the rheological experiments, and the HD fiber bundles to disintegrate and to disperse in the render. The cross section of fiber bundles was initially composed of more than 200 fibers with a 14 μm diameter. The cross section dimensions were precisely measured by following a procedure reported by Chalencon *et al.* [17]. The cross section of the bundle could be represented by an ellipse with a minor axis $d_{\min} \approx 50$ μm and a major axis $d_{\max} \approx 400$–800 μm. Four bundle lengths l were used in this study for the HP fiber bundles: 6.1, 10.9, 20.9, and 72 mm, and three lengths for the HD fiber bundles: 5.6, 11.1, and 24 mm. Consequently, the aspect ratio $r = l/\sqrt{d_{\min} d_{\max}}$ of bundles ranged between 40 and 1700. Four mass fractions of fiber bundles were also used, *i.e.*, $f_w = 0.005, 0.01, 0.02$, and 0.04, corresponding to fiber volume fractions $f = 0.003, 0.007, 0.014$ and 0.027, respectively. Note that the fiber volume fraction was estimated from the fiber mass fraction and the specific masses of the fibers and the mortar without fiber.

The specific processing route reported by Chalencon *et al.* [17] was used to prepare cylindrical samples of self-reinforced render with controlled initial microstructure (planar random fiber orientation distribution and initial porosity of 17%) and restrained variability. As reported in Chalencon *et al.* [17], the global density and the local porosity of the samples did not exhibit any significant variations with the fiber content and the fiber type.

2.2. Compression tests

The rheology of the self-reinforced render was studied using a simple compression rheometer that was initially developed for studying the rheology of highly concentrated fiber suspensions such as fiber-reinforced polymers [21–24,46] and that was adapted for fiber-reinforced mortars [17]. Experiments consisted of compressing lubricated cylindrical samples (initial diameter D_0, initial height h_0) at constant axial average strain rate, $D_{33} = \dot{h}/h$, between two parallel horizontal plates. Four strain rates D_{33} were set at $1, 0.1, 0.01$, and 0.003 s^{-1} on the basis of the cross-head velocity capacity of the testing machine. Note that these strain rates were in the range of potential strain rates the render could be subjected to during processing. The height h and the axial load F_3 were recorded during compression and were used to calculate the average axial logarithmic compression strain $\varepsilon_{33} = \ln(h/h_0)$ and the nominal compression stress $\sum_{33} = 4F_3/(\pi D_0^2 h_0)$. The final height h_f and the final diameter D_f of deformed cylindrical samples were also measured so that the volumetric strain $\varepsilon_v = \ln(h_f D_f/(h_0 D_0))$ of each sample after compression was estimated.

3. Rheometry results

3.1. Flow regimes

Fig. 1 shows four typical compression stress–strain curves obtained for the reference render (without fiber) and two self-reinforced renders ($f = 0.7$ and 1.4%) at two strain rates ($D_{33} = 0.1$ and 0.003 s^{-1}).

- The gray curve was obtained for a non-reinforced render at a moderate strain rate $D_{33} = 0.1$ s^{-1}. The measured volumetric strain ε_v was higher than -2%, showing that the compression deformation was nearly isovolume. For the same strain rate, adding HD fibers ($f = 0.7$%, $r = 793$) induced an increase of the compression stress, but again the volumetric strain remained lower than 2%. The photographs show the concentric circles drawn on the upper surface of the specimens also remained circular and concentric during compression, and followed homothetic transformation. These observations demonstrated that, thanks to lubrication, the flow was homogeneous at the macroscopic scale.
- For $f = 0.7$% and a lower strain rate, *i.e.*, $D_{33} = 0.003$ s^{-1}, the stress level increased sharply. This phenomenon was accompanied by a large volume strain $\varepsilon_v = -8$%, showing that the sample was consolidated during the compression experiment. The circular marks drawn on the upper surface of the deformed sample also revealed

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