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MULTITECHNIQUE CHARACTERIZATION OF CEMENT PASTE WITH AND WITHOUT GRAPHENE OXIDE

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1. Introduction

Graphene oxide (GO) is a "two dimensional" material that contains functional groups such as hydroxyl, carboxyl, and epoxy [1]. The oxygen-containing functional groups facilitate dispersion of GO in aqueous solution. GO sheets have a large tensile strength and a large specific surface area [2]. This provides the motivation to explore the effects of graphene oxide GO mixed into cementitious composites, in particular because it has been reported that GO improves the microstructure and mechanical properties [3]. This is setting the scene for the present contribution. It is focused on quasi-isothermal calorimetry for characterization of the hydration kinetics, and on uniaxial compressive strength tests providing insight into the most important mechanical material property in cement and concrete research.

2. Materials and methods

Ordinary Portland cement, GO, and distilled water are used for paste production. Cement pastes with and without GO are produced with an initial water-to-cement mass ratio (w/c) of 0.42. The dosage of GO is 0.09% by mass of cement. The cement paste without GO is referred to as "OPC paste", and the one with GO as "OPC+GO paste".

The used GO comes in aqueous solution. Thus, part of the mixing water required to reach the target w/c value comes from the GO solution. The amount of additionally added distilled water is defined accordingly.

2.1 Quasi-isothermal calorimetry

Quasi-isothermal calorimetry is carried out at 20 °C using TAM Air isothermal calorimeter. The specimens are mixed outside the calorimeter, in order to achieve the high mixing energy required to avoid agglomeration of any solid material constituents. Right after mixing, the fresh paste is inserted into the testing machine. According to the producer of the calorimeter, it takes 45 minutes until the stable quasi-isothermal conditions are reached, which are required for reliable measurements. Thus, the heat-release rate resulting from the hydration reaction can only be captured from 45 minutes onwards. The tests are continued until 24 hours after paste production.

2.2 Uniaxial compressive strength tests

Strength tests are performed on cylindrical specimens. Their diameter and height are equal to 30 mm and 60 mm, respectively. The destructive tests are conducted 1, 2, and 3 days after material production. For each cylinder, the uniaxial compressive strength is calculated as the maximum force sustained by the specimen divided by the cross-sectional area.

3. Results

3.1 Hydration kinetics

Heat evolution curves follow the typical cement hydration profile. They comprise an induction period, an acceleration period, and a deceleration period.

GO alters the cement hydration profile compared to the OPC paste. It increases the heat release rate during both the induction period and the acceleration period. In addition, the main hydration peak is reached earlier and it is higher in the presence of GO compared to the OPC paste, see Fig. 1. Thus, GO accelerates the hydration kinetics. This is in agreement with the pertinent literature [2].

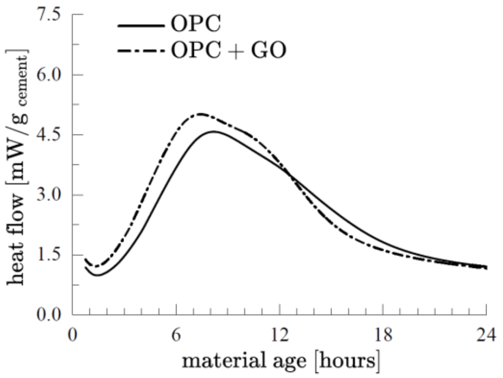

Fig. 1. Specific heat release rate as a function of material age

3.2 Uniaxial compressive strength evolution

The uniaxial compressive strength of the OPC paste and the OPC+GO paste, respectively, increases progressively with increasing material age. GO accelerates the early-age strength development, see Fig. 2. This is in agreement with the pertinent literature [2].

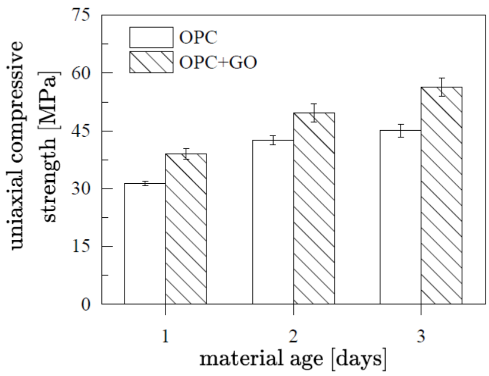

Fig. 1. Uniaxial compressive strength evolution as a function of material age.

4. Conclusion

From calorimetry and strength testing, the following conclusions are drawn:

1) The maximum heat flow of OPC with graphene oxide GO is increased by 10 % compared to OPC. Thus, GO exhibits the so-called "filler-effect" known from very finely ground limestone and/or silica powders.
2) The addition of graphene oxide GO with 0.09% mass of cement increases the early-age strength evolution of the OPC paste.

In the future, the presented experimental campaign will be enriched by adding two advanced characterization methods: (i) small angle oscillatory shear (SAOS) rheometry, in order to study the influence of GO on early-age structurization of cement paste in its gel-like state, and (ii) hourly three-minute creep testing, in order to study the influence of GO on the early-age evolution of the elastic modulus, the creep modulus, and the creep exponent of solid-state cement pastes [4].

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