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Improvement of the mechanical and physical properties of cement pastes and mortars through the addition isostatic graphite

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HIGHLIGHTS

- Isostatic graphite (IG) waste powder from the milling of molds for EDM, can be use as filler in advanced cement composites.
- Mechanical properties and thermal conductivity are increased with IG in cementitious composites.
- Carbonation of cementitious composites is increased with IG.
- IG-Cement Composite showed a sealed microstructure and close interphase by SEM analysis.
- IG used as addition can be new source for low-cost multifunctional cement-based materials.

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ABSTRACT

The following research work presents a characterization of cement pastes and mortars with Isostatic graphite (IG). This addition is a carbon-based addition from waste origin obtained after the mill of graphite blocks from synthetic origin to produce Electrical Discharge Machining (EDM). This carbon-based material presents a great carbon purity and a crystalline structure. The effects of IG addition on the fresh and hardened properties of cementitious composites have been studied. The microstructure of the composites and their mechanical and physical properties have been analyzed. IG addition modifies the fresh behavior of the composites, and later the physical properties, such as density and porosity. Moreover, the possibilities of using IG in cementitious materials have been highlighted in this work, thanks to the increase of the compressive and flexural strength, thermal conductivity and CO_2 entrapping capacity.

1. Introduction

Cement-based composites had been blended with many additions and admixtures, to achieve a multifunctional material and improve either its mechanical properties nor other properties: chemical, acoustical, thermal and so on. Some of these additions can come from waste materials which are this way revalorized forming part of new composites, also known as eco-friendly composites. In the last years many research works have been focused on the possibilities of additions derived from carbon in cement composites, like: carbon black [1], natural graphite [2–8], graphene nano-platelets [9–11], Colloidal graphite [12], graphene nano-tubes [13–15] Carbon nanofibers [16,17], graphene oxide [18]··· etc.

Some of these additions are commercialized and presented in a Nano-scale size, and can be used to obtain high-performance concrete, as they can increase the density of concrete through the increase of the packing density of the microstructure [16]. Moreover, these innovative composites with carbon additions have been studied to improve their thermal conductivity [1,5,6,17], thermal storage [5,20,21], piezoresistivity [6], electromagnetic wave-shielding [7,12,22]. Hence, they can be used for indoor electrical floor heating system [1], in airfield pavement deicing [19], as solar energy storage materials [5,20] as well as in vertical ground-heat exchangers [21].

Moreover, carbon derived additions can be used to increase the durability of cement based materials by increasing the abrasion resistance [2,11], reducing water and chloride ingress [9,10], and making them suitable also to protect reinforced concrete [3]. However, some of these additions, like carbon nano-tubes, nano-fibers

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or nanoplatelets, are expensive and are limited to be used in small volume fraction (VF) of the composite (<5%) [8,17]. Other composites which use carbon black or graphite, present a reduction of the mechanical strength of the cement-based composite, when their VF used exceeds only a 5% of the cement used in the composite [5].

Graphite is a material commonly used in many industrial manufactures. It can be obtained from quarries or synthetically, and it is a good electrical and thermal conductor, especially when a high purity is achieved [23,12]. The use of graphite as addition in cement based composites is gaining in significance because it increases the thermal conductivity and piezoresistivity of the composite [6].

However, it has been observed that the mechanical resistance can be drastically reduced [6]. For instance, an amount of only a 5% of natural graphite increases thermal conductivity a 100% but reduces the compressive strength and flexural strength by a 50 and 55%, respectively [20]. Ground expanded graphite (GEG) also improves the volume heat capacity and thermal conductivity of the cement composites, but when GEG content is only a 1%, thermal conductivity slightly varied but the compressive strength increased up to 16%. On the contrary, an increase of GEG up to 15% implied a reduction of the compressive strength to a 30% [5].

IG is a waste powder from the mill of Electrical Discharge Machining (EDM), which are previously produced synthetically with coke at high temperature and isostatic pressure. More than 700,000 tons graphite molds are consumed around the world every year, with a disposal of around 14,600 tons of powder material from the milling process [38,39]. IG used in this research, are graphite flakes with an average particle size (D50) of 10 μm.

In this work, a synthetic graphite obtained by isostatic pressure, isostatic graphite (IG), has been used as addition in cement based pastes and mortars. IG has been used before in gypsum-based materials by the authors with good mechanical and thermal results [24–26], but it has not been found yet in the cement composites. Thus, IG revalorized as addition, can increase the sustainability of cement-base composites in the construction field. To this end, mechanical and physical properties and the microstructure of the composites have been studied. Moreover, the behaviour of the different mixtures in fresh state, yield stress and flow diameter, with different IG percent and the carbonation through accelerated carbonation test after hardening have been also tested.

2. Experimental procedure

2.1. Raw materials

A Portland Cement CEM I/42.5 R manufactured by Cementos Portland Valderribas S.A.™, according to UNE-EN 197-1:2000 [27] was used. It has an initial setting time of 195 min and final setting time 260 min, and specific surface of 3.900 cm^2/g. Sand aggregate from siliceous origin, with a maximum size of 4 mm and a fine content less than 6% was used. Isostatic graphite filler with a 99% of carbon was used. It has an average particle size of 10 μm, its BET Surface Area is 26.3078 ± 0.2622 m^2/g, a bulk density of 0.44 kg/m^3 was used. In a previous study it was observed by Scanning Electron Microscope graphite powder presented a flaky shape [24]. A plasticizer/air-entraining with ethoxylated polymer recommended specifically for mortars, Sikanol-M by Sika™ Construction Products with a density of 1.05 g/cm^3 and tested according to ASTM C 457-71 and to EN 934-3 was used.

Several pastes and mortars were designed keeping the w/c ratio constant in each serial to analyze the properties of the mixtures with graphite addition, one a cement paste with 0.40 and two mortars with 0.55 and 0.70. As the consistency of the fresh mortar with 0.55 w/c was dry after adding graphite, the plasticizer was added. The batches were mixed with helical-ribbons impeller rotated by a turbine mixer providing a speed of 3000 RPM and the mixing steps for every test follows the standard procedure defined by UNE 196-7 [28]. After casting the samples were kept immersed the firsts 24 h and later kept in a curing chamber at 23 ± 2.0 °C and 95% relative humidity and dried before testing at 35 °C during 24 h.

2.2. Test methods

2.2.1. Fresh state tests

The workability of the samples was determined using the flow table test following the instructions determined in the UNE-EN 1015-3:2000 [29] with a standardized cone, of 50 mm top diameter, 100 mm base diameter and height 150 mm. Moreover, the Yield stress (τ_0) of the fresh samples was determined using a slump method, developed by Murata [30]. Yield stress (τ_0) can be obtained with Eq. (1):

$$\tau_0 = \frac{Wx}{2 * \pi * R^2} \quad (1)$$

where W_x is the dead weight of the fresh batch in the cone and R is the diameter of spread given by the paste after demolding.

The setting time of the different cement batches was studied according to the European Standard EN196-3.2005 [31] and the results are shown in Table 1.

2.2.2. Microstructural analysis

A microstructural analysis of the cement pastes and mortar was developed by the use of a Scanning Electron Microscope and Energy Dispersive X-ray Spectroscopy (SEM/EDX). To analyze the aggregate-matrix interface, a study through fractured samples on the MC-30G-0.55P mortar was performed.

Besides, carbonation depth was studied using a pH indicator based on a phenolphthalein solution sprayed directly on the cracked face of each cementitious sample [32]. The samples at 28 days of curing were stored in a carbonation chamber at 20 °C and 65% relative humidity during 160 days before testing and a constant flux of 2% CO_2. Depth of carbonation was carried out immediately after the broken surface was exposed to phenolphthalein indicator. Maximum depth carbonation and medium depth carbonation were measured.

2.2.3. Physical properties tests

The variation of the water absorbed by the samples through capillary test was also measured recording the weight of the sample at 5, 10, 20, 30, 60 and 1440 min, following the EN 13279-2:2014 standard [33]. Moreover, bulk density, immersion absorption coefficient (Cabs) and open porosity (Pop) of the composites after 28 days age was determined through the Archimedes method.

Ultrasonic Modulus (US Modulus) was determined by direct contact with the cementitious composites and it was calculated with Eq. (2):

$$UM = \rho.v^2\left[(kg/m^3)\cdot(m/s)^2\right](GPa) \quad (2)$$

where ρ is the density in kg/m^3 and v the velocity in m/s.

Thermal conductivity (k) was performed using an insulating house used in a previous research work [25]. The previously dried specimens 220 × 220 × 40 mm^3 were set in a hole of the insulating house where the sample was fixed. Five temperatures were recorded by means of thermocouples every minute since a stationary level was achieved during 30 min: Inside (50 °C) and outside of the house, laboratory temperature of 22 °C; on the internal face of the specimen; between a 3 cm polystyrene board and the sample and on the external face of the polystyrene board. Thermal