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Mechanical properties of alkali activated blast furnace slag pastes reinforced with carbon fibers

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HIGHLIGHTS

- The effect of CF aspect ratio and dosage in alkali-activated slag pastes is discussed.
- Blast furnace slag activated with water glass and CF reinforced to reduce shrinkage.
- CF could preserve AAS pastes from breaking due to high drying shrinkage.
- Compressive strength increases up to 19% were obtained with 3 mm long CF.
- Bending strength up to 18.6 MPa was achieved with 1% (by slag mass) 6 mm long CF.

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ABSTRACT

Alkaline activation has become a useful procedure to obtain high strength cement composites without Portland cement. Different industrial byproducts can be activated using this technique, e.g. blast furnace slag, fly ash or metakaolin. Thus a new generation of more sustainable composites is being developed. However, the main disadvantage of these composites is their high drying shrinkage strain. Among the different approaches to address this problem, shrinkage reducing admixtures are the most used, but the addition of fibers can be useful for this purpose. In this work, alkali activated slag (AAS) pastes have been reinforced with carbon fibers (CF). The effect of the concentration of alkaline activator (Na_2O% and silica modulus) and the fiber aspect ratio (using different length fibers with the same diameter) on the mechanical properties has been assessed. Mechanical characterization comprised bending and compressive strength tests, ultrasonic pulse velocity and density measurements, and drying shrinkage tests, in which CF were capable of improving the mechanical strengths of AAS pastes while controlling the specimens strain due to shrinkage. CF additions increased bending strength up to five times, and increases up to 20% in compressive strength were observed. Furthermore, CF can be a convenient addition to control the drying shrinkage of AAS composites. Even in adverse conditions, such us 50% RH, where unreinforced specimens actually broke, CF could guarantee the stability of the specimen.

1. Introduction

The application of industrial byproducts, some of which show hydraulic properties, has grown as an alternative to Portland cement in civil engineering applications [1–6]. Alkaline activation has become a useful procedure to obtain high strength cement composites without Portland cement [4–6]. Thus, besides of reusing a waste product, the energy and environmental costs, related to clinker production, can be reduced. And therefore, a new generation of more sustainable composites is being developed [5,7].

The use of water glass (sodium silicate base activators $Na_2SiO_3 \cdot xH_2O + NaOH$) as an alkaline activator for blast furnace slag (BFS) has been recommended for the mechanical and durability properties of their composites, usually know as alkali activated slag (AAS) [1,5]. The main advantage of AAS is their resistance against aggressive agents (e.g. sulphates or chlorides), due to their low porosity [3]. These composites also show high compressive strength, low hydration heat, high carbonation or freeze-thaw resistances [2–6]. But among their disadvantages, their important drying shrinkage cannot be ignored [8–11].

The addition of short fibers to brittle composites represents a commonly used technique to improve their mechanical properties, especially those related to their tensile and shrinkage behaviors

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[12–14]. There are multiple references reporting this mechanical gains in Portland cement composites [12–22]. Even in some cases the sustainability issue has been addressed by means of natural [18] or polymeric recycled fibers [19]. Besides, some reports can be found where different fiber types are used in AAS composites with different levels of success reducing the shrinkage of the material [23–31]. Fibers can be made on different materials such as: steel [23–26], PE [27], PVA [28], PP [29], AR glass fibers [30] or even carbon [31]. However there is a lack of information regarding the use of short carbon fibers for this purpose. Besides ref [31], there are few references using carbon fibers to reinforce geopolymers [7,32,33], but they don't use BFS as binder, e.g. in ref [7] alkali activated metakaolin was used, in ref [32] fly ash was activated, while in [33] metakaolin was reinforced with continuous carbon fibers. The current study uses CF with a twofold aim, first improve the mechanical performance of AAS pastes, and second the use of these CF reinforced composites as strain sensors (as will be introduced below). Hence, the CF dosages and lengths have been chosen according to previous research on Portland cement composites for their strain sensing capacities [34], and differ greatly from those used in ref [31].

Carbon fibers offer another extra advantage, as they can be used to functionalize cement composites due to electrical properties improvements [35]. These functional properties include strain or damage sensing [34,36,37], heating control [38,39], EMI shielding [40] or anode for electrochemical chloride extraction [41] among others. And some of them have been successfully applied in civil engineering structures, always using cement composites [36,42].

To achieve these functionalities, a certain level of electrical conductivity is necessary. Therefore, as concrete is a bad electrical conductor, conductive admixtures are needed. Several researchers have focused on these conductive admixtures (e.g., steel fibers, or carbon materials: carbon fibers, graphite powder, carbon nanofibers or nanotubes) in order to achieve a better electrical behavior without compromising the composite's mechanical properties [34,35]. Thus, for each type of admixture, the relationship between their dosage and the composite's conductivity has to be determined. Researchers have focused on the minimum amount of admixture which guarantees low resistivity [34,35,43,44].

Strain and damage sensing, i.e. the relationship between the material's resistivity and its stress or strain state, have been investigated in several research papers using fiber-reinforced cementitious materials based on Portland cement [34,35]. Nonetheless, these applications of carbon fiber-reinforced cement composites based on alkaline activation have been barely studied [32,45]. An interesting preliminary study, regarding the self-sensing capacity of AAS composites reinforced with CF, was reported with remarkable results compared to their cement composites counterparts [45].

Therefore, the main objective of the present paper is to evaluate the influence of carbon fiber addition on the mechanical properties of AAS composites, which will be used afterwards as strain sensors (despite this application is not discussed in this work). In this manner, a compromise between structural and functional applications can be achieved, and the optimal dosage can be selected to combine good strain sensing capacity and mechanical performance. Thus, the multifunctionality of carbon fiber reinforced cement composites would be improved by the sustainability and mechanical properties enhancement of alkaline cements.

2. Experimental procedure

2.1. Materials and specimen preparation

Carbon fiber alkali activated slag (CF-AAS) pastes were made using blast furnace slag (BFS) with an alkaline activator compound of commercial water glass (an aqueous solution with mass ratios of 27% SiO_2 and 8% Na_2O) and a NaOH solution (with different concentration for each particular dosage). The oxide composition of the BFS obtained by X-ray fluorescence (XRF) is included in Table 1. A test to assess the specific surface area of the BFS was made according to the Blaine method specified in UNE-EN 196-6:2010 [46]. The average specific surface area was 525.7 ± 19.1 m^2/kg.

A preliminary study of pastes without CF was performed to select the best AAS dosages to include the fibers. In this first stage the silica modulus of the activator ($M_s = SiO_2/Na_2O$ mass ratio) was 1.0, 1.2 or 1.4, the Na_2O dosage changed between 3, 4 and 5% (with respect to BFS mass), and the activator/BFS ratio was fixed at 0.56. Afterwards, two AAS dosages were selected (4% Na_2O with M_s = 1.4, and 5% Na_2O with M_s = 1.0) and different unsized PAN-based CF were included in the mix (see main properties in Table 2). Three CF lengths were used, 3, 6 and 12 mm (i.e. ⅛, ¼ and ½ inch), and three different mass ratios of each length were prepared, 0.2, 0.5 and 1.0% (by BFS mass).

In order to improve the CF dispersion in the mix an ultrasounds treatment was applied [31]. The CF were stirred in part of the mixing water and an ultrasonic device, model Hielscher UP200S was used at full power for 10 min. Afterwards, all the remaining components of the alkali solution were mixed and stirred by hand together with the treated CF. Then all materials, i.e. alkali solution (with or without CF) and BFS, were put in an automatic mixer for five minutes. The fresh mix was then poured into prismatic steel molds, 4 × 4 × 16 cm for the mechanical tests and 2.5 × 2.5 × 25 cm for the shrinkage tests. A mechanical treatment to remove any air bubbles was made using a vibration table, and the molds were kept in a controlled environment room (20 °C and >99% RH) for 24 h. Afterwards, specimens were demolded and conserved in the same conditions until tested.

2.2. Testing

First, a study of the paste workability has been carried out according to the flow table test included in UNE83811:1992EX [47]. Afterwards, mechanical properties tests – bending and compressive strengths (R_f and R_c), ultrasonic pulse velocity (*UPV*) and density – were run at curing ages of 7, 28 and 60 days. All tests were made according to European standards: Bending strength and compressive strength UNE-EN 196-1:2005 [48], UPV UNE-EN 12504-4:2006 [49] and density UNE-EN-1015-10:2000/A1:2007 [50]. Shrinkage tests according to UNE-80112:1989 EX [51] were also made, in which case the specimens were kept, after demolding, into a controlled environment of 20 °C and 50% RH, and the strain variations were measured daily until stabilization.

3. Results and discussion

3.1. Mechanical properties of unreinforced AAS

First, prior to any CF addition, the influence on mechanical properties of the sodium oxide concentration in the activator and its silica modulus were studied and compared with other researches. Table 3 includes the results of this first experimental campaign, where AAS pastes with no CF were tested. The average value and standard deviation (*SD*) for three or six measures, depending on each test set up, are included for three different Na_2O dosages (% by BFS mass) and three M_s for two different curing ages. There are no results for the dosage with 3% Na_2O and silica modulus equal to 1.0 because the paste did not totally set and hardened enough to be tested. On the other hand, the dosage with

Table 1
Chemical composition of the BFS obtained by XRF.

Chemical composition	Mass%
CaO	45.20
SiO_2	32.32
Al_2O_3	10.62
MgO	6.35
SO_3	2.03
Fe_2O_3	0.86
TiO_2	0.54
K_2O	0.44
ZrO_2	0.42
Na_2O	0.32
MnO	0.31
BaO	0.15
P_2O_5	0.13
Cl	0.13
Other oxides	0.19