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ECF22 - Loading and Environmental effects on Structural Integrity

# Influence of pyrolysis parameters on the efficiency of the biochar as nanoparticles into cement-based composites

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## Abstract

In this research, a particular kind of biochar provided by UK Biochar Centre has been added as nanoparticles into cementitious composites. Its principle characteristic lies in the standardization of its process production, that makes it suitable to be used as filler in cement-matrix composites, ensuring the reproducibility of the cement mix (I. Cosentino “The use of Bio-char for sustainable and durable concrete”, 2017). The pyrolysis parameters and the content of carbon in the standardized biochar influenced its efficiency to enhance the mechanical properties of the cement composites: the results, in terms of flexural strength and fracture energy, have been worse than those obtained in previous studies (L. Restuccia “Re-think, Re-use: agro-food and C&D waste for high-performance sustainable cementitious composites”, 2016), in which particles have been produced with higher temperature. However, also with standardized biochar a general enhancement of mechanical properties has been recorded, a sign that they can be used to create new green building materials.

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*Keywords:* pyrolysis; biochar; cement-based composites; carbon nanoparticles; mechanical properties; flexural strength; fracture energy;

## 1. Introduction

The use of green concrete is spreading through partial substitution of raw materials and partial replacement of clinker with alternative constituents such as fly ashes, blast-furnace slag, silica fume (Supplementary Cementitious Materials, SCMs) or Calcium Sulfoaluminate Cement, Magnesium Oxide based Cement, Geopolymers, CO<sub>2</sub>-cured cement (Alternative Cementitious Materials, ACMs).

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Pyrolysis process is a promising approach that can be used to convert biomass waste into energy, in the form of synthetic fuel gas (or syngas), fuel oils (or bio-oils) and a solid residue rich in carbon (biochar).

The biochar is used mainly in agriculture as soil improver, increasing soil fertility and allowing the capture of carbon in soils, contributing to climate change mitigation. (Schmidt, H.P., “55 Uses of Biochar”, Ithaca Journal, 1, pp. 286–289, 2012), but in the last few years, the interest in this material has increased enormously finding several applications in building materials (L. Restuccia “Re-think, Re-use: agro-food and C&D waste for high-performance sustainable cementitious composites”, 2016).

In this research work, standardized biochar nanoparticles have been used in cementitious composites, to understand its useful to obtain an enhancement of the mechanical properties of cement-based materials.

## 2. Materials and methods

Ordinary Portland Cement, deionized water, superplasticizer, and biochar were used for the preparation of cement mixes. Softwood Biochar (SWC), provided by the UK Biochar Centre, is a set of biochar produced from pyrolyzed feedstock with nominal peak temperature of 700°C and it has been used because its high degree of reproducibility. SWC was added as nanoparticles in the cementitious composites and the addition percentages of 0.8% and 1% with respect the weight of cement have been established by previous studies (L. Restuccia, G.A. Ferro “Promising low cost carbon-based materials to improve strength and toughness in cement composites”, 2016). The standardized biochar SWC has been grinded in planetary mill, by using alumina balls of 2 mm diameter and ethanol (Figure 1).



Fig. 1: SWC Biochar - Grinding operation with planetary multi-station mill

Through the scanning electron microscope with field emission source (FE-SEM) it was possible to characterize the morphology of the particles with a resolution around the nanometer. The recipes used for the preparation of cement mixtures are shown in the Table 1. For each experimental set, 8 specimens were prepared, 4 of whom were tested after 7 days of curing and the remaining 4 were tested after 28 days of curing. (Table 2)

Table 1. Cement Mix Recipes

Materials		Recipe N° 1	Recipe N° 2	Recipe N° 3
Cement	[g]	230	230	230
Water	[g]	80.5	80.5	80.5
w/c ratio	[-]	0.35	0.35	0.35
Superplasticizer	[g]	2.3	2.3	2.3
Biochar SWC	[g]	0	1.84	2.3

Table 2. Set of Experimental Specimens

Mixture ID		N° specimens (7 days)	N° specimens (28 days)
OPC	Sp 1%	4	4
SWC 0.8%	Sp 1%	4	4
SWC 1%	Sp 1%	4	4

### 3. Experimental procedure

#### 3.1. Specimens manufacturing

All the materials were weighed according to the amounts required in the cement mixes.

Deionized water, superplasticizer and the standardized biochar in powder were weighed and mixed together inside a plastic beaker, subsequently immersed in an ultrasonic bath for a duration of 10 minutes to allow a good homogenization of the compound. Then, the cement was gradually poured into the solution within the first minute and the mixture was then subjected to mechanical mixing by means of a vertical rod agitator with four wings steel propeller with a direct motor with variation of speed.

At the end of the mixing phase, the cement mixture was slowly transferred into the steel formwork, made up of four 20x20x80 mm<sup>3</sup> prismatic moulds, avoiding air entrainment and stored in a humid atmosphere for at least 24 hours and, once the specimens were unpacked, they were immersed in water for 7 and 28 days curing.

#### 3.2. Mechanical test activity

Each experimental specimen was submitted to three points bending test (TPB test). Before performing the TPB tests, a notch of depth equal to 6 mm and width equal to 2 mm was realized on each specimen by means of a metallographic truncator “TR 100 S Remet” in the centre line of the specimen face orthogonal to the casting surface, according to JCI-S-001 recommendation (JCI-S-001, Method of Test for Fracture Energy of Concrete by use of Notched Beam, Japan Concrete Institute, 2003). The TPB tests were performed using a Zwick Line-Z050, a single column displacement-controlled testing machine with load cell of 1 kN. All the specimens were tested in Crack Mouth Opening Displacement mode (CMOD) through a clip-on gauge. A span of 65 mm and a test speed of 0.005 mm/min were adopted.

Flexural Strength,  $\sigma_F$ , was determined as it follows:

$$\sigma_{f,max} = F_{max} \cdot \frac{3L}{2bh^2} \quad [MPa] \quad (1)$$

in which L is the span equal to 65 mm, b the specimen depth equal to 20 mm and h the net ligament height equal to 14 mm.

The TPB tests let to evaluate the Fracture Energy  $G_F$ , by using the equation proposed in the JCI-S-001 standard (JCI-S-001, Method of Test for Fracture Energy of Concrete by use of Notched Beam, Japan Concrete Institute, 2003):

$$G_F = \frac{0.75W_0 + W_1}{A_{lig}} = G_{F0} + G_{Fcorr} \quad \left[ \frac{N}{mm} \right] \quad (2)$$

in which  $A_{lig}$  [mm<sup>2</sup>] is the area of the nominal ligament,  $W_0$  [N·mm] is the area below CMOD curve up to rupture of specimen and  $W_1$  [N·mm] is the work done by deadweight of specimen and loading:

$$W_1 = 0.75 \left( \frac{S}{L} m_1 + 2m_2 \right) g \cdot CMOD_c \quad [N \cdot mm] \quad (3)$$

in which S is the loading span, L is the total length of specimen,  $m_1$  is the mass of the notched specimen,  $m_2$  is the mass of the loading arrangement part not attached to testing machine but placed on beam until rupture, g is the gravity acceleration and CMOD<sub>c</sub> is the crack mouth opening displacement at the rupture.

### 4. Results and discussion

The results were elaborated with statistical tools, specifically the mean value and the standard deviation of the Maximum Force,  $F_{max}$ , the Flexural Strength,  $\sigma_F$ , and the Fracture Energy,  $G_F$ , were calculated.

The specimens realized with the addition of the biochar had a clear effect on the increase in unpredictability of the results probably due to the non-uniform dispersion of the nanoparticles into the cement paste (L. Restuccia, G.A. Ferro “Promising low cost carbon-based materials to improve strength and toughness in cement composites” Construction and Building Materials 126 (2016) 1034–1043).

However, samples characterized by the addition of biochar have a greater Flexural Strength than samples without it, both at 7 and 28 days; this increase amounts to around 20%. Furthermore, there was no substantial difference between the two additions percentages of biochar used. (Figure 2)

Starting from TPB tests, it was possible to study the Fracture Energy of the experimental specimens and it was observed that it slightly increased with the introduction of biochar in the cement paste, both at 7 and 28 days. (Figure 3)

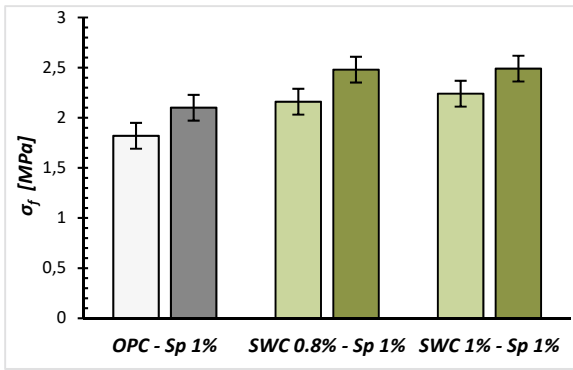


Fig. 2: TPB Test: Flexural strength – 7 and 28 days

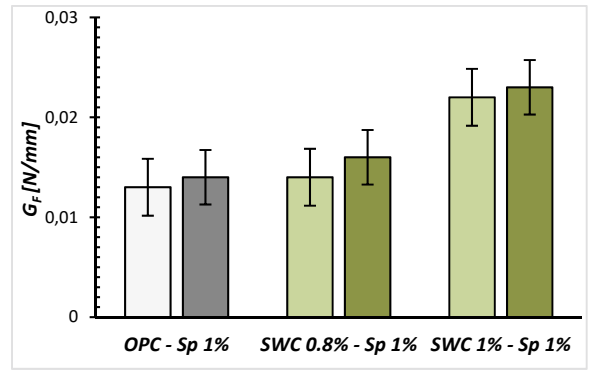


Fig. 3: Fracture Energy – 7 and 28 days - JCI-S-001 standard

From the Load-CMOD curves graph (Figure 4) it was possible to notice that the pyrolyzed nanoparticles within the cement-based composites determine a better mechanical behavior in terms of flexural strength as well as toughness.

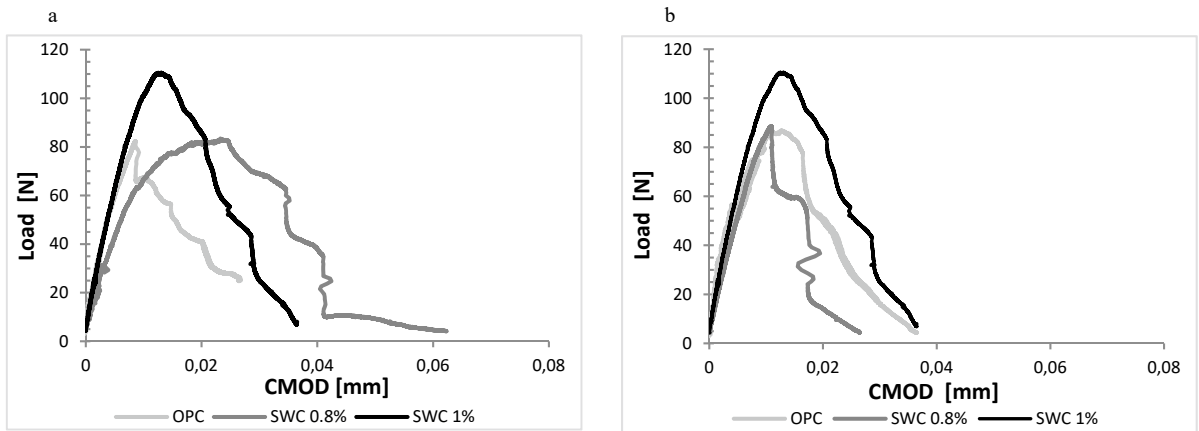


Fig. 4: (a) Load vs CMOD Curve, 7 days; (b) Load vs CMOD Curve, 28 days

Probably, the percentage of carbon present inside the standardized biochar used in this study (90.21%) compared to the one inside the self-produced biochar used in the previous studies (97.8%) as well as the pyrolysis temperature (700°C in this study compared to 800°C in the previous studies) have a great influence on the efficiency of the biochar, as also highlighted by other literature studies (S. Gupta and H. W. Kua “Factors Determining the Potential of Biochar as a Carbon Capturing and Sequestering Construction Material\_ Critical Review “; S. Gupta, H. W. Kua, Sin Yee Tan Cynthia “Use of biochar-coated polypropylene fibers for carbon sequestration and physical improvement of mortar”, Cement and Concrete Composites 83 (2017) 171-187). Gupta and Kua (2017) underlined key production factors (specifically, pyrolysis temperature, heating rate, and pressure) determining the potential of biochar as a carbon capturing and sequestering construction material.

## 5. Conclusions

The new building materials must always be more performing and innovative, at the same time the traditional manufacturing process will have to become increasingly efficient and sustainable to cope with the environmental emergency, with the aim of producing traditional building materials with better performance and with a lower energy consumption.

The present research focused on the use of the biochar in cement-based composites in different percentages of addition with respect to the weight of cement, in line with previous experimental studies (L. Restuccia, G.A. Ferro “Promising low cost carbon-based materials to improve strength and toughness in cement composites”, 2016). In these previous studies, the biochar used was self-produced through the pyrolysis of agro-food waste unlike that used in the present experimental activity, which was standardized to simplify the high-performance cement paste packaging process. (I. Cosentino “The use of Bio-char for sustainable and durable concrete”, 2017)

The results of the mechanical tests showed a promising improvement in strength, toughness, and ductility.

In fact, higher flexural strength and fracture energy values were recorded for specimens with the addition of biochar compared to those of the specimens without it.

However, the flexural strength and fracture energy values are lower than those of previous studies. This fact could be linked to the different pyrolysis parameters used in the production of biochar (temperature, heating rate, and pressure). The results could therefore be influenced by the type of carbonaceous material and by the production parameters rather than by the carbon particles size.

From an economic point of view, these carbon particles have zero costs, as they are the waste of the biomass pyrolysis process. For this reason, they represent a good material for new green construction materials.

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