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Mechanical characterization of different biochar-based cement composites

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Abstract

The attention on the use of raw materials, the energy consumption as well as carbon dioxide production of cement factories are boosting the experimentation on innovative and sustainable materials in concrete technology. In recent years, biochar has become an emblematic material with a thousand facets. Mainly investigated up to now as amending in the agricultural field, biochar can be explored as a building material due to its innumerable properties. Indeed, several applications have been studied to use it as a filler to modify the nanogranular nature of the cement matrix, or as a substitute for clinker, aggregates and clay, reducing the carbon footprint and the emissions of greenhouse gases linked to the production processes of cementitious materials.
In this paper, nano/micro-particles of biochar, the solid by-product from the gasification process of biomass derived from wood waste, has been used in different cement composites aiming at determining the optimal percentage of addition while trying to guarantee an improvement of mechanical properties. The results showed that an optimized percentage of biochar nano/micro-particles can increase the strength and toughness of the composites.

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Keywords: Biochar; nanoparticles; filler; cement-based composites; flexural strength; toughness

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1. Introduction

Global cement production is the third largest source of anthropogenic carbon dioxide emissions, (Andrew 2018). In general, there are three aspects of cement production that result in emissions of CO_2. The first is the chemical reaction involved in the production of cement: the heating step to obtain clinker is responsible for 50% of CO_2 release in the atmosphere. The second source of emissions, responsible for 35% of them, is the combustion of fossil fuels necessary to reach about 1450 °C in the kiln, while the third one is related to the indirect emissions from the electricity use, for example during the grinding process, (Scrivener et al. 2018). Although cement production is highly energy consuming and it has a severe impact on the environment, concrete is an essential product in our society. For this reason, there is a growing interest in finding sustainable solutions to reduce its carbon footprint and the utilization of raw materials (Imbabi et al. 2012, Miller et al. 2018, Suhendro 2014).

Manufactured nanomaterials (MNMs) and nanocomposites are being considered for various uses in construction and related infrastructure industries, because they improve vital characteristics of construction materials such as strength, durability and lightness (Lee et al. 2010). Unfortunately, leaving aside the relative high cost due to their production and functionalization, the use of nanoparticles in construction materials is still difficult especially for potential adverse effects on human and environmental health (Figarol et al. 2015). Some MNMs could potentially be considered as emerging pollutants: regulation for their use has not currently been studied, so concerns about the risks associated with public and environmental health are raising (Berger 2010).

Recently, The International Biochar Initiative (IBI) has defined biochar as "a solid material obtained from the thermochemical conversion of biomass in an oxygen-limited environment" (IBI 2015), or rather, as the carbonaceous waste of the biochemical thermochemical conversion process. This 2,000-year-old material makes it possible to transform agricultural waste such as wood or municipal solid waste, crop residues, rice husks, quinoa and lupin residuals, tobacco seeds, paper mill and olive mill sludges, algal biomasses (e.g. Duku et al. 2011, Shackley et al. 2011) into soil transformers; therefore, it is mainly used as soil amendment (among others, Gonzaga et al. 2018, Li et al. 2017, Agegnehu et al. 2017). However, in the last years, biochar market has grown, and its use is becoming very flexible, including for example also humidity sensors (Afify et al. 2017, Ziegler et al. 2017).

Lately, biochar has been explored as a building material and there is an emerging trend of its use as additive/replacement in cementitious composites (Khalid et al. 2018, Gupta et al. 2017, Gupta et al. 2018[b], Gupta and Kua 2019, Akhtar and Sarmah 2018, Zeidabadi et al. 2018, Zhao et al. 2019, Khushnood et al. 2016, Restuccia et al. 2017, Restuccia and Ferro 2016, Restuccia and Ferro 2018, Belletti et al. 2019).

Gupta et al. (2017, 2018[b]) used biochar derived from mixed food waste, rice and wood waste as carbon sequestering additive in mortar, obtaining a quite satisfactory mechanical strength compared to control mix by adding 1–2 % (by weight of cement) of biochar. Then, Gupta and Kua (2019) found that finer biochar particles guarantee an improvement of early strength and water tightness compared to normal biochar (with macro-pores) when biochar is used in cement mortar mixtures and recommend that biochar from wood waste can be used as filler material for improved strength development and water tightness of concrete constructions. Akhtar and Sarmah (2018) investigated the effect of biochar mixed with cement on the mechanical properties of concrete replacing the cement content up to 1% of total volume with three different types of biochar, such as poultry litter, rice husk and pulp and paper mill sludge biochar. Results showed that compressive strength was almost equal to that of reference one by using pulp and paper mill sludge biochar at 0.1% replacement of total volume. Regarding the flexural strength, 20% increment in comparison with the control specimens was found when poultry litter and rice husk biochar were added to the mixture at 0.1%. Zeidabadi et al. (2018) replaced up to 10% of cement (by weight) in concrete mixture with rice husk and bagasse biochar. Concrete samples containing 5% biochar had a compressive and tensile strength improvement by more than 50% and 78% respectively, compared to ordinary mix. Moreover, the samples in which 10% of biochar was used as a replacement showed a compressive strength improvement by more than 22% (with respect to the control concrete). In addition, Zhao et al. (2019) incorporated different percentages of biochar in vegetation concrete to study the trend in porosity, permeability and compatibility of plants. Discovering that the height of the plant, the length of the root and germination rate increased by more than 22% in the mixtures with approximately 2.30 wt% biochar, additionally, obtaining a slight increase in the compressive strength in comparison with the mixture without biochar.

At Politecnico of Torino some studies concerned the use of various pyrolyzed organic wastes to improve cement performance and reduce its environmental impact. In detail Khushnood et al. (2016) added peanut and hazelnut shells biochar to cement paste by up to 1 wt%, obtaining an increase of the flexural strength and toughness with respect to plain cement, but also gaining an increase in electromagnetic radiation shielding effect when 0.5 wt% of biochar was used. Moreover, Restuccia and Ferro (2016) reported an increase in fracture energy on cement paste samples after 28 curing days by more than 70% by addition of 0.8 wt% biochar, derived from hazelnut shell.

Notwithstanding the many benefits of using biochar in cement paste, mortar or concrete, there is not yet an ideal mix design for its use, obtaining dissimilar results, since biochar used in literature came from different raw materials and from production plants with different characteristics. Moreover, also the curing conditions of the obtained cement-based specimens influenced the benefits of incorporating biochar. In addition, the kind of treatment on biochar particles, such as sieving, grinding or pre-soaking, before their addition to the cement/concrete admixtures is expected to lead to different results. Within this context, this paper summarizes the main experimental results obtained from a collaborative research program developed by the Politecnico of Torino and the University of Parma, aimed at investigating the optimal percentage of addition of biochar nano/micro-particles from gasification waste in different cementitious materials.

2. Materials and methods

The experimental program consisted of tests on cement pastes conducted at Politecnico of Torino as well as on cementitious mortars, conducted at University of Parma, by using the same biochar, named “Grey Borgotaro”. This kind of biochar was produced from virgin wood chips through gasification. Its peculiarity is the fineness: it represents the finest waste in cogeneration energy production process, which cannot be used as a soil improver because of its particle size.

Cement pastes were realized by using Ordinary Portland Cement (OPC) type I 52.5 R. Biochar was mixed in a solution of deionized water and superplasticizer (Mapei Dynamon SP1) and then the cement was added. Five different percentages with respect to the weight of cement were used (0.8%, 1%, 1.5%, 2%, and 2.5%), with a water to cement w/c ratio equal to 0.35 and 1% of superplasticizer SP1, as reported in Table 1.

Table 1. Mix-design of different cement paste mixes.

Batch	Cement		Superplasticizer		Water		Biochar
	Type	[g]	Type	[g]	w/c	[g]	[g]
OPC	I 52.5R	460	Dynamon SP1	2.3	0.35	161.0	-
GBC 0.8%	I 52.5R	460	Dynamon SP1	2.3	0.35	161.0	3.68
GBC 1.0%	I 52.5R	460	Dynamon SP1	2.3	0.35	161.0	4.60
GBC 1.5%	I 52.5R	460	Dynamon SP1	2.3	0.35	161.0	6.90
GBC 2.0%	I 52.5R	460	Dynamon SP1	2.3	0.35	161.0	9.20
GBC 2.5%	I 52.5R	460	Dynamon SP1	2.3	0.35	161.0	11.50

Table 2. Mix-design of different mortar mixes.

Batch	Cement		Aggregates		Superplasticizer		Water		Biochar
	Type	[g]	CEN sand [g]	0-6.3 mm [g]	Type	[g]	w/c	[g]	[g]
M 0%	II/A-LL 32.5R	330	160	960	Dynamon SX42	2.6	0.55	181.5	-
M 1.0%	II/A-LL 32.5R	330	160	960	Dynamon SX42	3.0	0.55	181.5	3.30
M 2.5%	II/A-LL 32.5R	330	160	960	Dynamon SX42	3.3	0.55	181.5	8.25
N 0%	I 42.5R	400	200	980	Dynamon SP1	3.0	0.4	160.0	-
N 1.0%	I 42.5R	400	200	980	Dynamon SP1	3.4	0.4	160.0	4.00
N 2.5%	I 42.5R	400	200	980	Dynamon SP1	3.8	0.4	160.0	10.00

As regards mortars, two different admixtures were prepared, and for each type biochar was added in three different percentages with respect to the weight of cement (0%, 1%, 2.5%). The admixtures denoted as M in Table 2, which try to simulate in scale a ready-mix concrete, were prepared by using cement II/A-LL 32.5R and with a water-cement ratio w/c equal to 0.55. Type N, which refers to a mixture for precast concrete, was instead prepared with cement I 42.5R and with a water-cement ratio w/c equal to 0.40. It is worth noticing that superplasticizers, i.e. Mapei Dynamon SX42 and Mapei Dynamon SP1 for admixture M and N respectively, were added at slightly different proportions in each mortar mix, as reported in Table 2, in order to get about the same flowability as the control batch. Since biochar tends to absorb water, the superplasticizer percentage was increased with increasing biochar addition.

A proper mixing procedure was developed for mortars B and C, on the basis "method of concrete equivalent mortar" (MBE) suggested by Schwartzentruber and Catherine (2000). The aggregates and half of the total water were mixed for 120 s at low speed; after 180s of stop, cement and biochar (where applicable) were added and the admixture was mixed for 30 s. Then the remaining water and the superplasticizer were added and the mixing was conducted for other 30 s at low speed. After additional 60 s of mixing at high speed, the mixer was stopped for 90 s while the edge of the bowl was scraped. Lastly, the admixture was mixed at high speed for 30 s.

All the experimental composites were cast by using the geometry and the dimensions recommended in JCI-S-001 standard (2003). In more detail, cement pastes were cast into 20×20×80 mm prisms while mortars into 40×40×160 mm prisms. Once the curing in water was finished – i.e. after 7 and 28 days for cement pastes and 14 and 50 days for mortars – a U-shaped cut (6 mm deep for cement pastes and double for mortars) was made in the middle of the orthogonal face of the pouring surface of the specimens (Figure 1).

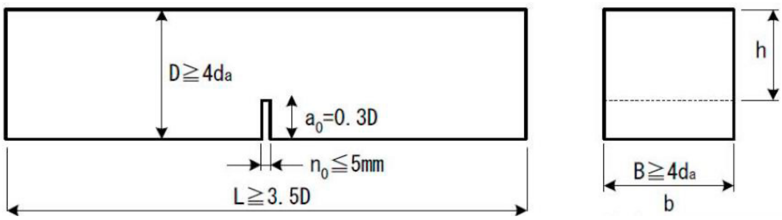

Fig. 1. Specimen geometry and dimensions recommended in JCI-S-001 standard (2003).

3. Experimental set-up and calculation

The prismatic notched specimens were subjected to three-point bending tests in Crack Mouth Opening Displacement (CMOD) by using a clip-on gauge in order to evaluate both flexural strength σ_f and Fracture Energy G_F. All the tests were performed by using a Zwick Z050 and an Instron 8862 Universal Test Machine, for cement pastes and mortars, respectively. Flexural strength σ_f was determined as follows:

$$\sigma_f = P_{\max} \frac{3S}{2bh^2} \tag{1}$$

where S is the net span of the specimen, while b and h represent the width and net depth of the mid-cross section, respectively. Moreover, P_{max} represents the peak load of the load (P) - CMOD curve.

Fracture Energy G_F was evaluated by integrating the area below the load (P) - CMOD curve, W_0, using the equation proposed in the JCI-S-001 standard (2003):

$$G_f = \frac{0.75W_0 + W_1}{A_{lig}} \tag{2}$$

where A_{lig} is the area of the broken ligament (bxh) and W_1 is the work done by deadweight of specimen and loading equipment.

4. Results and discussion

4.1. Flexural strength and fracture energy of cement pastes

Results of each batch in terms of flexural strength and fracture energy both at 7 and 28 days are reported in Table 3. Each batch is composed of four specimens and the value reported in the Table corresponds to the mean ± standard deviation. For a better comparison, the same mean values are also reported in Figures 2 and 3.

Table 3. Mechanical properties of the cement paste batches

Batch	Flexural Strength σ_f [MPa]		Fracture Energy G_F [N/mm]	
	7 days	28 days	7 days	28 days
OPC	1.82±0.17	2.10±0.22	0.013±0.007	0.019±0.003
GBC 0.8%	1.36±0.67	1.78±0.39	0.011±0.010	0.053±0.040
GBC 1.0%	1.70±0.31	2.25±0.77	0.027±0.006	0.030±0.020
GBC 1.5%	1.44±0.43	2.31±0.59	0.033±0.011	0.013±0.003
GBC 2.0%	2.10±0.52	2.06±0.92	0.033±0.005	0.033±0.011
GBC 2.5%	1.98±0.91	1.64±0.65	0.037±0.009	0.027±0.006

It can be recognized that the batches in which "Grey Borgotaro" has been used as a filler in small quantities (GBC 0.8%) show a decrease of flexural strength compared to the plain cementitious paste, both at 7 and 28 days. For 1 wt% and 1.5 wt% specimens, it can be noted that the values differ between 7 and 28 days: in the case of 7 days curing, there is a decrease in flexural strength, while at 28 days a significant increase, compared to the reference samples, is observed (+7% and +10% for GBC 1.0% and GBC 1.5%, respectively).

The biochar addition of 2% and 2.5% by weight of cement led to an increase (+15% and +9%, respectively) of the flexural strength at 7 days, while the results at 28 days are profoundly different between the two batches. In fact, GBC 2% roughly has the same flexural strength as the reference, while GBC 2.5% shows a significant decrease (almost 22%), probably due to a not optimal dispersion of biochar in the cementitious matrix.

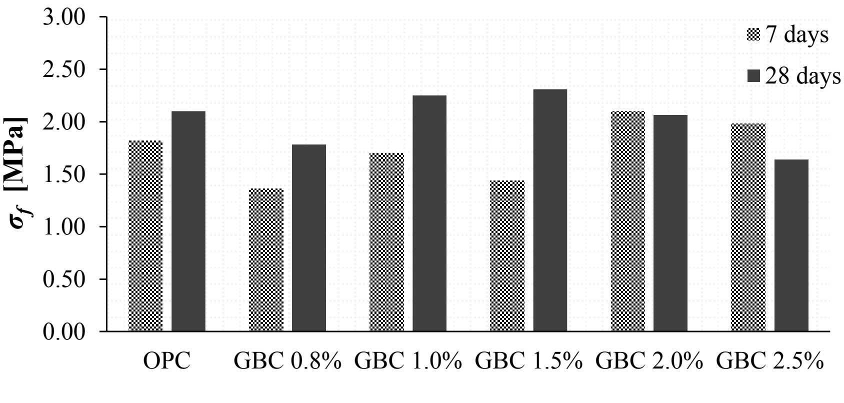

Fig. 2. Flexural strength [MPa] of cement pastes: average value for each batch, 7 and 28 days.

The results of fracture energy at 7 and 28 days do not follow the same trend of the bending strength. Results at 7 days show an increasing trend, the higher is the percentage of particles of biochar, the higher are the values of fracture energy (the composition with 2.5 wt% of biochar shows an increase up to 83% if compared to the pure cement sample). On the other hand, the 28-day results are difficult to interpret, because of fluctuations in values that can perhaps only be explained by the biochar's ability to interact with the hydration of the samples during the curing phase.

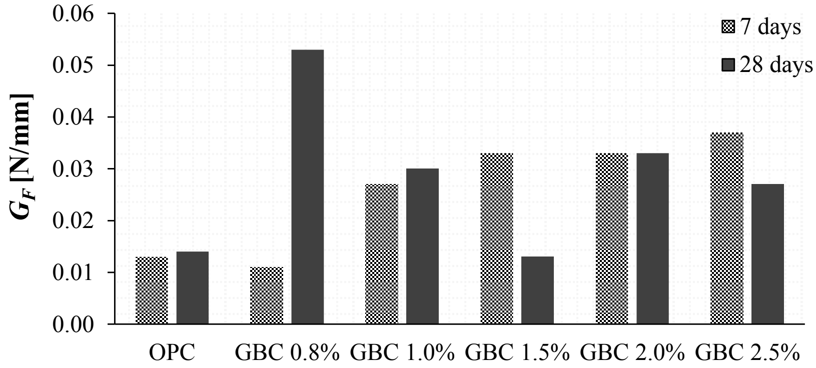

Fig. 3. Fracture energy [N/mm] of cement pastes: average value for each batch, 7 and 28 days.

4.2. Flexural strength and fracture energy of mortars

The medium values of flexural strength and fracture energy of each batch (composed of three specimens) are reported in Table 4, together with the corresponding standard deviations.

Table 4. Mechanical properties of the mortar batches

Batch	Flexural Strength σ_f [MPa]		Fracture Energy G_F [N/mm]	
	14 days	50 days	14 days	50 days
M 0%	4.21±0.33	5.37±0.26	0.078±0.005	0.089±0.003
M 1.0%	4.20±0.35	5.05±0.54	0.068±0.009	0.082±0.005
M 2.5%	4.90±0.39	5.37±0.49	0.091±0.010	0.091±0.005
N 0%	6.62±0.49	6.62±0.15	0.115±0.012	0.098±0.006
N 1.0%	7.04±0.80	7.20±0.12	0.111±0.007	0.109±0.015
N 2.5%	6.53±0.24	7.15±0.30	0.118±0.007	0.125±0.029

As regards mortar type M, the addition of 2.5 wt% of "Grey Borgotaro" biochar provides promising results not only in terms of strength but also in terms of toughness. While for 1 wt% both flexural strength and fracture energy decrease, an improvement is registered for 2.5 wt%, especially at 14 days curing, as shown in Figures 4 and 5. Biochar tends indeed to produce beneficial effects on early age development of mechanical properties, since it acts as accelerator that leads to early generation of more hydration products, as outlined e.g. by Gupta et al. (2018[a]). So, the obtained results suggest to consider for future research different percentages (and in particular higher) of biochar. However, it is worth noticing that 2.5 wt% is chosen as maximum percentage in this work, since higher biochar additions involve a considerable increase of water (or superplasticizer), in order to obtain good flowability.

As regards mortar type N, a different trend is observed with respect to mortar type M, probably due to how "Grey Borgotaro" biochar interacts with the type of cement and the w/c ratio. In this case, the addition of 1 wt% leads to an increase of flexural strength, while for 2.5 wt% only the results at 50 days are improved, as shown in Figure 4. The highest percentage of addition (2.5 wt%) seems the most promising in terms of fracture energy, even if the role of biochar on toughness for mortar type N is a little bit difficult to interpret, since the results at 14 and 50 days curing do not follow the same trend, as reported in Figure 5. This could be explained by considering that the values of fracture energy of control batch at 14 days present a high scatter, resulting in a mean value at 14 days quite greater than the corresponding one at 50 days.

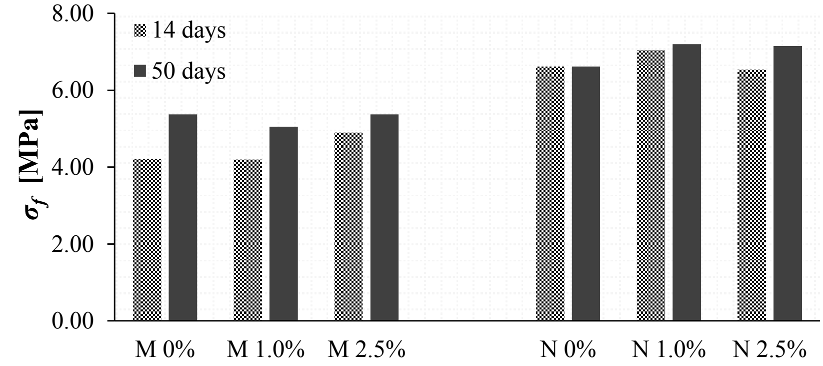

Fig. 4. Flexural strength [MPa] of mortars: average value for each batch, 14 and 50 days.

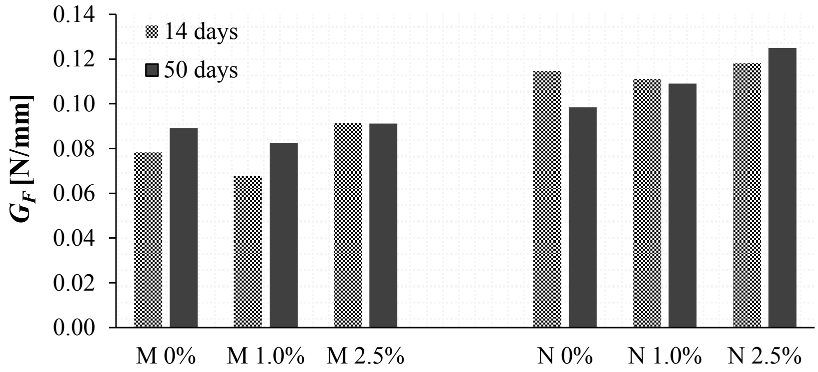

Fig. 5. Fracture energy [N/mm] of mortars : average value for each batch, 14 and 50 days.

5. Conclusions

Nowadays, the construction field demands materials with better performance and lower environmental impact. Concrete is the most used construction material in the world and, at the same time, it is responsible for a large portion of annual CO_2 emissions. This research work explored the possibility of using biochar in cementitious composites with different percentages of addition with respect to the weight of the cement, in order to improve the mechanical properties and reduce the carbon footprint of cementitious materials. In this study, the biochar used was obtained through a standardized process of gasification of wooden waste, allowing the development of high-performance cement pastes and mortars with low carbon footprint.

Based on the experimental results, it can be observed that the addition of biochar can increase the flexural strength and fracture energy of cementitious composites. However, in this study the percentage that generally led to the best results (2 wt% and 2.5 wt% for cement pastes and mortars, respectively) does not correspond to the percentages used in previous studies (Khushnood et al. 2016, Restuccia et al. 2017) and this could be related to the different production process (of biochar) and to the biomass source. Anyway, the results obtained show that the addition of biochar in the tested percentages provides a material with enhanced or comparable flexural strength and toughness with respect to plain specimens, so the tests can be considered quite positive and satisfactory and the objective of the research reached.

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