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The influence of biochar shape in cement-based materials

Daniel Suarez-Riera[1], Luca Lavagna[2,3]*, Mattia Bartoli[3,4], Mauro Giorcelli[2], Matteo Pavese[2,3], Alberto Tagliaferro[2,3]

[1]Department of Structural, Geotechnical and Building Engineering, Politecnico di Torino, C.so Duca degli Abruzzi 24, 10129 Torino, Italy

[2]Department of Applied Science and Technology, Politecnico di Torino, C.so Duca degli Abruzzi 24, Turin, 10129, Italy

[3]Consorzio Interuniversitario Nazionale per la Scienza e Tecnologia dei Materiali (INSTM), Via G. Giusti 9, Florence, 50121, Italy

[4]Center for Sustainable Future, Italian Institute of Technology, Via Livorno 60, Turin, 10144, Italy

*Corresponding author E-mail(s): luca.lavagna@polito.it; Tel.: (+39)0110904598 (L.L.).

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Abstract

Biochar-containing cement composites are gaining interest in the last years due to the possibility of reducing the environmental impact linked to cement. It can also improve the mechanical properties and the electrical conductivity of cement-based materials. A parameter that can affect mechanical performance of biochar-containing cement composites is the morphology of biochar. While it is reasonable to expect a different behavior for different shapes (rod-like, spherical or sheet), a full understanding is yet to be achieved of the competing effects of aspect ratio, morphology, and composition linked to the specific biochar source. This study shows that biochar can improve the mechanical properties of cement-based composites. 1 wt. % of biochar improves of 7 % the compression strength and 15 % the flexural strength. The results presented in this work confirm that biochar has a positive effect on both the mechanical and environmental behavior of cement-based materials.

Keywords: Biochar; Mechanical properties; Recycling; Cement-based composites.

1. Introduction

The European Union has been trying for years to find solutions for the problem of waste through specific regulations and by encouraging the reuse and recycling of materials. The problem is also addressed to a lesser extent by industries, specifically in the case of food, wood, and textiles production processes, where waste is problematic in the management and disposal phase. For example, nearly one-third of the foods produced for human intake (1.3 billion tons per year), are wasted (Gustavsson et al. 2011). Approximately 50 million cubic meters of wood waste are generated each year in the European Union (Bergeron 2016). Finally, a significant fraction of the post-consumer textile waste is not recycled but either disposed of in landfills or thermochemically treated for energy production (Lopatina et al. 2021).

Pyrolysis is a type of thermochemical treatment that is also a promising approach for converting biomass waste into energy in liquid form (bio-fuel) or gas (syngas) (Luque et al. 2011; Basu 2018; Tamborrino et al. 2021). The residual solid part of this type of treatment is biochar. Biochars can have different sizes, shapes and chemical characteristics, depending on the source material from which they derive and the production process used to obtained them (Cha et al. 2016).

The use of biochar in the construction field is a topic explored only in recent years. Some works show how the improvement of mechanical properties with biochar is comparable to other carbon-based materials like carbon nanotubes, carbon fibers and graphene (Musso et al. 2009; Lavagna et al. 2018; Sirico et al. 2020a; Lavagna et al. 2021a). In a study carried out by Gupta and Kua (Gupta and Kua 2018), biochar was added to the mortar mix trying to reduce the mixing water during initial stage of mortar hardening. Mortar has a relatively lower water-cement ratio thus leading to the formation of a compact pore structure with lower connectivity of pore network(Maljaee et al. 2021). Higher dosage of biochar in cement paste can cause a worsening of performance. A drastic reduction was observed in mortar with 5–8 % biochar (Gupta et al. 2018b). The same deleterious effect was observed in the fracture energy of concrete containing over 10% biochar (Cuthbertson et al. 2019). Sirico et al. (Sirico et al. 2020b) shown slight reduction of compressive strength at 7- and 28-days age after addition of 1 and 2.5% biochar from mixture of woodchips of local forests. In a study by Ahmad et al. (Ahmad et al. 2015), shown that a small content of bamboo biochar (0.08%) can enhance compressive strength and toughness of mortar matrix.The effect of biochar mixing procedure on the strength of cement paste was assessed in a study by Suarez-Riera et al. (Suarez-Riera et al. 2020) 2% of biochar added in the former state improved the flexural strength up to 15%. The use of these materials, however, involves an increase in the costs of production of the final cementitious material, as they have high production costs. By using biochar, on the other hand, production costs are reduced since biochar derives from waste materials. The environmental issue is positively tackled by giving added value to a recycled waste. Moreover, its use in cementitious matrices has already been explored in the literature, demonstrating that it can improve the mechanical properties of the matrix (Gupta et al. 2018a; Restuccia et al. 2020; Dixit et al. 2021). Additionally, depending on feedstock and preparation conditions, more than 60% of carbon from the parent biomass can be sequestered in biochar, leading to a net reduction in greenhouse gas emissions of 530–570 kg CO_2 per ton of dry feedstock (Roberts et al. 2010). The incorporation of bioderived filler into concrete a could represent a game change event for improving the sustainability of the building materials. The rational design of bioderived carbon filler represent a key issue that this work firstly addressed. However, to our knowledge, no investigations discuss the effect of the form factor and the feedstock of the biochar. Therefore, the present research studies the use of biochar as a nano/micro reinforcement in cement-based composite with different shape factors and different origins. The pyrolyzed material was added to the cement and the experimental specimens were tested to evaluate the flexural strength, compressive strength, and fracture energy. The objective of this study is therefore to understand the effect on the mechanical properties of the different biochars used, in particular regarding their shape.

2. Materials and Methods

2.1 Materials used

Cellulose nanocrystals were purchased from Alberta-Pacific Forest Industries (Batch COMP170823-H). Waste de-inked paper and waste cotton fibers were recovered from local suppliers.

The cement used in this study is type I Portland cement Ultracem 52.5 R purchased from Italcementi.

2.2 Production of biochar and cement-based composites

Biochars were produced through pyrolysis carried out in a vertical furnace processing 25 g of each feedstock separately. The reactor was sealed, and a flux of nitrogen (4 mL/min) was used as inert carrier gas. The heating rate was set at 50 °C/min up to the treatment temperature at 400 °C, with a dwelling time of 30 min followed by cooling down at room temperature. Carbonized cotton fibers (rod-shaped) and de-inked paper (sheet-shaped) were pulverized for 10 min using a mechanical blender prior the utilization as cement filler while cellulose nanocrystals (sphere-shaped) were used without further treatments.

First, the different biochar powders used in this study were dispersed in water with an ultrasonic tip (Vibra-cellTM) for 15 min at 100 W, then the water-biochar suspension was mechanical stirred while cement powder was slowly added to the continuously stirred solution. The water to cement ratio was fixed at 0.35 and 1% of superplasticizer (SP) was employed for the preparation of the mix. Next, the mix was poured into suitable molds 20 x 20 x 80 mm. After that, the cement composites were cured in water at room temperature for 28 days. Biochar content was fixed at 1% by weight of cement. Table 1 shows the mix design for every typology of sample prepared.

Table 1. Mix design of samples prepared.

Sample name	Water (g)	Cement (g)	SP (g)	Biochar (g)
Cement	80.5	230	2.3	0.0
Sheet	80.5	230	2.3	2.3
Sphere	80.5	230	2.3	2.3
Rods	80.5	230	2.3	2.3

2.3 Materials Characterization

Biochar's morphology was investigated using a Field Emission Scanning Electrical Microscope (FE-SEM, ZeisSupraTM 40). Raman spectroscopy was used to investigate the produced biochars in the range from 500 to 3500 cm^{-1} using a Renishaw® Ramanscope InVia (H43662) model equipped with

The mechanical properties of the cement composites were measured with a three-point bending test (ASTM C348) in crack mouth opening displacement (CMOD) mode following the standard JCI-S-001-2003. The specimens were tested using a single-column Zwick-Line z050 flexural testing machine with a load cell having a maximum capacity of 1 kN. As described in literature (Lavagna et al. 2021a), this measurement requires the samples to be notched in the middle, the notch having a width of 2 mm and a depth of 6 mm, equal to one fourth of the width of specimen. CMOD was controlled at a fixed displacement rate of 0.005 mm/min by placing a clip-gage extensometer on the two sides of the notch. All tests were performed at least on four specimens.

The flexural strength was calculated by using the standard formula for un-notched bending tests, using the thickness in the notched area. The fracture energy was calculated by integrating the load-displacement curve. The compressive strength tests were conducted on at least four cubic specimens per sample of size 20x20x20 mm with a force-controlled measurement at a fixed rate of 600 N/s.

3. Results and discussion

3.1 Biochar characterization

The use of different cellulose feedstock represents a suitable approach for templating different shapes of biochar (Bartoli et al. 2019, 2020). In this work, three different cellulose materials were used to produce sheet, sphere, and rod-like materials as shown in figure 1.

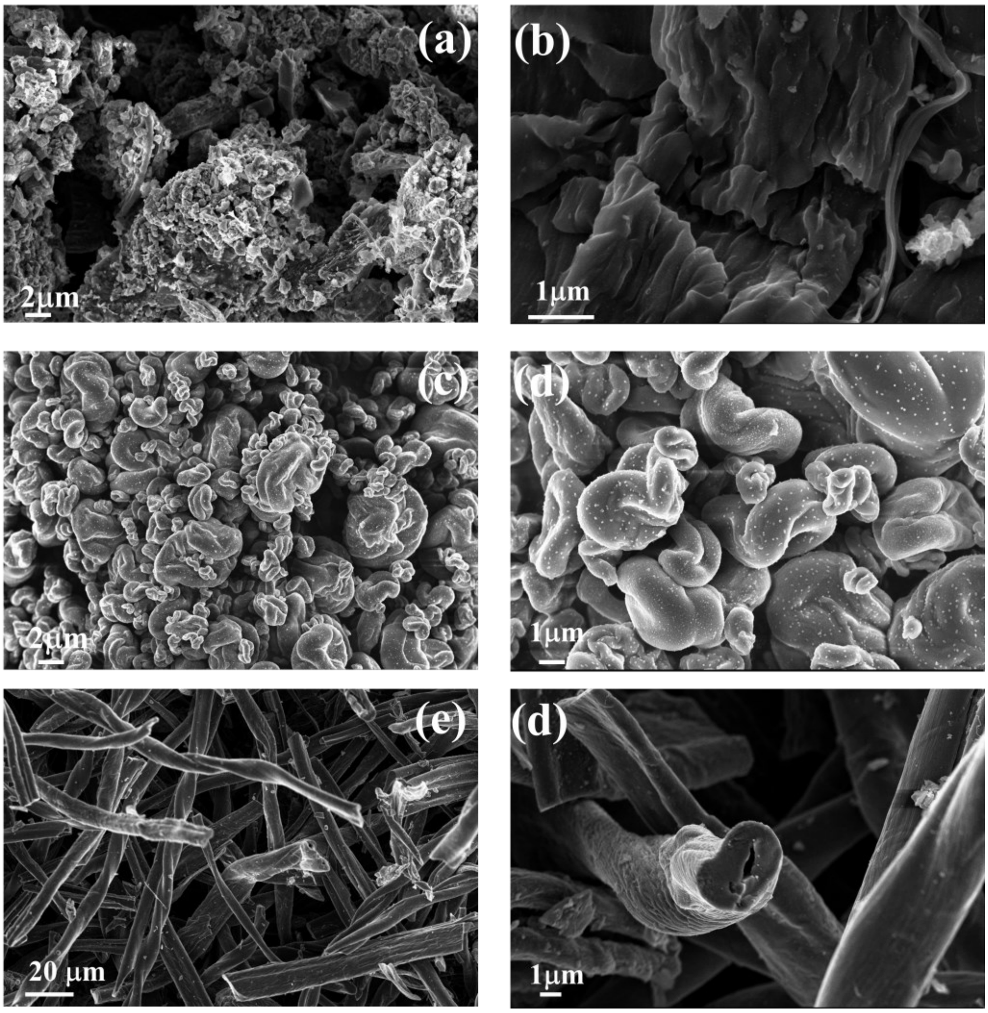

Figure 1: FESEM images of a-b) sheet, c-d) sphere and e-d) rod-shaped biochar produced by using de-inked paper, cellulose nanocrystals, and waste cotton fibers respectively.

Sheet-like materials showed a compact structure with tightly collapsed elements of micrometric size. These elements were composed by square-like substructures not greater than 1 μm (see figure 1b). Sphere-like biochar was composed of twisted particles with an average diameter ranging from 5 to 10 μm as shown in figure 1c-1d. Rod shaped biochar displayed a length of 10 to 20 μm and an average diameter of around 1 to 2 μm as reported in figure 1e-1f.

These materials were also investigated through Raman spectroscopy as reported in figure 2.

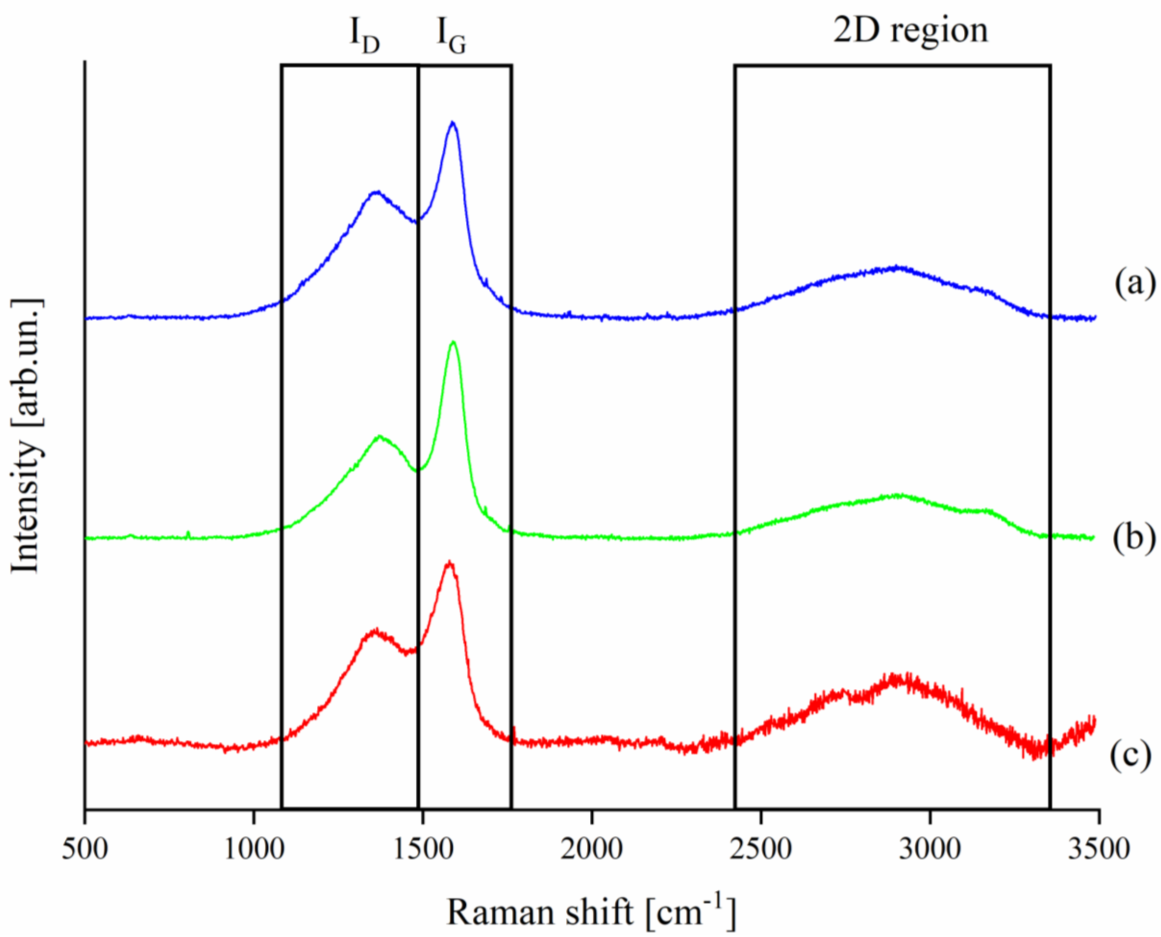

Figure 2: Raman spectra of a) sheets, b) spheres and c) rods shaped biochar produced by using de-inked paper, cellulose nanocrystals, and waste cotton fibers respectively.

All spectra are typical of highly disordered carbon with I_D/I_G ratio of 1.6 and 1.1 for sheets and rod biochars respectively while sphere shaped have an I_D/I_G ratio of 0.7. This last value is probably due to the small graphitic domains that were oriented by the multiple interactions of residuals oxygen based functions as reported by Bartoli et al. (Bartoli et al. 2019).

3.2 Development of mechanical properties

Table 2 Mechanical results

Sample name	Flexural strength (MPa)	Compression strength (MPa)	Fracture energy (N/mm)
Cement	2.2 ± 0.6	74.6 ± 13.9	0.036 ± 0.013
Sheet	2.4 ± 0.6	81.8 ± 2.6	0.044 ± 0.014
Sphere	2.7 ± 0.8	80.6 ± 8.2	0.042 ± 0.010
Rods	2.5 ± 0.4	80.2 ± 4.2	0.041 ± 0.002

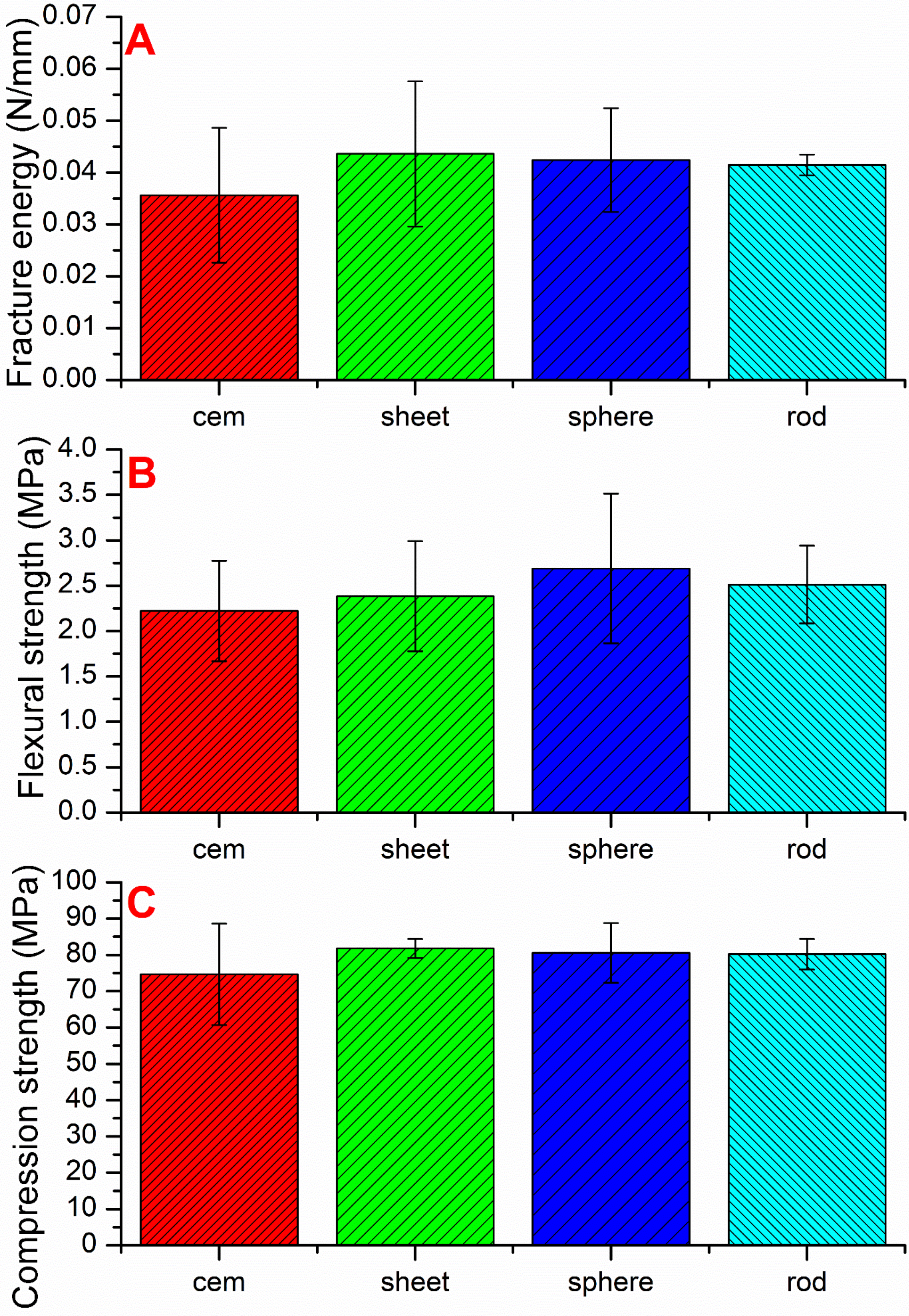

Figure 3: Mechanical properties of different shape biochar in cement-based composites

Figure 3a, 3b and 3c represent respectively the results in terms of flexural strength, fracture energy and compressive strength of cementitious composites with biochar used as a filler, at 28 days. It can be recognized that the batches in which biochar has been used show a mechanical performance improvement (compressive strength, flexural strength, and fracture energy) compared to the plain cementitious paste. In terms of compressive strength (Fig. 3c) and fracture energy (Fig. 3b), the addition of 1% biochar (regardless of its type and shape) lead to an average increase of more than 8% (from 74.6 MPa to more than 80 MPa) and almost 18% respectively at 28 days. In both scenarios the sheet-shaped biochar presents the best mechanical performance, specifically an improvement of around 10% and 24% in compressive and flexural strength (Fig. 3a) respectively was obtained, contrary to the case of the rods biochar that shows an enhancement of around 7% and 15% in terms of compressive and flexural strength, correspondingly. On the other hand, the flexural strength results do not follow the previous trend. In this case, the results give a different trend, in which the higher value of flexural strength enhancement was present in the spheres shape biochar samples by more than 20%, from 2.2 MPa to 2.7 MPa, and around 8% and 11% in the sheet and rods shape biochar samples correspondingly, compared to the pure cement batch.

3.3 Discussion

An explanation of this behavior can be found in the different shape and dimension of the biochars used. It is expected that both the sheet and rod-shaped biochars have a bridging effect similar to that of graphene (Hou et al. 2017), while a small size filler can saturate the micro porosities of the cement and at the same time improve the nucleation of hydrated cement phases, resulting in a more uniform porosity. Finally, the mechanical resistance of the filler phase must also be considered. Since biochar is obtained by a pyrolysis treatment, a porous material is expected (Bartoli et al. 2020), with limited strength. The difference in mechanical performance of the cement samples containing biochar with different shapes is indeed small. However, in all cases the mechanical properties are increased, suggesting that biochar in itself has a positive effect on cement strength, probably due to its small size that causes more homogeneous nucleation and pore-size reduction. This effect, that causes the reduction of the size of the largest flaw, is important for flexural strength, where in fact the sphere-shaped biochar presents the best results, due to its smaller size. Also the rod-shaped biochar presents a good flexural strength, due to its high shape factor, that allows a better bridging of the initial small cracks. The post-peak behavior, connected with the fracture energy, favors again the smaller size biochars, that improve the cement compactness and thus limit the crack propagation. The compressive strength follows the same trend of fracture energy, since compression tests provoke a complex stress state inside the samples, where cracks start to propagate before fracture.
Evaluating the results reported so far, it seems clear that adding 1 wt. % of biochar is suitable to guarantee a mechanical properties improvement. The effect of the biochar shape seems marginal, suggesting that the main strengthening effect is linked to the dispersion of a very small phase. This effect confirms the results obtained for graphene oxide, graphene nanoplatelets and other nano-size carbon-based materials (Lavagna et al. 2021b). When properly dispersed, these small size phases can improve nucleation, reduce capillary porosity, and thus improve the overall cement performance.

4. Conclusion

The environmental impact of construction materials is considerable, and it can be reduced either by improving the strength of construction materials or by limiting the quantity of cement used. This article explored the possibility of using 1% of three types of biochar with different morphologies as fillers in cement paste mixtures to improve their mechanical properties. This approach has a double benefit as it reduces the environmental impact and the costs of treating the large amounts of biomass generated each year. The pyrolysis of biomass can achieve a substantial reduction in environmental impact with respect to the standard incineration process of waste.

The biochars used were obtained through a standardized process of pyrolysis of cellulose nanocrystals, waste de-inked paper, and waste cotton fibers, resulting in biochar with different shapes: spherical for cellulose nanocrystals, platelets for paper, rods for cotton fibers. The mechanical test results showed that the addition of biochar increased all the mechanical properties: flexural strength, fracture energy and compressive strength. In particular, when spheroidal biochar was used, a flexural strength improvement was observed of more than 20% at 28 days.

The improvements of mechanical properties are only marginally related to the shape of the biochar, as the test results show. On the other hand, it is evident that the small amount of biochar used in the composites (1%) allows a significant improvement in mechanical properties. This is probably due to the small size of the biochar itself, that allows a reduction of porosity and improves the nucleation, generating a more homogeneous porosity, with a smaller size of the largest flaw.

The coupling of improved properties with the very low environmental impact of biochar suggests that biochar can be used as an addition in cement-based materials, with a positive effect both on their mechanical and environmental behavior.

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