Biochar-containing construction materials for electromagnetic shielding in the microwave frequency region: the importance of water content

Biochar-containing construction materials for electromagnetic shielding in the microwave frequency region: the importance of water content

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Abstract

Electromagnetic waves in the X-Band (8.2 – 12.4 GHz frequency) are used for radar, satellite communication, and wireless computer networks in some countries. Shielding allows to protect humans and electronic devices from harmful effects of these waves. Cement based composites containing conductive material can be used for this purpose, and pyrolyzed carbonaceous residues (biochars) are promising in this respect. Two different biochars originated from wood (CB) and sewage sludge (SSB) pyrolysis were used as fillers in cement-based composites. The electromagnetic shielding properties of these composites have been tested vs the type and amount of biochar added. The influence of water content arising from different curing and ageing in ambient conditions has been investigated for one set of samples. Results show that CB and SSB contain 74% and 30% of graphitic carbon, respectively. In the composites, SSB particles are bulky and scarcely dispersed, while CB particles are elongated and homogenously distributed. High values of Shielding Effectiveness (SE>20 decibel, dB) are achieved at 10 GHz frequency for the composites containing 18% of CB biochar. A large role of ageing was also discovered in sample B18: increasing wet curing increases the shielding effectiveness up to 29 dB, while increasing ageing in air decreases the shielding effectiveness values: after 10 weeks the measured value is about 15 dB. The evidence suggests that the amount of physically adsorbed water is responsible for this behavior, and it should be taken into account when dealing with cement based composites used for electromagnetic shielding.

Keywords: Electromagnetic shielding; Biochar; Cement-composites;

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1. Introduction

Exposure to electromagnetic waves may have harmful impacts on biological systems and affect human health. Non-ionising electromagnetic radiation is typically absorbed by molecules via vibrational motion, and therefore affects biological tissues by generating heat. Moreover, evidence was also reported of non-thermal effects due to microwave irradiation in microorganisms (Banik 2003) and, most importantly, of possible effects of electromagnetic field exposure on human central nerve system (Kim 2019). Besides, also sensitive electronic elements should sometimes be shielded from interference due to radiation emitted from other devices (telecommunication systems, computers, etc.). An effective way to shield both humans and devices from electromagnetic waves is to apply or embed shielding materials in edifices walls. Yet, as of today, electromagnetic shielding in buildings is still limited to niche sectors due to technical difficulties. In fact, shielding in buildings is traditionally achieved through metal sheets (Geetha 2009), that show excellent intrinsic electrical conductivity. Metallic sheets are overlapped on the building envelope, but suffer two main drawbacks, such as heavy weight and difficulty of producing structures in complicated forms. Indeed, the shielding from electromagnetic waves can be obtained via materials capable of absorbing, reflecting and refracting electromagnetic waves (Geetha 2009) (i.e. a material capable of interacting with the electromagnetic field, especially a good electric conductor). Since cement-based materials commonly used in buildings are not good electrical conductors, they lack this capability. To overcome this limitation, an option is to embed a carbonaceous conductive component into the building material, either in the form of a powder (Cao and Chung

2003) or as a mesh like carbon nanotubes (CNTs) (Singh 2013). Moreover, previous studies already addressed the use of steel fibers blended into the cement matrix. Wen and Chung (2004) analyzed the addition of 0.36 vol.% of stainless steel fibers (8 μm of diameter and 6 mm of length) reaching, at 1.5 GHz, a SE of 58 dB increased up to 70 dB in the case of 0.72 vol% of fiber content.

Other authors demonstrated that using 1 vol% of polyacrylonitrile (PAN) carbon fibers it was possible to obtain SE values equal to 30 dB at 1 GHz (Muthusamy and Chung 2010) while 0.4% by weight of cement yielded 19.2 dB (Li et al. 2008).

Furthermore, Mazzoli et al. (2018) highlighted the possibility to improve the shielding performances combining a steel fiber content of 2.0% by weight of cement with 10% of graphene, observing 40 dB of SE in a frequency ranged from 1.8 to 8.0 GHz.

The potential application of the fibers has been also investigated in combination with Fe_3O_4 nanoparticles as done by Liu et al. (2017), that incorporated the iron oxide nanoparticles with 0.4% of carbon fibers by weight of cement, with a resulted SE value of 29.8 dB. The same percentage was then tested by Chen et al. (2015) that, investigating the use of graphene oxide-deposited carbon fibers, measured a SE value of 34 dB in the frequencies between 8.2 and 12.4 GHz.

Nonetheless, their high cost and difficult dispersion in cement-matrix (Nam 2012) restrict their wide use. In search for a lightweight (respect to metals) and cost-effective (respect to graphene and carbon nanotubes) material, capable of electromagnetic shielding in buildings, biochar popped out as a viable option.

Biochar (abbreviation for bio-charcoal) is a carbon-rich material produced through pyrolysis of many kind of biomasses, and its applications are already rapidly spreading in farming (Maroušek 2017), for soil improvement (Amin 2016), to increase water retention in soil (Ahmad 2014) and fertility of agricultural land, as adsorbent for aqueous heavy metals, and as fuel material when it has low ashes content (Tan 2015). The selection of the adequate biomass source and utilisation should also take into account economic and environmental assessments (Fan et al. 2019), while an extensive assessment of the production of green energy can be found in (Kravanja et al. 2015).

Among other sources, biochar can be obtained also from sewage sludge (Agrafioti et al. 2013). Sewage sludge is a side-product in wastewater treatment, which may be responsible up to 50% of total operational costs and to approximately to 40% of the total greenhouse gas emissions of a wastewater treatment plant (Capodaglio 2017). Wastewater treatment is one of the key areas regarding cleaner production as illustrated in (Varbanov et al. 2021).

In Italy, the disposal of sewage sludge is also a severe problem, and its dispersion on agricultural land is strictly regulated (Italian Parliament, Legge 130 - 16 Nov 2018). Therefore, applications of pyrolyzed sewage sludge in construction materials could transform a waste in a value-added product. Its possible use in construction materials has already been proposed, either as a means for disposal, in stabilization/solidification processes (Malliou 2007) or to exploit its pozzolanic properties (de Azevedo Basto 2019). Respect to application in cement-based composites, the thermochemical conversion process (pyrolysis) of carbonaceous materials enhances the chemical stability of biochar by eliminating labile chemical groups (Ghani 2013) and the primary sources of sugars, that are detrimental to cement hydration. Its high porosity introduces additional pore surface area in the cement paste, which regulates effective water-content, provides internal curing and sites for precipitation of hydration products, accelerating cement hydration and promoting early strength development (Gupta 2019).

As of today, studies on the use of biochar in cement-matrix composites as electromagnetic shielding aid in the microwave region are limited, although the conductivity of pure biochar is sufficiently high (0.1-0.2 Siemens per meter, S/m) to make it an interesting filler for this purpose (Geetha 2009). The effectiveness of the filler in shielding electromagnetic waves is measured as Shielding Effectiveness (SE), that is the ratio of the strength of the electromagnetic field before and after the attenuation (see section 2.3). Khushnood and coauthors (Khushnood 2015) analytically evaluated using permittivity measurements the theoretical shielding effectiveness of biochar-containing cement. They found that by addition of only 0.5 % by weight of biochar the SE of a cement sample of 10mm thickness could be increased by about 6dB at 10GHz. Savi et al. (2019) directly measured the shielding effectiveness of a biochar-cement composite containing 10% biochar by weight of cement, obtaining a promising value of SE of 15 dB, and reported it was due to an absorption-type shielding mechanism.

Considering that the use of this biochar-cement composite is intended for plastering building walls and ceilings, cost and sustainability aspects are essential for a viable development in the construction sector.

The application on wide building areas makes steel fibers, carbon fibers or carbon nanotubes not suitable for this scope.

In light of what we have shown up to this point, it is worth investigating how various biochar concentrations can affect the shielding effectiveness of a biochar-cement composite, since this further analysis is currently ongoing. Moreover, if we take into account sustainability aspects, an assessment on the shielding effectiveness of composites filled with sewage sludge biochar should be addressed in order to understand if this can be a viable option when compared with commercial biochar.

Another aspect that can be considered relevant for a correct evaluation of the shielding effectiveness of a cement composite is the presence of embedded water. Indeed, water can affect the electrical conductivity of a material in a significant way.

Having considered the aforementioned reasons, this paper aims at exploring the electromagnetic shielding properties of cement pastes containing 0%, 12% and 18% respect to cement of two different types of biochar: a commercial one and a sewage sludge residue. The shielding effectiveness of the composites were tested in the X-Band region of the microwave frequency, between 8.2 and 12.4 GHz. Moreover, the influence of curing and ageing conditions on the development of shielding effectiveness was investigated. Indeed, it is worth noting that it was reported that hydration can affect the measured SE of cementitious materials in the early stages; specifically, Prasad and coauthors (Prasad 2019) found increasing SE values as hydration evolved in the early stages of cement setting, and this was accounted for the progress of the density and compactness of the manufact. To the best of our knowledge, this kind of investigation is lacking when dealing with biochar-containing cement pastes, and on longer timespans. One can expect that - being water molecules absorbers in the microwave region - free water molecules in the cement matrix can influence the shielding effectiveness of the composites. Curing and ageing conditions of course can influence the water content of the samples, that is in turn expected to influence the shielding effectiveness. Electromagnetic shielding performance of biochar-containing cement composites reveals indeed a high dependency on ageing history of the samples, which highlights the primary role of physically adsorbed water.

2. Experimental procedures

2.1. Specimens manufacturing

Commercial biochar (CB, wood pyrolysis) was supplied by Carlo Erba, while sewage sludge biochar (SSB) was obtained from a sewage sludge residue pyrolysis. Both were used in form of powder. CB biochar originated from wood pyrolysis and its particles show mainly needle-like elongated shapes of few microns thickness and tenths of micrometers long, while SSB biochar is mainly constituted by big lumps with roughly circular shape and radiuses of few tenths of micrometers (See Figure 4 in section 3.2). Both biochars were thermally treated (reactivation process) before use, at 750 °C for 4h in a packed and full alumina crucible, to avoid combustion. A set of CB-cement composites using mix formulations with 12% (B12) and 18% (B18) by weight of ordinary Portland Cement (PC) were prepared. For the fabrication of SSB-cement composites, only 18% (S18) by weight of cement was used. In both cases Portland cement grade 52.5 R compliant with ASTM C150 (2012) was used. Water and superplasticizer (necessary for water reduction and better workability) were added with percentages of 60% and 1.8% by weight of cement, respectively. Biochars were mixed with cement and superplasticizer using a mechanical mixer, for 5 minutes. Then, each homogeneous mixture was poured into rectangular moulds, shaped to suit the requirement of the measurements of the scattering parameters in a rectangular waveguide and, consequently, the evaluation of the shielding effectiveness. Finally, after keeping all the specimens at 90 ± 5 % relative humidity for the initial 24 hours, they were demoulded and immersed in water at room temperature (20 ± 2 °C) for 1 week. Afterwards, samples were kept in air at ambient conditions. Additional specimens of B18 were prepared and cured in water for 2 and 4 weeks in order to investigate the influence of the wet curing time on the shielding properties of the composites. Furthermore, a reference set (CEM) was prepared using only cement, water and superplasticizer, 35% and 1.5% by weight of PC, respectively. A flowchart of the sample preparation process is reported in Fig. 1.

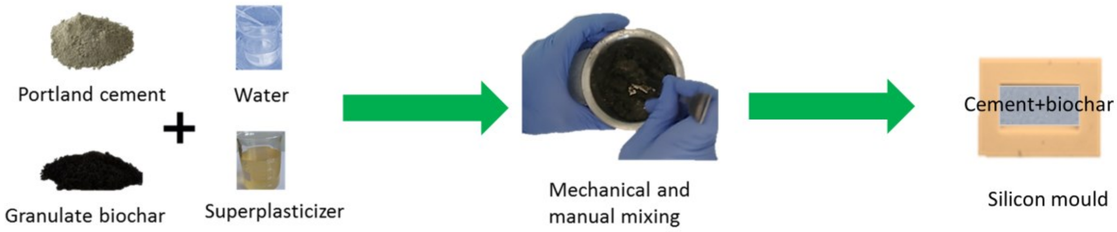

Fig. 1: Flow chart of the sample preparation procedure.

2.2. Materials Characterization

X-ray powder diffraction patterns for CB and SSB biochars were collected at room temperature using a Bruker D8 diffractometer in θ-2θ arrangement (Cu-kα radiation), equipped with a Ni filter and a gas proportional scintillation detector. Measurements were performed in the 2θ range 5-70°, with steps of 0.02° 2θ and counting time of 10 s/step. Phase analysis was performed with the DIFFRAC.EVA software (Bruker AXS) using the International Center for Diffraction Data - Powder Diffraction Files Database, year 2019 (ICDD-PDF 2019).

Thermogravimetric analyses (TGA) were performed in air up to 950°C with a temperature ramp of 3 °C/min, on about 10 mg of biochar sample, by a simultaneous TG-DTG Seiko 6300 instrument.

Cross sections of cement-biochar composites were analyzed using a Hitachi S-2500C Scanning Electron Microscope (SEM). Fresh cross-section surfaces were cut from each specimen, polished and gold coated before measurement to prevent charging effects.

2.3 Electromagnetic shielding measurements

The shielding effectiveness (SE) was investigated in X-band (8.2-12.4 GHz), measuring the scattering parameters in a rectangular waveguide (cross section 22.86 x 10.16 mm). Concerning this, it must be noted that the important point is to measure samples of the same thickness, in order to compare the results. Obviously, the higher is the thickness the better is the shielding. A waveguide spacer of 5 mm thickness is used as sample holder and the sample thickness was 4 mm. An Agilent E8361A network analyser is used to measure the scattering parameters and a Maury Microwave X7005E calibration kit is used to perform a standard short-thru-matched load calibration. Three samples for each composition are measured to ensure the reproducibility of the results. The SE can be defined for the electric field in decibel as:

$$SE_{dB} = 20\ Log \left|\frac{Ei}{E_t}\right| \qquad (1)$$

where Ei is the incident electric field and Et is the transmitted field (Paul 2006). It can be obtained from the measured transmission coefficient (S_{21}) in the rectangular waveguide as:

$$SE_{dB} = -20\ Log|S_{21}| \qquad (2)$$

3. Results and discussion

3.1 Biochar Characterization

In Figure 2 the X-Ray Diffraction patterns of biochars are reported. As shown in Fig. 2-A the XRD pattern of CB shows a broad background peak at ~23 °2θ, indicating a large amorphous component. Crystalline peaks are identified as calcite ($CaCO_3$, PDF # 01-071-3699). Traces of residual lignite (PDF # 00-0005-0625) are also detected, consistent with the origin of the biochar (Mohanty 2013). In SSB diffraction pattern (Fig. 2-B) the amorphous content is less noticeable; the crystalline components are identified as calcite ($CaCO_3$, PDF # 01-071-3699), quartz (SiO_2, PDF # 00-046-1045), anorthite ($CaAl_2Si_2O_8$, PDF # 04-011-1371) and an iron phosphate ($Fe_3(PO_4)_2$, PDF # 04-016-0295). This latter is not surprising as SSB biochar is derived through pyrolysis of a sewage sludge and can often contain iron sources (de Azevedo Basto 2019).

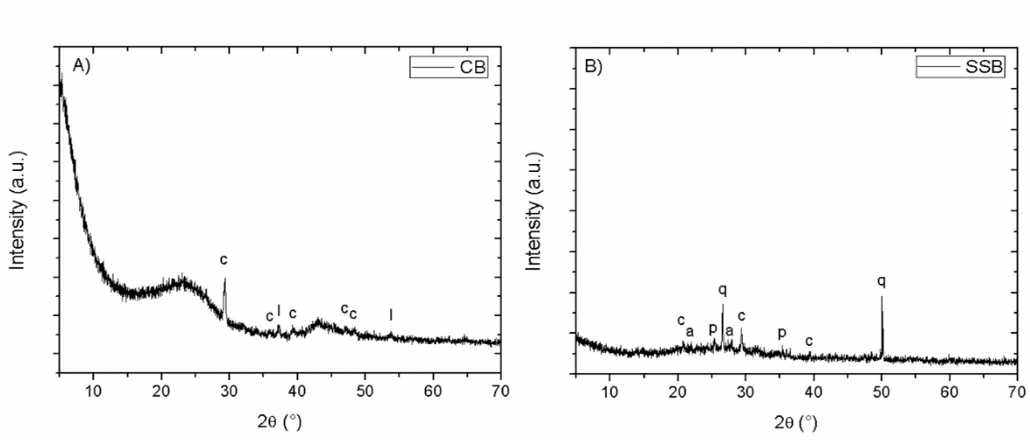

Fig. 2: XRD patterns of CB (panel A) and SSB (panel B) Biochar samples. c = calcite, l = lignite, q = quartz, p = iron phosphate, a = anorthite.

The initial water and carbon content in the two biochars were then investigated in TGA experiments (Fig. 3). Two main weight losses can be identified: a low temperature loss (below 100 °C) due to the evaporation of the physically adsorbed water, and a high temperature loss (ca. 400-450 °C) where combustion of the graphitic carbon fraction occurs. A significant difference is found in the two biochars: in CB sample (Fig. 3-A) the water content is about 16%, much higher than in SSB biochar (4%). The reasons of different water uptake in biochar is usually related to different surface areas or different number of surface oxygenated groups (Behazin 2016). By the way, a significant discrepancy is observed between CB and SSB carbon content, which is about 74% by weight in CB and only about 30% in SSB. No other relevant losses can be identified in both samples. At 950 °C, a residue as low as 5% in weight is observed in CB sample, while it is more than 60% in SSB. These results suggest that the actual graphitic carbon content is much higher in CB that in SSB. Likely, SSB contains a large amount of ashes materials, as also hinted by the presence of more crystalline phases in the XRD pattern of SSB.

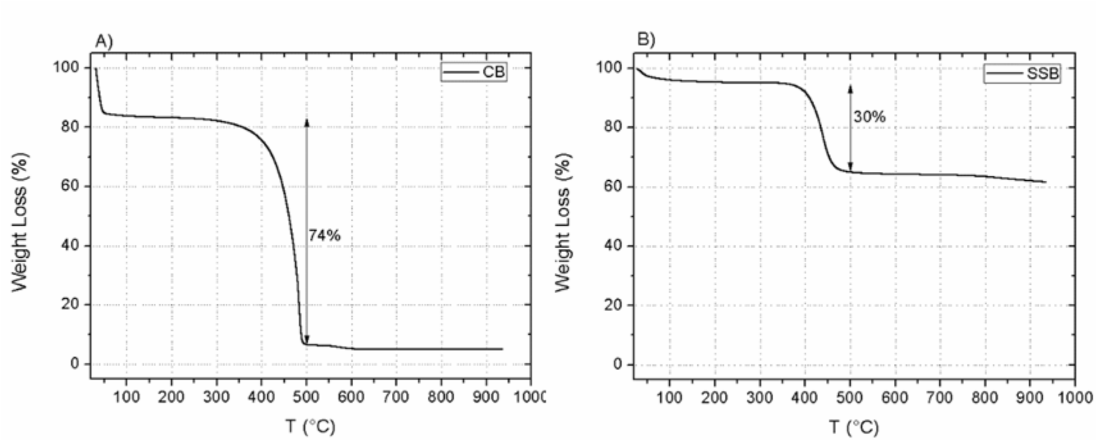

Fig. 3: Thermogravimetric curves of biochars: CB (Panel A) and SSB (Panel B)

3.2 Biochar-containing cement-composites

The composition of the biochar-containing cement-composites is given in Table 1.

Table 1: Mixture proportions* in cement-biochar composites as wt% of Portland Cement.

Samples	w/cem wt.%	sp/cem wt.%	b/cem wt.%	C/cem wt.%	a/cem wt.%	C/s wt.%
S18	60	1.8	18	5-6	11	5

B12	60	1.8	12	9	0.6	8
B18	60	1.8	18	13	0.9	11-12

* w=water, cem=cement, b=biochar, C=graphitic carbon, a=ashes, s=solids, sp=superplasticizer. ‘solids’ are the sum of cement, graphitic carbon and ashes.

SEM micrographs of composites are reported in Fig. 4. The carbonaceous particles (in black in the micrographs) of samples B12 and B18 look well dispersed in the matrix and well embedded. The shape of CB particles is mostly elongated, with a large dispersion of sizes, starting from a few µm and up to 100 µm in length, characteristic of fiber (wood) derived biochar. Comparing B18 and S18 samples, the latter clearly shows fewer carbonaceous particles. This observation well agrees with the TGA and XRD measurements, which revealed a high amount of ash material in the SSB biochar that are difficult to identify in micrographs. The shape of carbon particles is irregular and the diameters of SSB agglomerates range from a few micrometers to hundreds of micrometers, most of them being particularly bulky. The resulting picture is that in sample S18 there are less particles and unevenly distributed.

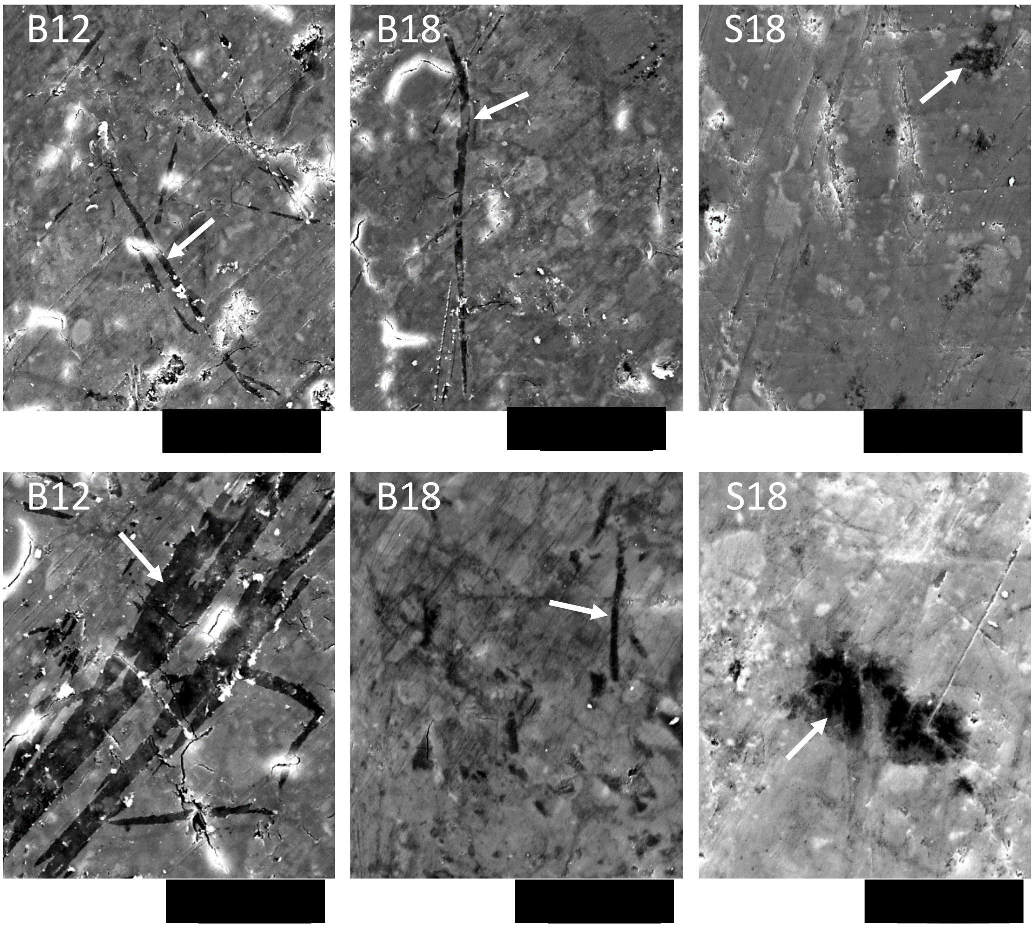

Fig. 4: SEM micrographs of the cross section of cement composites containing CB biochar (B12 and B18) and SSB biochar (S18). Images are taken at 1000x (above) and 1500x (below) Some biochar particles are labeled with a white arrow.

3.3 Shielding Effectiveness analysis

The shielding effectiveness is calculated from the measured transmission coefficients of samples B12, B18 and S18 according to equation (1) and it is illustrated in Fig. 5. All the samples show an increase of the shielding effectiveness value with respect to the plain cement sample (CEM). Comparing the composites with commercial biochar (CB) filled with 12% and 18%, an increase of about 5 dB of the shielding effectiveness with the increase of the percentage of the filler can be observed in the whole frequency band. Comparing the samples with 18% filler of sewage sludge biochar (S18) and the sample with commercial biochar (B18), it can be observed that B18 exhibits the highest SE with almost 20 dB at 8 GHz and 23 dB at 12 GHz. This is likely due to the higher effective carbon content in B18 compared to S18. The thermo-gravimetric analysis (Section 3.2) indicates that the actual content of carbon in CB is 74 wt.%, whereas the content of carbon in SSB is only 30 wt.%. This means that the actual content of carbon in the composites is about 5% for S18 and 11-12% for B18 (8% for B12).

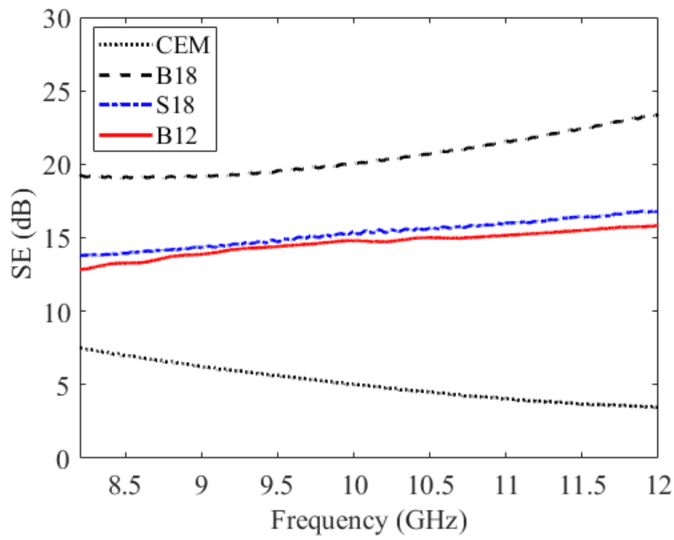

Fig. 5: SE values of CEM, and composites B12, B18, S18 after 1 week of curing time.

On the best performing composite (B18), a study of the influence of curing and ageing conditions on the shielding effectiveness was conducted; the investigated samples are reported in Table 2.

Table 2: Shielding Effectiveness (SE) values at 10 GHz for B18 composites cured in different conditions.

Samples	wet curing time (weeks)	ageing in ambient conditions (weeks)	SE (dB)
B18	1	0	20
B18	1	2	19
B18	1	10	15
B18_2	2	0	23
B18_2	2	2	20
B18_2	2	10	15
B18_4	4	0	29
B18_4_dried	4	0	13

The SE values measured at 10 GHz for these cases are reported in Fig. 6. Wet curing time is one week for sample B18, two weeks for sample B18_2 and 4 weeks for sample B18_4. These samples were measured after 1 day (0 week), 2 weeks and 10 weeks in ambient conditions, or after drying in oven at 80 °C (sample B18_4_dried). The temperature was chosen so to not affect the common cement hydration products (i.e. calcium silicate hydrate and calcium aluminate hydrate gels) that can be dehydrated only at higher temperatures in the range 115 -150 °C (Sharma 2012). Therefore, we expect that only free water should be affected.

Wet curing period and ageing affect the shielding effectiveness. SE increases with wet curing time and decreases with ageing time in ambient conditions and with drying in oven. Both cement hydration reactions and oven treatment decrease the amount of free water in the manufact, the first by using water in chemical reactions, the second by evaporation. It seems that physically adsorbed water must play an important role in attenuating the electromagnetic waves at 10 GHz. Physically adsorbed water may have two sources: i) water retained by the biochar and ii) evaporable water in the hardened PC paste. Among other positive aspects concerning the adding of biochar into soil (Maroušek et al. 2019), it has also been reported that the water retention capacity in the biochar modified soil is significantly enhanced (Karhu et al. 2011). Both explanations have been suggested for the enhanced water retention capacity: high amount of small pores in the biochar (Major 2009) and the negative electric potential (zeta potential) values when the pH of the medium is > 5, due to the adsorption of hydrated ions at the biochar surface (Batista et al. 2018). It is worth noting that the hardened PC paste is an alkaline medium, contributing to the water retention capacity.

The other source associated with the adsorbed water is the evaporable water, which resides partially in the capillary pores and partially in the gel pores within the cement hydration products. The high porosity of biochar provides additional pore surface in the cement matrix, which controls real water content. Hence, care should be taken when testing unaged biochar-containing cement composites (or with any filler/substrate adsorbing water), as electromagnetic shielding effect of the filler and that of (temporarily) adsorbed water cannot be isolated.

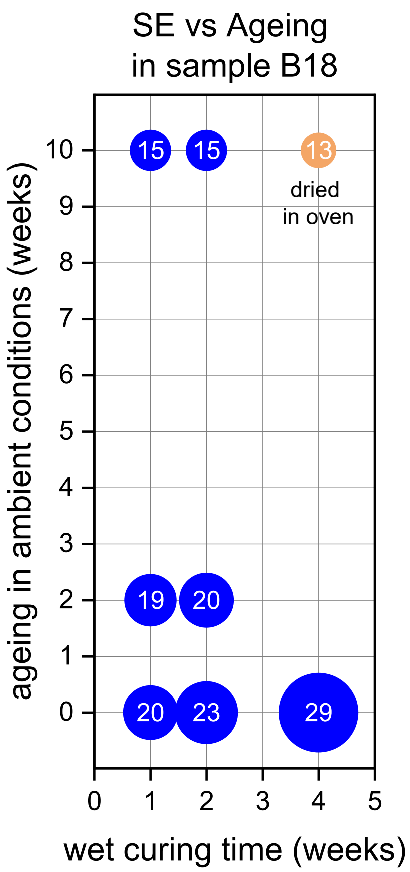

Fig. 6: SE at 10 GHz of B18 sample vs ageing. Bubble size is proportional to SE value in dB. *in orange, sample dried in oven at 80 °C for 1 hour.

4. Conclusions

Commercial wood biochar (CB) and biochar obtained from sewage sludge (SSB) are analyzed and used as filler in cement composites to enhance electromagnetic shielding properties. The biochar obtained through pyrolysis of sewage sludge (SSB) contained 30% of graphitic carbon, while CB contained 74 wt.%. The particles shape is also different: bulky and scarcely dispersed in the cement paste for SSB, elongated and homogenously distributed for CB. The shielding effectiveness of various composites is evaluated from the scattering parameters measurements in a rectangular waveguide in the frequency band 8.2-12.4 GHz. The key findings can be summarized as follows:

1. The addition of biochar always enhances the shielding effectiveness of the composites.
2. Wet curing time and ageing of the B18 sample up to 10 weeks in ambient conditions show that these parameters influence the SE values, that vary from 29 dB down to about 13 dB at varying conditions. These effects are accounted for physically adsorbed water in the samples.
3. Since electromagnetic shielding values are strongly affected by the presence of water, measurements of this property should be performed on samples after an appropriate ageing process, in order to avoid misleading interpretations. For example, if such a measurement is taken only a few days after the wet curing time, an overestimated value of shielding effectiveness will likely be obtained, causing an error in all the subsequent evaluations.

Having said that, the use of cementitious construction materials added with a small amount of biochar can pave the way to the use of low-cost materials for electromagnetic shielding. Construction industry, always resistant in accepting new materials and technologies, can indeed be the ideal sector where such experimentation can take place. This material would in fact slightly differ from the original, making it less difficult to be accepted for that peculiar scope, where currently it is necessary to adopt more invasive technologies to obtain similar benefits.

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