

Shielding Properties of Cement Composites Filled with Commercial Biochar

Original

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1 Article

2 Shielding Properties of Cement Composites Filled 3 with Commercial Biochar

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9 **Abstract:** The partial substitution of non-renewable materials in cementitious composites with eco-
10 friendly materials is promising not only in terms of cost reduction, but also in improving the
11 composites shielding properties. The water and carbon content of a commercial lignin-based biochar
12 is analyzed with thermal gravimetric analysis. Cementitious composites samples of lignin-based
13 biochar with 14wt.% and 18wt.% are realized. Good dispersion of the filler in the composites are
14 observed by SEM analysis. The samples are fabricated in order to fit in a rectangular waveguide for
15 measurements of the shielding effectiveness in X-band. A shielding effectiveness of 15dB was
16 obtained at a frequency of 10GHz in the case of composites with 18wt.% of biochar. Full-wave
17 simulations are performed by fitting the measured shielding effectiveness to the simulated shielding
18 effectiveness by varying material properties in the simulator. Analysis of the dimensional tolerances
19 and thickness of the samples is performed by the help of full/wave simulations. Lignin-based
20 biochar is a good candidate for partial substitution of cement in cementitious composites as the
21 shielding effectiveness of the composites increase substantially.

22 **Keywords:** shielding effectiveness; biochar; eco-friendly material; cementitious composites;
23 waveguides.
24

25 1. Introduction

26 The human population has seen rapid growth in the past decades. With increasing population,
27 the demand for construction industry has increased manifold [1]. This has resulted in increasing
28 greenhouse gas emissions from cement production [2]. The substitution of non-renewable raw
29 materials used in construction industry with eco-friendly materials derived from waste is promising
30 in terms of cost and environmental protection [3]. Agriculture and forestry waste is primarily burnt on
31 field in order to reduce the cost of disposal. When converted into biochar, this waste can be used as a
32 partial substitute to cement resulting in a significant reduction in greenhouse gas emissions and
33 improving the mechanical properties of concrete [4,5].

34 Increasing number of devices working at microwave and millimetre wave frequencies has
35 resulted in an overall increase in electromagnetic radiation [6,7]. Electromagnetic shields are deployed
36 to protect sensitive devices against electromagnetic interference [8,9]. In places that are vulnerable to
37 electromagnetic interference, shielding materials can be applied as a coating on wall surfaces [10]. A
38 number of equipment working at microwave and millimetre wave is used in the health sector for
39 applications like imaging, tomography etc. [11,12]. The X-band is particular is important for radar
40 communications including air-traffic control, weather monitoring, maritime vessel traffic control,
41 defence tracking, vehicle speed detection. The use of shielding materials in building can be helpful in
42 isolating equipment that is sensitive to electromagnetic interferences [13,14]. Different measurement
43 techniques can be deployed for the determination of shielding effectiveness of materials. The most
44 common measurement techniques are reverberation chamber [15], free-space measurements in

45 anechoic chamber [16], coaxial and waveguide methods [17-19]. Each measurement technique requires
46 specific samples dimensions and frequency band. The X-band is very important for applications like,
47 satellite communications and radar.

48

49 The use of carbon based materials in epoxy composites and the analysis of their morphological
50 and electrical properties has been vastly studied [20-23]. Conventional carbon based materials like
51 graphene and carbon nanotubes are expensive and require a complex synthesis. In recent years, the
52 use of biochar substituting carbon nanotubes and graphene in composites as filler is investigated [24-
53 25]. Biochar is cost effective as compared to other carbon based materials. Biochar is a porous
54 carbonaceous material produced by thermal treatment of biomass in absence of oxygen [26]. It can be
55 made from a number of different waste products such as agricultural, food waste or sewage sludge
56 [27]. Until recently biochar has been used for soil amendment in agriculture and landfilling
57 applications [28]. The use of biochar in alternative applications is being studied at a vast scale,
58 specifically for carbon sequestration, energy storage applications [29] and in construction and building
59 [30-31].

60

61 In this paper, lignin-based commercial biochar is used as a partial substitute to cement in
62 composites. The water, carbon and other residues of the biochar is studied by TGA. Composites of
63 4mm thickness with plain cement, 14 wt.% biochar and 18 wt.% biochar are fabricated with specific
64 dimensions for measurements of the shielding effectiveness inside a waveguide working in the X-
65 band microwave frequency. The sample with 18 wt.% biochar were cured in water for 7 days or 28
66 days. For examining the microstructural properties of the composites and dispersion of the filler in the
67 composite matrix, SEM is adopted. Measurements of the shielding effectiveness are compared with
68 simulated results obtained with a full-wave simulator. As expected the shielding effectiveness
69 increases with the increase of the percentage of filler (11dB for 14wt.%, and 15dB for 18wt.% at 10GHz).
70 Analysis of fabrication tolerances and sample thickness are performed by the help of a full-wave
71 simulator.

72

73 Finally, the effect of the curing period in water on the shielding effectiveness values is analysed
74 for the samples with 18wt.% biochar. The shielding effectiveness increases by approximately 5dB in
75 the whole frequency range for the sample cured in water for 28 days with respect to the sample cured
76 in water for 7 days.

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76 2. Materials and Methods

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2.1. Composites preparation

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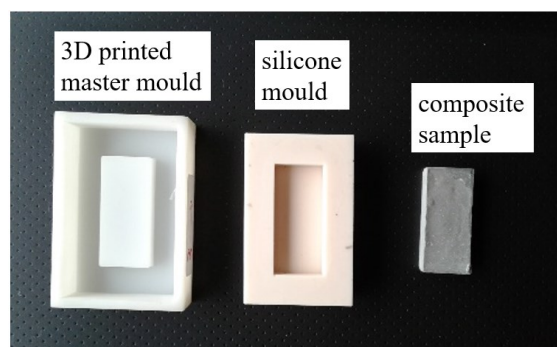
79 The composite samples produced are with 14wt. % and 18 wt.% of biochar in Portland cement. For the
80 sake of comparison, a composite without biochar is also produced, which is referred to as plain cement
81 composite. The biochar used to realize the samples is a commercial product provided by Carlo Erba
82 Reagents. It is pyrolysed in the form of powder at a temperature of 750 °C for four hours in an alumina
83 crucible. For preparation of cementitious composites ordinary Portland Cement (PC) (grade 52.5 R)
84 compliant with ASTM C150 is used along with water and superplasticizer to form an adequate
85 consistency of the paste. The percentages of water and superplasticizer used are equal to 60 wt.% and
86 1.8 wt.% respectively. A mechanical mixer is used to work the mixture for a duration of 5 minutes.
87 Silicon moulds of adequate shape and size are then used to give the composites the required shape
88 and dimensions.

88

89 Portland cement is blended with biochar by using a mechanical mixer for 5 minutes with two different
90 percentages by weight of cement, 14% and 18%, water (60%) and superplasticizer (1.8%). Furthermore,
91 a reference specimen is realized using only Portland cement matrix blended together with a water and
92 superplasticizer equal to 35% and 1.5%. The obtained composite are then poured into rectangular
silicone moulds for shielding effectiveness analysis. The silicon moulds are fabricated in a 3D printed

93 master mould of specific dimensions (see Figure 1). The reusable and flexible silicone moulds helps in
 94 easy extraction of composite samples once they are cured.

95



96

97 **Figure 1.** 3D printed master mould with silicone mould and an example of composites.

98 Initially, the composite samples are kept at a relative humidity of $90 \pm 5\%$ for 24 hours. The composites
 99 are then demoulded and immersed in water at a temperature of $20 \pm 2^\circ \text{C}$. The samples are then cured
 100 in water for a period of 7 days. Two different curing methodologies are used for curing of the 18wt.%
 101 samples in water for 7 days and 28 days in order to evaluate the impact of water curing duration on
 102 the shielding effectiveness (see Table 1). In Table 1 the different steps of fabrication and measurements
 103 of the cement composites are reported.

104

Table 1. Fabrication and measurements of the cement composites.

Day	Plain cement	14 wt. % (7 days)	18 wt.% (7 days)	18wt.% (28 days)
0	fabrication	fabrication	fabrication	fabrication
1	demoulded	demoulded	demoulded	demoulded
1	cured in water	cured in water	cured in water	cured in water
7	extracted from water	extracted from water	extracted from water	--
21	SE meas. 2 weeks	SE meas. 2weeks	SE meas. 2 weeks	--
28	--	--	--	Extracted from water
42	--	--	--	SE meas. 2 weeks
70	SE meas. 10 weeks	SE meas. 10 weeks	SE meas. 10 weeks	--
98	--	--	--	SE meas. 10 weeks

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2.2 Morphological analysis

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2.3 Radiofrequency measurements

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The total shielding effectiveness can be defined as the ratio of the incident and transmitted field. It can be obtained from the measured transmission loss (S_{21}), in a waveguide as:

$$SE = -20 \text{ Log}(|S_{21}|) \quad (1)$$

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122 The total shielding effectiveness of a material comprises of dissipation loss, L_D , and mismatch loss,
123 L_M [32]:

$$SE = L_D + L_M \quad (2)$$

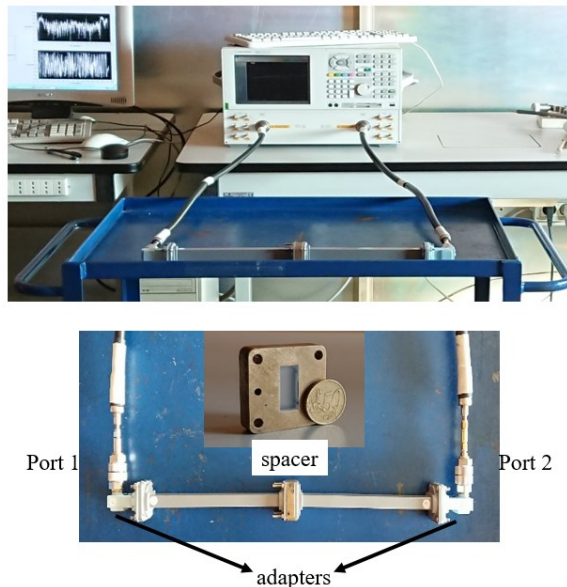
125 where L_M can be calculated from the reflection scattering parameter by:

$$L_M = -10 \log_{10}(1 - |S_{11}|^2) \quad (3)$$

$$L_D = -10 \log_{10} \left(\frac{|S_{21}|^2}{1 - |S_{11}|^2} \right) \quad (4)$$

128

129 The scattering parameters of the composites are measured in a WR90 rectangular waveguide from
130 8GHz to 12GHz using a setup similar to [33]. The samples are fabricated in order to fit the rectangular
131 waveguide cross section ($a=22.86\text{mm}$, $b=10.16\text{mm}$). The thickness of the samples is 4mm. The setup is
132 shown in Figure 2. It consists of a two-port Vector Network Analyzer (VNA) (Agilent E8361A); two
133 coaxial cables connected to the two ports of the network analyzer; two coaxial to waveguide adapters
134 and two rectangular waveguides. Between the waveguides flanges is inserted a spacer holding the
135 sample. Before the measurements, a two-port calibration (short, matched load, thru) is performed. The
136 reference planes are at the ends of the spacer.
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138

139 **Figure 2.** WR90 waveguide measurements setup.

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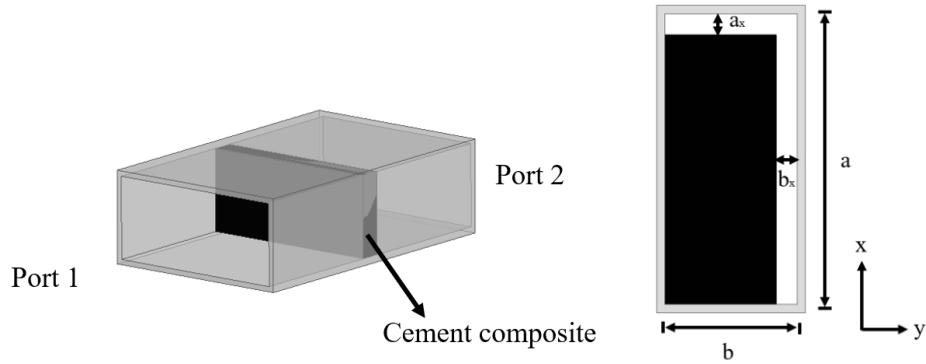
141 2.4 Finite element simulations

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143 A commercial finite element modelling tool, Ansys HFSS is used to simulate the waveguide with
144 the composite sample as shown in Figure 3. The material properties of the composite inserted in the
145 waveguide are chosen by fitting the simulated shielding effectiveness values to the measured shielding
146 effectiveness values. The composite dimensions and thickness are varied to analyze the impact of
147 fabrication tolerances and thickness on the values of shielding effectiveness.

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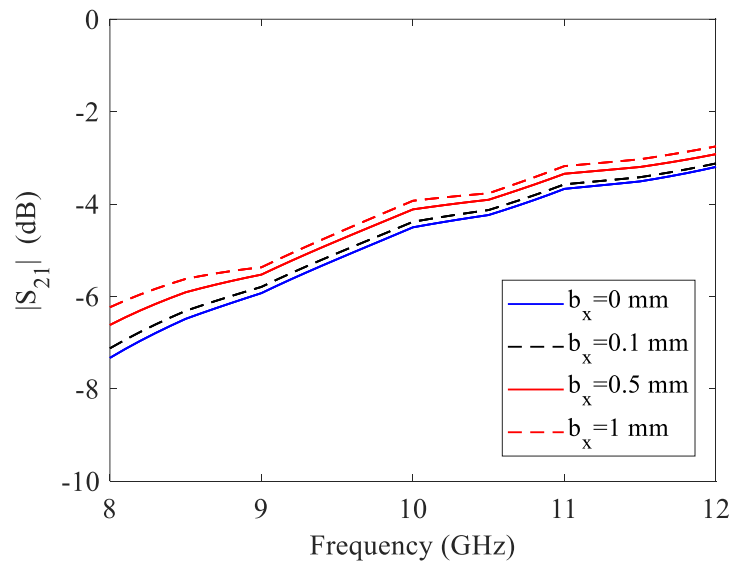


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Figure 3. Geometry of the simulated waveguide with composite (left panel). Geometry for the dimensional analysis (right panel).

154 *2.5. Dimensional tolerance analysis*

155 In order to take into account the dimensional tolerance of the cement composite, simulations were
156 performed based on varying the two dimensions along the x and y axis (see Figure 3). In case of plain
157 cement composites, it was found that there is negligible variation of the transmission properties by
158 varying the a_x dimension of the sample, while the impact of a variation of b_x is significant. A variation
159 of 0.5mm in b_x results in a variation of almost 1dB in the transmission coefficient as shown in Figure
160 4. It has been ensured that the tolerance in the dimensions of the cement composites is below this
161 value.



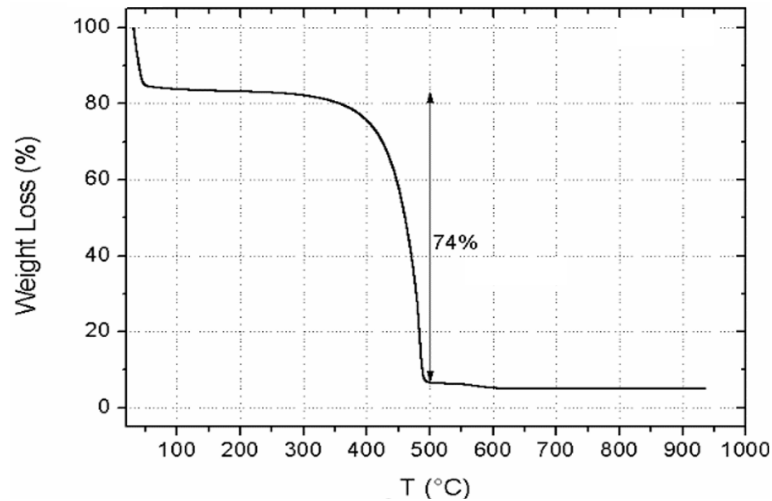
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Figure 4. Analysis of fabrication tolerances of the plain cement composites.

164 **3. Results**

165 *3.1. Biochar and composites characterization*

166 The water and carbon content of the biochar is investigated by TG-DTA experiments. TGA curve
167 of biochar is reported in Figure 5. Below 100 °C, the weight loss is about 16%, due to the evaporation
168 of the physically adsorbed water. From 350°C to 500°C the weight loss is due to the combustion of the
169 graphitic carbon fraction (about 74% of the total weight of the sample). At 950 °C, a residue of around
170 5 % in weight is observed respect to the initial amount.



171

172 **Figure 5.** TGA curve of biochar filler.

173 Figure 6 illustrates the SEM image of composites with the highest content of biochar (18wt.%) recorded
 174 with secondary electrons. The black structures shown in the SEM image are the carbonaceous particles.
 175 The expected elongated structure of the particles is due to the fiber origin of the biochar. The particles
 176 show a good dispersion in the matrix.

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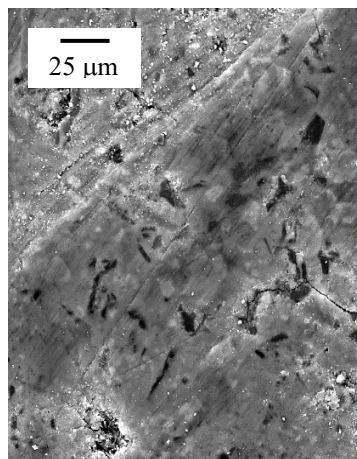
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Figure 6. SEM Micrograph of cement containing biochar 18% at 1000x magnification.

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3.2 Shielding effectiveness analysis

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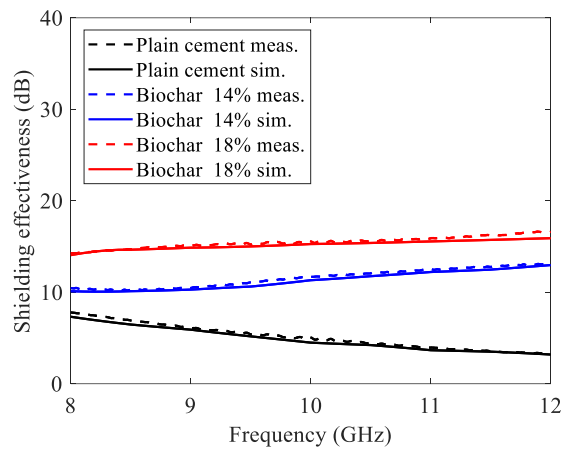
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Shielding effectiveness can be found from the measured transmission coefficient, S_{21} , in a waveguide (see Figure 2) as defined in equation (1). The measured shielding effectiveness of the plain cement used as reference sample, sample with 14wt.% and 18wt.% filler cured in water for 7 days and measured after 10 weeks are shown in Figure 7. At the center frequency of 10GHz, the shielding effectiveness of plain cement is almost 5dB, which increases to 11dB for the samples with 14wt.% of biochar. The maximum shielding effectiveness measured for the sample with 18wt.% is around 15dB. These results are obtained with 4mm thick samples. The shielding effectiveness values can be further increased by increasing the sample thickness and/or the percentage of biochar. The shielding effectiveness of the plain cement composites decreases with frequency. This behaviour is similar to other cement

200 composites [34]. The different behaviour in frequency of the biochar composites with respect to plain
 201 cement composites can be attributed to the presence of entrapped water in the biochar [35].



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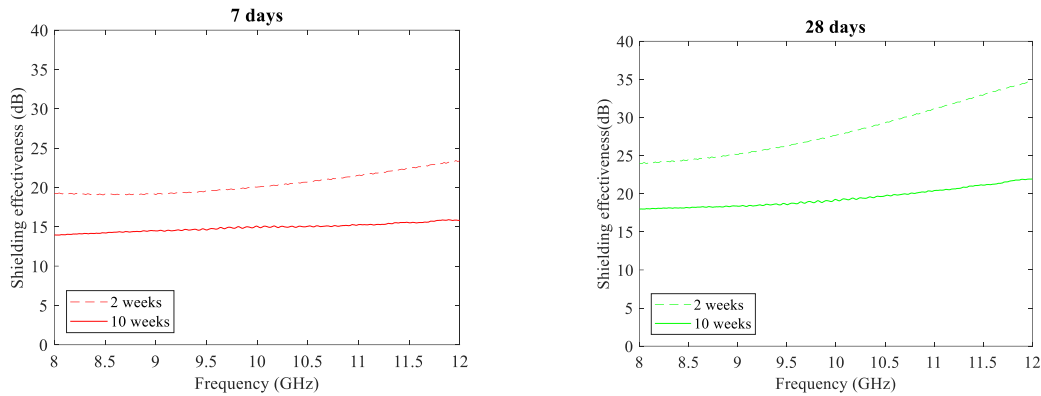
204 **Figure 7.** Measured and simulated Shielding effectiveness values for plain cement, sample with
 205 14wt.% of biochar and sample with 18wt.%. Samples cured for 7 days in water. Measurements
 206 performed after ten weeks ageing.

207

208 In Figure 7 the simulated shielding effectiveness obtained with full-waves simulations are reported
 209 (dashed lines). The values of complex permittivity are varied to fit the simulated shielding
 210 effectiveness values to the measured shielding effectiveness values and a good correlation between the
 211 measured and simulated data is obtained.

212 There is a strong correlation between the curing period in water and the mechanical strength of
 213 cement composites [30]. In order to evaluate the effect of the curing period in water on the shielding
 214 effectiveness values, samples with 18wt.% biochar cured in water for a period of 7 days and 28 days
 215 are analysed. The shielding effectiveness of the cement composite with 18wt.% biochar cured in water
 216 for seven days and 28 days measured after 2 weeks and 10 weeks are shown in Figure 8. It can be seen
 217 that the sample cured in water for 28 days has higher shielding effectiveness when measured both
 218 after 2 weeks and 10 weeks. The variation of the shielding effectiveness over time of the cement
 219 composite cured for 28 days is also higher than the one cured in water for seven days. This shows that
 220 the shielding effectiveness is increased due to the presence of water, the loss of water from the sample
 221 over time results in a reduced value of the shielding effectiveness value.

222



223

224 **Figure 8.** Measured shielding effectiveness of cement sample with biochar 18wt. % cured in water for 7days (left
 225 panel) and 28 days (right panel). Measurements performed after 2 weeks and 10 weeks.
 226

227 **4. Discussion**

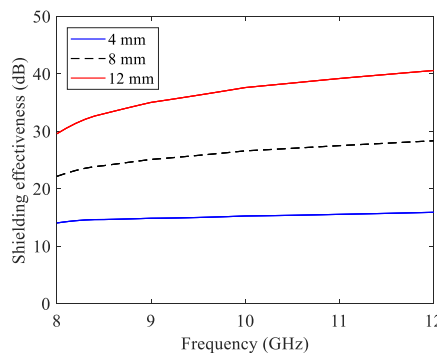
228 In order to evaluate the impact of the presence of biochar in the cement composites on the shielding
 229 effectiveness, a comparison has been performed with other works in literature (see Table 2). The case
 230 considered in this comparison is filled with 18wt.% biochar cured in water for 7 days and measured
 231 after ten weeks. The thickness of the samples considered is 4mm which provide a shielding effectiveness
 232 value of almost 14dB. In comparison with literature, other cement samples reported gives higher
 233 shielding effectiveness values due to a higher value of thickness. In order to evaluate the impact of the
 234 thickness on the shielding effectiveness values, simulations are performed with higher thickness values.
 235 The results are shown in Figure 9. As expected the shielding effectiveness increases considerably
 236 increasing the thickness of the sample.

237

Table 2. Comparison with literature

Ref.	Frequency	Measured after (days)	Thickness (mm)	Shielding effectiveness (dB)	Materials
[34]	3 GHz	36	100	17.5	cement
[36]	10 GHz	95	150	20	cement
This work	10 GHz	70	4	15	cement+18wt.% biochar

238



239

240 **Figure 9.** Simulated results for cement composites with 18wt.% biochar with different thicknesses.

241 5. Conclusions

242 Biochar is obtained by thermal treatment of waste products. It has been vastly used for soil
243 amendment. More recently, it has been used for applications as energy storage, carbon sequestration
244 and construction. The effect of a commercial biochar on the shielding properties of cement composites
245 is investigated in X-band. The conclusions drawn based on the results presented can be extended to
246 other microwave frequencies. Cementitious composites with ordinary Portland Cement (PC) were
247 prepared without biochar and with biochar as filler (14 wt.% and 18wt.%). Samples are prepared in
248 order to fit a WR90 waveguide (8-12 GHz). With the help of a full-wave simulator, the fabrication
249 tolerances of the samples are analysed. A variation of ± 0.5 mm results in a change of the shielding
250 effectiveness of ± 1 dB. Shielding effectiveness can be obtained from the measurements of scattering
251 parameters. Samples with 14wt.% and 18wt.% biochar as filler are cured in water for 7 days. As
252 expected the shielding effectiveness increases with the increase of the percentage of filler (11dB for
253 14wt.%, and 15dB for 18wt.% at 10GHz). In order to evaluate the effect of the curing period in water
254 on the shielding effectiveness values, different curing period are analysed. Samples with 18wt.%
255 biochar are cured in water for a period of 7 days and 28 days. The shielding effectiveness increases by
256 approximately 5dB in the whole frequency range for the samples cured in water for 28days as
257 compared to samples cured in water for 7 days.
258

259 **Author Contributions:** composites fabrication, D.D., and G.R. ; waveguide measurements and discussion of the
260 shielding effectiveness, D.D, M.Y. and P.S; microstructure characterization and TGA, I.N.S.; full-wave
261 simulations, M.Y; original draft preparation, M.Y. and P.S.; writing—review and editing M.Y. , P.S. and I.N.S.;
262 supervision, P.S.; conceptualization M.Y., P. S. and I.N.S., funding acquisition G.R. All authors have read and
263 agreed to the published version of the manuscript.

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266 **Conflicts of Interest:** The authors declare no conflict of interest.
267

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