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APPLIED RESEARCH

Characterization of the Electromagnetic Shielding Effectiveness of Biochar-Based Materials

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ABSTRACT Biochar and biochar-based composites are multi-functional materials with a wide range of environmental and other applications. A new and emerging application is in the field of modern communication systems as materials with significant electromagnetic shielding effectiveness. In this study, biochar was prepared from olive tree prunings which was subsequently used at various dosages to prepare composite samples with carbon black and polytetrafluoroethylene as binder. The electromagnetic shielding effectiveness of the samples were measured in the frequency range of 1-3 GHz, using a simple, user-friendly and reliable experimental set-up. The results showed that raw, unmodified biochar had a low shielding effectiveness in the range of 1.5-4 dB however, it slightly increased as the thickness of the sample increased from 0.1 to 0.5 mm. Modifying biochar with the 20 %w/w acetylene black largely increased its shielding effectiveness, reaching the value of 39 dB in the same frequency range. Our approach demonstrated that raw biochar can be used as a substrate to develop composite materials with significant shielding effectiveness and assess their performance with a quick and simple laboratory method.

INDEX TERMS Electromagnetic interference, shielding effectiveness, biochar, electric resistivity, carbon.

I. INTRODUCTION

The widespread use of electronic devices and instruments in various scientific, military and commercial applications, and the spectacular growth of modern communications have promoted the development of new materials appropriate for the mitigation of electromagnetic interference (EMI), as a topic of great interest [1]. Among the materials with the desired characteristics are metals in solid or powder form, metal-fiber filled plastics, polyacrylonitrile nickel-coated reinforced polymers, aluminum structures, coatings, nickel and copper metalized fabrics, as well as nanoreinforced polymer composites [2], [3], [4]. In order to determine the electromagnetic properties of the new materials and their possible applications, a reliable measurement of EMI Shielding Effectiveness (SE) over a broad frequency range is essential.

The EMI shielding effectiveness of carbon materials, such as graphene, carbon nano-tubes and carbon aerogels, has been well studied and reviewed by Gupta and Tai [5]. Despite their EMI shielding performance, the preparation of these materials often involves complex methodologies and costly reagents, which may not be sustainable at large scale. Therefore, there is a need for carbon materials of increased EMI shielding performance that can be prepared at low cost through simple and sustainable methodologies. One such material is biochar, which can be prepared by pyrolysis of certain biomasses and agricultural waste. Given the intensive production of olive oil in the Mediterranean region, olive tree prunings are probably the most abundant agricultural waste, both in terms of mass and volume. The current practice for

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such biomass is open burning in the fields and there is no established exploitation methodology.

The physicochemical properties of biochar depend on the feed-stock properties, pyrolysis temperature and residence time. Biochar is a multi-functional material with a continuously increasing number of diverse applications. Until now, it has been demonstrated that it can improve the water and nutrient holding capacity of sandy soils, act as a catalyst in wastewater treatment and reduce the greenhouse gases emissions from cattle when added in their fodder.

Biochar application as an EMI shielding material is still in its infancy. Recently, biochar was combined with gypsum and the composite material showed a positive EMI shielding performance [6]. At 1 GHz and biochar contents of 10, 20, and 40% w/w the authors reported shielding effectiveness of 3, 5.5, and 9.5 dB, respectively. A similarly positive EMI shielding behavior was observed by Torsello et al. from their biochar-based epoxy resin composite [7]. Up to date, the information on the electromagnetic shielding efficiency of biochars is very limited and only refers to biochar-based composite materials, whereas measurements of raw, unmodified biochars are missing.

The rationale of this work was to assess the EMI shielding efficiency of unmodified, as-produced biochar, using a simple and user-friendly measuring methodology. The assumption is that if raw biochar and biochar amended with commercial activated carbon show positive EMI shielding performance, then biochar may be sustainably used as a filler in building materials or combined with other materials to develop composites with adjustable EMI shielding properties. Furthermore, if the proposed method successfully and quickly provides information on the shielding performance of materials, it can be later used a preliminary screening tool before detailed investigations occur. The significance of this work is that the use of costly materials that are prepared from non-renewable fossil fuel sources (such as graphene and commercial activated carbons) can be gradually reduced and replaced by engineered biochars. Furthermore, since calcium is a good electrical conductor, Ca-rich biochar may have a promising EMI shielding capacity, without the need of adding metals or metal oxides at an additional preparation step to increase conductivity. Therefore, the specific objective was to produce Ca-rich biochar from olive tree prunings and biochar-based composites and investigate their EMI shielding properties through a quick, accurate and user-friendly methodology.

II. RESULTS AND DISCUSSION

A. BIOCHAR CHARACTERIZATION

The elemental analysis, ash content and surface area of OTB can be seen in Table 1. The reported values were within the typical range of other reported biochars prepared from woody residues [9] and in line with the European Biochar Certificate standards [11]. Compared to activated carbons and graphene, OTB had a relatively low surface area and not ideal for EMI applications. It is known that the surface area participates in the reflection mechanism of EMI, therefore high surface area materials contribute more to this shielding mechanism.

TABLE 1. OTB characterization.

Composition	%
C	76.7
H	1.65
N	0.808
S	Non-detected
O*	12.96
Ash	7.91
Na_2O	0.26
MgO	0.58
Al_2O_3	0.19
SiO_2	0.33
P_2O_5	1.00
K_2O	2.45
CaO	25.97
TiO_2	0.04
Cr_2O_3	Non-detected
MnO	0.05
Fe_2O_3	0.48
Cl	0.10
Sr (ppm)	610
Cu (ppm)	104.3
Zn (ppm)	161.8
Rb (ppm)	49.6
Zr (ppm)	73.4
BET surface area (m^2/g)	375
Total pore volume (cm^3/g)	0.1796
Mean pore diameter (nm)	1.9117
Particle size distribution	μm
D (v, 0.1)	2.27
D (v, 0.5)	14.64
D (v, 0.9)	50.32

* Calculated by difference

The mean pore diameter value indicated an essentially microporous material. The analysis of the ash revealed high

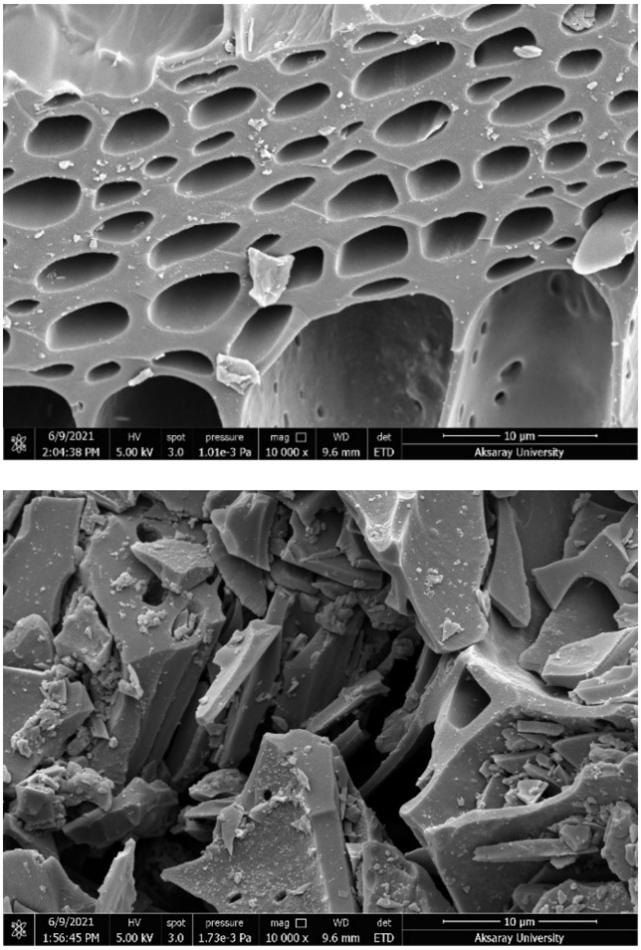

FIGURE 1. Amorphous irregular regions of biochar in electron microscopy consisting of a mixture of micropores in the range of 4-8 μm.

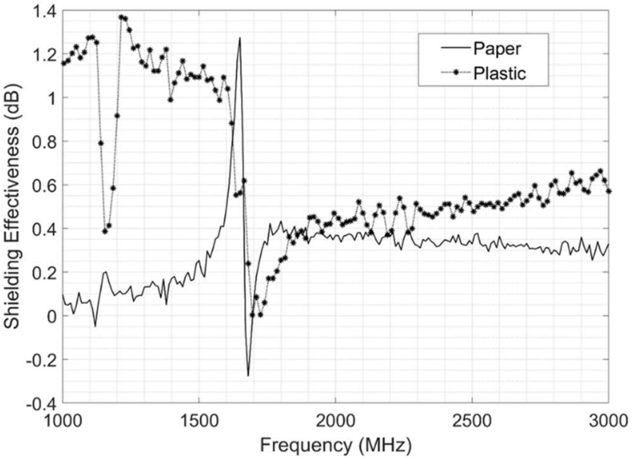

FIGURE 2. Measurements of the very low shielding effectiveness of dielectric (not conductive) material samples.

concentration of CaO, due to the growth of olive trees in calcareous soils, typically known for their high concentration of $CaCO_3$. Calcium is a good electrical conductor (0.298 x 10^6 cm Ω), therefore a valuable conductivity aid in the composite.

The milling process successfully reduced the particle size of all biochar below 100 μm. Particle size reduction resulted in a homogenized biochar mass, necessary for successful mixing with the binder and the solvent. The scanning electron microscopy indicated a surface consisting of a mixture of micropores in the range of 4-8 μm (Fig. 1 top) and amorphous, irregular regions (Fig. 1 bottom). The voids shown in Fig. 1 (top), restrict the spreading of EM waves and produce heating because of the impendence dissimilarity, boosting the microwave absorption properties. Furthermore, increased porosity renders the material more lightweight, which is a beneficial attribute for polymer-based EMI shielding composites.

It has been established that as the pyrolysis temperature increases beyond 700°C, biochar surface becomes more uniform and microporous, whereas at lower temperatures amorphous regions typically occur [9]. Therefore, since OTB was produced at ~600°C, the SEM observations agree with earlier results.

B. SHIELDING EFFECTIVENESS – METHOD VALIDATION AND MEASUREMENT OF SAMPLES

Figure 2 shows the shielding effectiveness of paper and plastic sheets. As expected, these non-conductive materials exhibited minimal SE in the range of 0-1.2 dB. Fig. 3 confirms that the aluminum foil sample exhibited a strong shielding effect, as its SE values were in the range of (i) 70 – 92 dB for the frequency range 0 – 800 MHz, (ii) 59 – 70 dB for 800 – 1150 MHz, and (iii) 66 – 80 dB for 1150 – 3000 MHz. Obviously, frequency plays a determining role in the shielding response of any material, and thus the study of the behavior of all materials at the widest possible frequency range is necessary. As aluminum foil is quite impenetrable for electromagnetic waves in the frequency range of 0 – 800 MHz, but more penetrable in the range of 800 – 1150 MHz, likewise other materials may be shielding effective in a specific frequency range and at the same time quite transparent in another frequency area. All measurements have been performed with the methodology described in section III-A2), while the paper, plastic and aluminum foil materials have been used for comparison and the evaluation of the measurement technique.

Figure 3 reveals that the shielding performance of the OTB3-AB20 sample (dashed line) with SE values in the range of 26 – 39 dB is comparable to the shielding efficiency of aluminum foil with SE values in the range of 59 – 92 dB, for the frequencies 0 – 3 GHz. The addition of commercial carbon black to the raw biochar considerably improved the conductivity of raw biochar.

This observation was confirmed by the low resistivity value obtained by the DC measurement and reported in Table 2. Although OTB3-AB20 achieved lower SE values than aluminum however, it is a promising composite material with tunable properties and positive EMI shielding behavior.

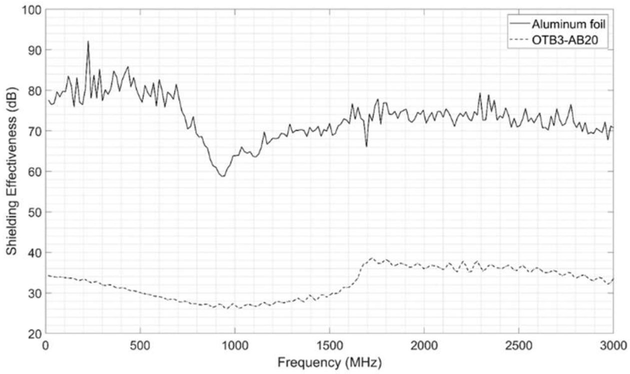

FIGURE 3. Measurements of the high shielding effectiveness of aluminum foil (very conductive) sample, in comparison to the OTB3-AB20 sample.

TABLE 2. DC resistivity of the various test samples.

Sample	DC resistivity (R)
OTB01	1010 kΩ
OTB02	940 kΩ
OTB03	810 kΩ
OTB05	700 kΩ
OTB3-AB10	270 kΩ
OTB3-AB20	70 Ω

Measurements of the raw biochar samples OTB01, OTB02, OTB03, and OTB05 shown in Fig. 4, demonstrate relatively low shielding effectiveness values in the range of 1 – 4.2 dB for the frequencies 1 - 3 GHz. In the frequency region of 1.6 – 3 GHz, the increasing thickness of the biochar sample had a small but noticeable effect on the shielding effectiveness. This relative independence of the SE on the thickness of materials is has been shown earlier and is typical for materials of <1 mm thickness. It is due to the large skin depth (i.e. the penetration depth of an oscillating electric field) in such materials, rendering EMI shielding applications of very thin materials unfavorable [15], [16]. Still, the EMI monitoring method was sensitive enough to detect these small SE differences between the samples.

The composite sample OTB3-AB10 exhibited a considerably better and worth-mentioning shielding performance with SE values in the range of 4.2 – 12.8 dB, due to the conductivity enhancement originating from acetylene black addition to the raw biochar. However, the SE behavior of the OTB3- AB10 sample is not the same throughout the whole 1 – 3 GHz frequency range, since (i) between 1.0 – 1.6 GHz the SE changes only a little (4.2 – 5.5 dB), (ii) between 1.6 – 1.7 GHz an incremental transition occurs over-doubling the SE values, and (iii) between 1.7 – 3 GHz the SE gets substantially higher (10 – 12.8 dB), suggesting that the material may be useful for partial shielding from electromagnetic waves within the 1.7 – 3 GHz range. Moreover, the raw biochar samples also exhibited a noticing peculiarity of different performances between the 1.0 – 1.6 GHz and the 1.7 – 3 GHz frequency ranges. More specifically: (i) between 1.0 – 1.6 GHz there is no specific comparative performance between the 4 raw biochar specimens, as for some frequencies OTB01 has the higher and OTB05 the lower SE, while for other frequencies OTB05 has the higher and OTB01 the lower SE, (ii) between 1.7 – 3.0 GHz OTB05 per-formed better than OTB03, OTB03 had better SE than OTB02, but OTB01 appeared less efficient (in the 1.7 – 2.5 GHz subrange) or slightly better than OTB02 (in the 2.5 – 3.0 GHz subrange). The transitions in Fig. 4 are analogous to the resonance peaks which were closely corresponding to the resonant frequencies of the radial transmission line modes of the measurement configuration [14].

Although biochar itself cannot form a highly conductive network, however, when it is applied as substrate in composites, the shielding effectiveness may be improved due to the increased number of wave-scattering centers, despite its lower conductivity [17]. Therefore, hybrid composites (i.e. biochar/acetylene black) can provide higher shielding performance and be fine-tuned to control the percolation threshold and conductivity of composites, parameters that are troublesome to control when using only highly conducting fillers. Furthermore, assuming linear frequency dependency of SE, the composites could likely provide effective shielding also at lower frequencies (<1 GHz). As reported by Torsello et al., a biochar produced at temperatures >1000^{o}C is highly conductive due to the increased level of ordered graphitic structure and could therefore be used alone without the need for conductive additives [7]. However, maintaining a pyrolysis process at these conditions is not energy-efficient and – depending on feedstock – the yield losses are considerable.

Table 2 reveals the very low resistivity value of OTB3-AB20, while all other samples are measured with high resistivity values between 270 and 1000 kΩ. Figure 5 demonstrates the low resistivity (high conductivity) of the OTB3-*AB*20 biochar sample, using a very simple electric circuit, consisting of a red light-emitting diode (LED) being bridged by the sample under test and powered by a 5 V DC power supply. When the circuit is closed (as shown in Fig. 5), the red LED turns on. On the contrary, when there is no contact with a biochar piece (open circuit), or when a sample of another material is used, the LED remains off.

III. MATERIALS AND METHODS

A. MATERIALS

The raw biochar sample was prepared from dry and homogenized olive tree prunings using a flame-curtain pyrolysis kiln. The technical details of the kiln can be found in our earlier publication [8]. Polyethylene terephthalate (PTFE) 60% w/v emulsion in water was used as the biochar binder (Redox.me, Sweden). 1-methyl-2-pyrrolidone (Suprasolv˙, Merck) was used as the solvent. Acetylene black (Alfa Aesar, 100% compressed, 99.9+%) was added in selected specimens as a conductivity aid.

1) BIOCHAR PREPARATION AND CHARACTERIZATION

The olive tree prunings were thermochemically converted to biochar at 600oC and 1h residence time. The mode of

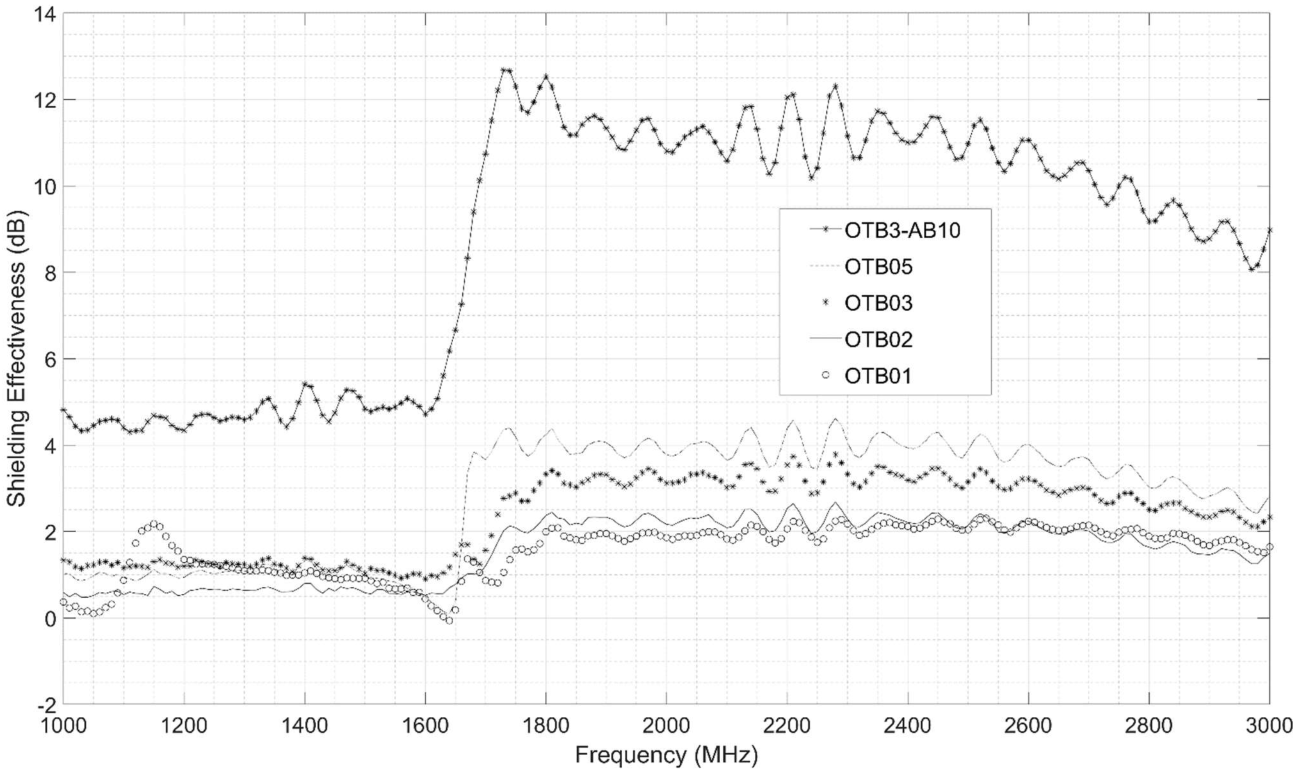

FIGURE 4. Measurements of the shielding effectiveness of OTB samples of various thicknesses.

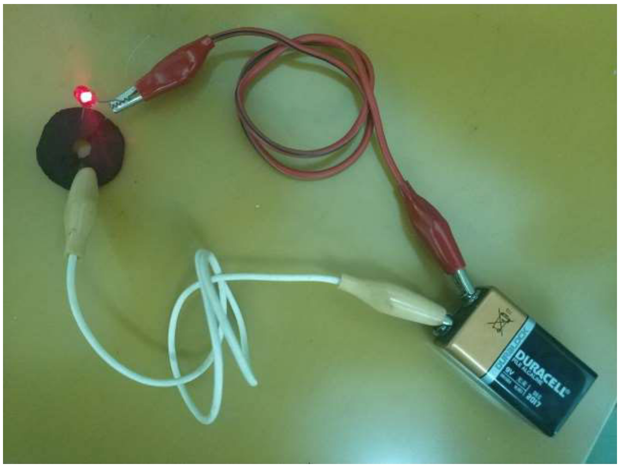

FIGURE 5. Photo demonstrating the low electric resistivity of biochar sample OTB3-AB20. The LED light turns on when a 5 V DC electric circuit supporting a red LED is in contact with the sample.

operation has been described in Kalderis et al. [9]. It has been demonstrated that flame-curtain pyrolysis can produce biochars with reproducible properties, suitable for advanced technologically applications [10]. The raw biochar sample was ground in a Sepor type rod mill for 30 min and the ground sample was used for the composite sample.

The particle size distribution was determined using a Malvern type S Mastersizer (Version, Malvern Instruments, Malvern, UK) (particle size range between 0.05 and 850 μm) combined with laser diffraction. Elemental analysis (C, H, N, S) was performed on a Thermo Flash 2000 combustion analyzer (Thermo Fisher Scientific, UK). The ash content was determined according to the European Biochar Certificate (EBC) Guidelines [11]. The oxygen content was calculated by difference. The chemical composition of OTB was determined using an X-ray fluorescence (XRF) spectrometer (Rigaku ZSX Primus II, Japan). The nitrogen adsorption isotherm of OTB was measured at 77 K in a commercial apparatus (BELSORP mini II, MicrotracBEL Corporation, Japan). The data of N2 adsorption isotherm were used for the characterization of the textural properties of the sample. The Brunauer–Emmett–Teller (BET) method was used for the estimation of the specific surface area. OTB was morphologically characterized in a Zeiss/Supra 55 (Germany) high-resolution scanning electron microscope.

A modified version of the method described in Tsubota et al. was applied to prepare the biochar-based composites [8]. The modification involved the use of PTFE in a water emulsion instead of using PTFE powder. The composite samples were prepared by mixing 10 ml of raw biochar powder with 3 ml of PTFE emulsion and 3 ml of 1-methyl-2-pyrrolidone. The mixtures were rolled into sheets of the following thickness: 0.1, 0.2, 0.3, and 0.5 mm, followed by vacuum drying in a dessicator at 60oC for 24 h. The raw biochar samples are referred to as OTB01, OTB02, OTB03, and OTB05 thereof.

FIGURE 6. Top: SE monitoring equipment, midle: monitoring devices, bottom: close-up of OTB samples prepared for shielding effectiveness tests.

For comparison reasons, raw bio-char was amended with 10 and 20% w/w acetylene black. These samples had a thickness of 0.3 mm and are referred to as OTB03-AB10 and OTB03-AB20, respectively.

2) SHIELDING EFFECTIVENESS MEASUREMENTS

Shielding effectiveness (SE) is typically defined as the ratio of the incident electric field magnitude E_i to the transmitted electric field magnitude E_t. All SE tests intend to determine the insertion loss (IL) coming from the interposition of the material under investigation between the source and the signal analyzer. SE is determined by two consecutive comparative measurements, when the material is either the reference or the loaded specimen, corresponding to the cases of the absence or the presence of the measured shielding material, respectively. Thus, comparing the measured electric field strength for the reference (E_R) and the load (E_L) specimens, we have for the shielding effectiveness of the composite samples:

$$SE\,(dB) = 20\log_{10}\left(\frac{E_R}{E_L}\right) = E_R\,(dB) - E_L(dB) \tag{1}$$

Alternatively, we can compare the measured power level for the reference (P_R) and the load (P_L) specimens and have:

$$SE\,(dB) = 10\log_{10}\left(\frac{P_R}{P_L}\right) = P_R\,(dB) - P_L(dB) \tag{2}$$

Shielding effectiveness is defined for incident transverse electromagnetic (TEM) waves, which are similar to plane waves coming from a distant source. In coaxial transmission lines, the TEM mode is always present, and the magnetic and electric field vectors are perpendicular to each other and to the direction of the wave propagation [12].

Currently, there are several methods to test and measure SE, based on the type and the applications of the materials under investigation, and the frequency range of interest. In this work, the ASTM D4935-18 standard was selected, since other standards have limited dynamic range, need relatively large specimen dimensions (square samples with 46 cm^2 surface), result in relatively expensive testing procedure, and they are impractical and inadequate to test the newly developed nano-engineered and nano-reinforced materials [13]. Moreover, we have used a different tester, which is accurate, economical, easy to manipulate and able to address the current requirements for SE testing [14], instead of the 18 kg mass device provided by the D4935-18 standard, which is this standard's main drawback, as it makes frequent handling cumbersome during assembling and dis-assembling. This device (holder) of the ASTM D4935-18 has a complex shape and is rather difficult to be manufactured, since it is a flanged circular coaxial transmission line with internal conical shape, which secures the sample and capacitively couples the coaxial conductors. However, the considerable ad-vantage of the proposed standard SE tester is the small sample size. The required size of the sample is small in comparison to the size required by other SE testing methods, leading to very low specimen production costs. This way, the SE measurements of this work were simple, effortless, easy, fast and cost-effective, due to the convenient coaxial holder used, the relatively small test samples required (circular disks with 133 mm diameter), the minimum time required for preparation and measurement and the low cost of specimen production. Measurement of IL or SE using the ASTM D4935-18 standard tester could also be used to estimate the electric conductivity and the near-field SE of electrically thin samples. For the evaluation of the OTB samples, the measuring device of Fig. 6 was used. Details of the construction of the device can be found elsewhere [6]. In order to test and verify our measurement methodology, materials with known shielding capabilities were measured up to the 3GHz (available VNA equipment limit) although this measurement device can be used up to 18 GHz. The following common materials were selected: (i) plain paper sheet used for printing, (ii) transparent plastic film used for food wrapping, and (iii) aluminum foil used for kitchen purposes. Each of these sheets had a thickness of 0.015 mm. The first two, which are established dielectric materials with no conductivity, negligible SE was expected, while from the last one, which is an established conductor, a high shielding performance with very high values of SE was expected. All measured samples (both the composites and the references) were shaped in the dimensions needed with the equipment (silver one) of Fig. 6 middle figure (on the upper part).

IV. CONCLUSION

We have examined the shielding properties of biochar composites at the microwave frequencies of 1 – 3 GHz, using the ASTM D4935-18 standard method to measure and directly quantify their corresponding shielding effectiveness. Our preliminary results show that the biochar content and the sheet thickness affect shielding response, allowing us to engineer the biochar composite and thus modify its properties to achieve specific electro-magnetic shielding values. The described experimental procedure represents a straight-forward way to investigate new materials for EM shielding. Furthermore, adding metallic particles from waste materials could be a viable approach to further increase the SE of biochar. Future work involves the development of a method to combine biochars with Fe- and Al-rich waste materials and consequently tune the electromagnetic shielding effectiveness of the produced composite in the expanded frequency range of 0 – 18 GHz to cover multiple applications as telecommunications in 6G and beyond.

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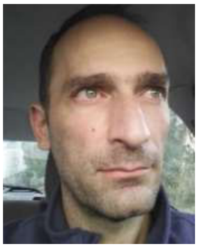

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