Microwave characterization of polymer composite based on Biochar: A comparison of composite behaviour for Biochar and MWCNTs

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Comparison between Biochar and MWCNTs composite behaviour

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***Abstract* — In this work, Biochar is used as a filler in Epoxy resin for composite preparation. The electrical characteristics of composites are analyzed as a function of different filler percentage. Results obtained will be compared with composites based on multi-walled carbon nanotubes.**

***Keywords* — Biochar; carbon nanotubes; microwave characterization**

I. Introduction

Electrical properties of polymer composites based on carbon filler are a hot spot of recent years [1,2]. As sp^2 hybridised carbon is a good conductor due to the mobility of electrons in its outer valence shells, it can be used as filler material. The consequent possibility to tune electrical properties of composites lead to new applications for them. Carbon naontubes (CNTs), in particular multi wall CNTs (MWCNTs), are the most commonly used form of carbon in composites. Recently, less expensive and more environment friendly carbon-based fillers derived from recycled materials are gaining interest. Biochar is a recalcitrant carbonaceous product obtained from pyrolysis of biomass and other biogenic wastes [3]. Pyrolisis temperature is a key point during its production. Lower pyrolysis temperatures produce biochar with higher yields, and greater levels of volatiles, electrical conductivity and cation-exchange capacity. Conversely, higher temperatures generate biochar with a greater extent of aromatic carbon, alkalinity and surface area with microporosity. Biochar treated at high temperature increase its surface area and is known as Activated Biochar.

In this paper, Biochar as produced and Activated Biochar were used to realize composites based on Epoxy resin. The influence of carbon filler on the electrical properties in the frequency range 1-10 GHz was analyzed. Results are compared with those obtained using MWCNTs as a filler for the same polymer.

II. Samples Preparation and Characterization

A. *Composite preparation*

Composites based on epoxy resin were prepared as reported in [4]. Briefly, epoxy resin was mixed for few minutes using a high-performance micro dispersing tool with a pre-weighted quantity of carbon filler in powder form. The hardener was added and a mechanical mixing performed. The composite, in liquid form, was poured in a cubic mold and let to rest until completely hardened. In figure 1c is reported an example of the cubic shaped samples used for electrical characterization. Sensor for microwave characterization need a plane and smooth surface with a thickness related to the dielectric constant of the tested material. In our case, a cubic shape is the best choice because it has a plane surface where the sensor can be positioned in different positions and it has an appropriate thickness. Results reported are an average of different measurements on a given face for different sensor positions. The characteristics of the fillers used are given in Table 1.

Table 1: Carbon material fillers characteristics

Name	Diameter [nm]	Length [µm]	Purity [%]	Surface area [m^2/g]
Biochar	-	-	>90	>160
ABiochar	-	-	>90	>800
MWCNTs	25-45	>10	>97	>200

B. *FESEM analysis*

FESEM (Zeiss 40, Field Emission Scanning Electron Microscopy) was used to investigate the morphology of Biochar and its dispersion in composites. Surfaces analyzed by FESEM were obtained by cryo-fracture. This procedure consists of immersing the sample in liquid nitrogen (77 K, -

196°C) in order to achieve rapid freezing. After a few minutes of immersion, the sample is removed from the liquid nitrogen and immediately broken into pieces by fast mechanical compression. This technique maintain the internal structure of the specimens intact, avoiding resin elongation with subsequent filler displacement. The cryo-fractured composites were then coated with a thin chromium layer of a few nanometers (<5 nm), in order to create a conductive surface.

Selected images of Biochar as prepared and Activated are shown in Fig. 1a,b. The main difference is that the surface of Activated Biochar is more irregular. This could be related to the high temperature treatments used to produce Activated Biochar. Both types present a honeycomb internal structure. Figure 1d shows the achieved uniform dispersion of Biochar in polymer matrix.

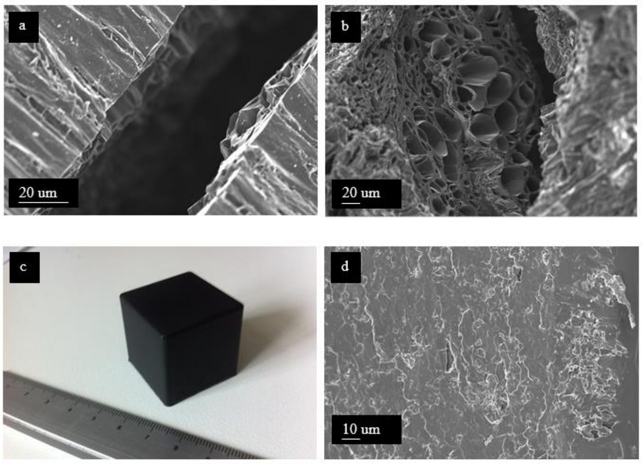

Fig. 1. FESEM for a) Biochar b) Activated Biochar c) composite (ruler included to highlight size) d) composite.

III. PERMETTIVITY AND CONDUCTIVITY COMPOSITE MEASUREMENTS

The complex permittivity of pure epoxy resin and composites was measured in the frequency range of 1 to 10 GHz. The samples were measured using a commercial open ended coaxial probe (Agilent 85070D) and a network analyzer (E8361A). The measurement setup is reported in [5]. A standard calibration short/air/water was adopted. This type of measurements was chosen because of its wider frequency band of reliability with respect to waveguide measurements or free-space measurements. Moreover, samples can have relatively small dimensions. The drawback is that they should have a very smooth and flat surface, as previously discussed.

Biochar as produced and Activated have been used as fillers in the quantity of 20 wt.% to produce cubic samples as shown in figure 1c. Real permittivity and conductivity measurements as a function of frequency and compared with pure Epoxy resin are reported in figure 2. Considering the same weight percentage (20 wt.%) of the fillers, the values obtained with Activated Biochar are approximately twice those obtained with Biochar.

Data obtained using 3 wt% of MWCNTs as filler (taken form ref. [6]) are also reported in figure 2. We observed that similar values are obtained although with different filler amount (3 wt.% for MWCNTs vs 20 wt.% for Biochar). However, we point out the cost and eco-friendly advantage of using Biochar. MWCNTs are much more expensive and they need somewhat harsh chemical process for production. Biochar is cheaper and it derives by a process that involve the recycling of waste material [3].

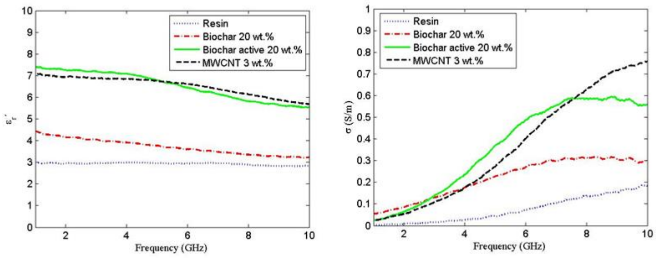

Fig. 2. Real permittivity (left panel) and conductivity (righ panel) of Biochar as it is, activated (both at 20 wt.%), MWCNTs (3 wt.%) and Epoxy resin.

IV. CONCLUSIONS

In this work, Biochar as it is and Activated is used as filler in Epoxy resin at 20 wt.%. Permittivity and conductivity measurements up to 10 GHz are compared with pure Epoxy resin and MWCNTs at 3wt.%. Results show a good performance for Activated Biochar and its undiscussed advantage in economical and eco-friendly terms.

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