

Airbrushing of carbon nanotubes on glass fibers for electromagnetic shielding epoxy composites

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Abstract

Tricomponent epoxy-matrix nanocomposites were prepared by airbrushing multiwalled carbon nanotubes (MWCNT) on glass fiber fabric (GF), aiming to establish a scalable route to produce electromagnetic interference (EMI) materials. The MWCNT deposition on GF by airbrushing was evaluated by scanning electron microscopy (SEM), showing a very reasonable dispersion even at high MWCNT concentrations. Electrical conductivity measurements have shown a maximum of 1.2×10^{-3} S/cm for GF with 3.4 wt% MWCNT. Electromagnetic shielding response for GF airbrushed with MWCNT and epoxy-matrix nanocomposites were analyzed considering reflection, absorption and transmission mechanisms and have shown an increasing trend as the MWCNT content increases, reaching the best result of 7.6 dB of shielding effectiveness (SE) in X-band spectra for the composite with 3.4 wt% MWCNT. The results showed that the airbrushing process can be a promising and easy route for manufacturing of MWCNT/GF/epoxy nanocomposites.

Keywords: nanocomposites, epoxy, airbrushing, carbon nanotubes, EMI materials.

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1. Introduction

Electromagnetic waves have been intensively used along the years, due to innovations such as mobile phones, antennas for data transmission and receiving, and security systems in aircraft and automobiles, amongst others. Therefore, there is a high level of microwave pollution causing interference in electronic equipment, leading to a rush for searching new materials with the capacity of attenuating the electromagnetic interference (EMI) caused by this pollution^[1,2].

EMI shielding is evaluated in terms of reflectance (R), absorbance (A), and transmittance (T) of the material, which refer to the fractions of the incident wave reflected, absorbed, and transmitted, respectively. The shielding efficiency (SE) in terms of T, R and A, can be calculated by Equation 1^[3]:

$$SE = 10 \log\left(\frac{1}{T}\right); SE_R = 10 \log\left(\frac{1}{1-R}\right); SE_A = 10 \log\left(\frac{1-R}{T}\right) \quad (1)$$

In nanocomposites containing carbon nanotubes (CNT), the absorption mechanism involves the interactions between electrons in CNT and the incident wave, thus converting the wave energy into heat. The reflection process occurs when the incident wave returns from the surface of the material by the difference of impedance (Z) between the surface and the air^[4,5]. Impedance is a barrier that electromagnetic waves find to penetrate the material, and it is related to the electrical conductivity (σ), frequency (w), and magnetic permeability (μ) as shown in Equation 2^[6].

$$Z = \sqrt{\frac{\omega\mu}{2\sigma}}(1+j) \quad (2)$$

If the effect of multiple reflection between both interfaces of the material is negligible, the relative intensity of the effectively incident EM wave inside the materials after reflection is based on the quantity as 1-R. Therefore, the effective absorbance (A_{eff}) can be described as $A_{eff} = (1-R-T)/(1-R)$ with respect to the power of the effectively incident EM wave inside the shielding material^[7].

$$A_{eff} = \left(\frac{A}{1-R}\right) \quad (3)$$

Comparing the effective absorbance (A_{eff}), i.e. the ratio between the absorbed (A) and the reflected fractions (1-R), as shown in Equation 3, is possible to find the overall shielding mechanism: if A_{eff} fraction is higher than reflection fraction, it suggest an absorptive shielding mechanism, and if A_{eff} fraction is smaller than reflection fraction, it suggest a reflective shielding mechanism.

The major goal in EMI shielding is to increase the attenuation, finding the most adequate combination of some parameters such as thickness, concentration of CNT and manufacturing techniques. The effect of CNT conductive networks, including individual carbon nanotubes and clusters, have been recently reported^[8-10]. These studies showed that

CNT/CNT-cluster networks are highly efficient for improving electrical conductivity and microwave shielding^[8], and that dense MWCNT percolated networks are required for SE enhancement^[9]. Decreasing the size of CNT clusters, the EMI SE of silicon rubber/CNT composites enhanced, due to the increase of interfacial contact area^[10]. The use of CNT in nanocomposites with epoxy, other resins and glass fibers (GF) for EMI shielding has been reported by several authors^[5,11-21]. In one of the first works on this type of EMI material, Park et al.^[5] reported that epoxy resin/GF composites containing up to 5 wt% CNT have reached 20 dB (99% of effectiveness) in 300 MHz – 1 GHz, with the highest value of electrical permittivity obtained for composites with 3% and 5 wt% CNT. Polystyrene composites were evaluated as well, showing 23.5 dB (99.5% of effectiveness) with 7.98×10^{-3} S/cm of electrical conductivity^[11]. More recently, tire rubber nanocomposites with 5 wt% CNT achieved shielding effectiveness (SE) of 66.9 dB (>99.99% of attenuation), with 109.5 S/m of electrical conductivity^[12], while oxidized multi-walled carbon nanotubes/waterborne polyurethane composites reached 23 dB at 5% CNT and 66.5 dB at 50% CNT^[13], all in the X-band frequency range (8–12.5 GHz).

Other studies also proposed the surface modification of glass fiber fabrics with nanoparticles, such as CNT^[14-19], Fe₃O₄^[18,19], and graphene^[22], aiming to enhance the electromagnetic shielding effectiveness of epoxy-matrix composites. Some techniques to disperse CNT in epoxy resin/GF composites include sonication with solvent and then deposition by hand lay-up, immersion, conventional painting with paintbrush, or growth of CNT on GF surface^[5,13-21], but there are some problems such as waste of raw materials, thickness control and industrial scalability. Da Silva et al.^[15] showed that resin transfer molded (RTM) epoxy composites reinforced with plain weave GF fabric coated with 2 wt% CNT by roll painting reached 18.3 dB (98.54% of attenuation) in the X-band. Epoxy composites with CNT functionalized with nitric acid have been reported by Phan and coworkers^[16], with 6.6 dB in X-band and electrical conductivity of 5.7×10^{-3} S/cm. Recently, epoxy composites reinforced with nanocarbon-coated glass fibers (NCGF), prepared by a pyrolysis deposition technology, have been proposed for as functional composite materials (FCM) with superior electrical/thermal conductivity and electromagnetic shielding performance^[17]. The electrical conductivity of the FCM containing 20 wt.% NCGF reached 6.68×10^{-6} S/m, while the electromagnetic shielding properties exceeded 20 dB. Yesmin & Chalivendra^[18] investigated the effect of CNT, micro-carbon fibers, and Fe₃O₄ nanoparticles, deposited by paintbrushing on GF fabric, on electromagnetic shielding effectiveness of epoxy-matrix composites in the X-band frequency range and found that the nanoparticles had a major influence on the total shielding effectiveness as well as on the absorption. Airbrushing could thus be an efficient method for CNT deposition on GF to achieve a thin layer with high CNT concentration, since it is an easily controllable process and requires few devices to perform the deposition.

In view of the CNT's potential for EMI shielding, this work aims to evaluate a route for deposition of CNT on GF as thin layers using airbrushing method, looking for a route to impregnate a higher CNT content in polymer composites

and study the influence of this process in EMI shielding properties in the 8.2 – 12.4 GHz frequency range.

2. Materials and Methods

2.1 Materials

The nanocomposites were manufactured using Araldite® LY 1316 epoxy resin (epoxy value = 6.5 eq kg⁻¹) based on diglycidyl ether of bisphenol A (DGEBA), Aradur® HY 951 hardener and Byk®-A 535 degassing agent, all supplied by Huntsman; acetone PA (Merck); Multiwalled carbon nanotubes (MWCNT) from Chengdu Organic Chemicals (outer diameter: 10-30 nm, length: 10-30 μm, purity: 90%) and GF plain weave fabric with an aerial weight of of 300 g/m² supplied by Abcol Brazil Composites Ltda.

2.2 Dispersion and deposition of the MWCNT on GF surface

For deposition of CNT on GF fabric, a formulation containing 50 g of acetone, 0.5% of MWCNT, 6% of epoxy resin and 0.8% hardener (weight percentages relative to acetone), was developed using a tip sonicator (Sonic VCX-750) for 30 minutes and 40% amplitude to disperse CNTs. The mixture was deposited on GF using a commercial airbrush (with a 5 mm nozzle) at a pressure of 4 bar, and then dried at 60 °C in an air-circulating oven for 2 hours. The amounts of MWCNT deposited on the GF surface were fixed in 0.05, 0.10, 0.15 and 0.20 g, meaning final concentrations of 1.3, 2.2, 2.7 and 3.4% of MWCNT, relative to the weight of the GF fabric. To achieve the correct weight of MWCNT, after the drying process the coated GF fabric was weighted, and the airbrushing process repeated until reaching the desired mass.

2.3 Preparation of tricomponent composites

MWCNT/GF/epoxy composites were manufactured by casting in silicone soft molds, using epoxy resin and the GF previously coated with MWCNT. Figure 1 shows a flowchart of the experimental procedure.

Hardener (13% wt) was added to epoxy resin (25 g for each batch) under mechanical stirring for 10 minutes, followed by the addition of the degassing agent under mechanical stirring and vacuum for 30 minutes to remove air bubbles. The resin was poured into soft molds (120 x 80 x 3mm) containing the coated GF and cured at room temperature. The final composition of the composites had (10.25 ± 0.56) wt% of GF, corresponding to ca. 5% v/v, and MWCNT content varying from 0.16 (for GF coated with 1.3%) to 0.64 wt% (for GF coated with 3.4%). Pictures of MWCNT-coated GF and MWCNT/GF/Epoxy composites are shown in Figure 2.

2.4 Characterization

Scanning electron microscopy (SEM) analyses, to evaluate the dispersion of MWCNT after deposition onto GF, were performed in a JEOL JSM 6701F microscope at 15kV. The electrical conductivity of the systems was measured by 4-point probe method using a Keithley 6517A electrometer. EMI shielding measurements were carried out over the

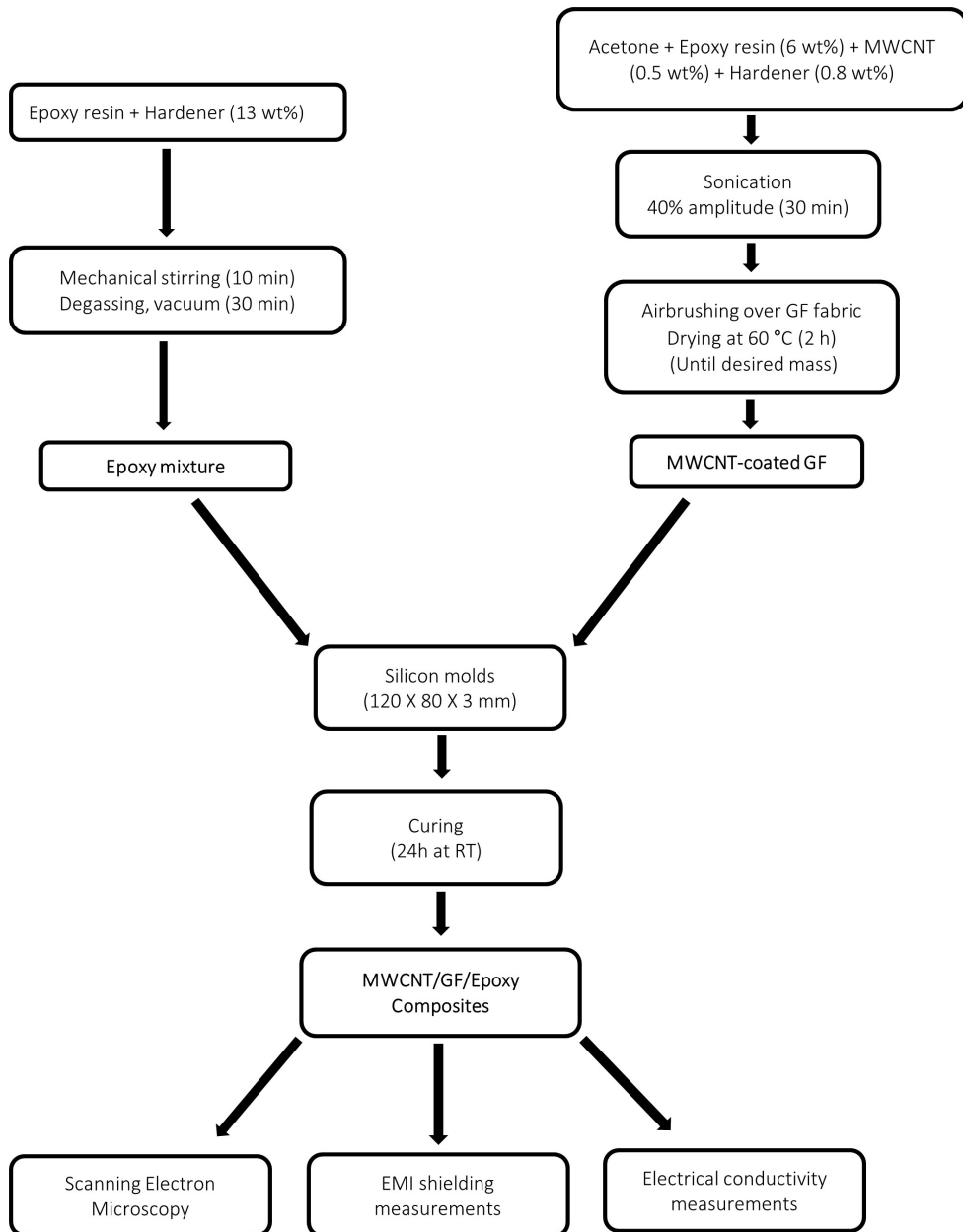


Figure 1. Flowchart of the experimental procedure for the preparation of MWCNT/GF/epoxy composites.

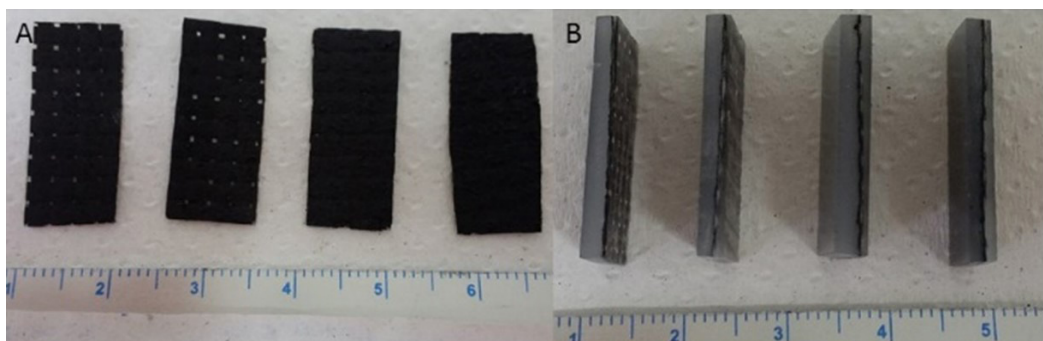


Figure 2. Photographic images of A) MWCNT-coated GF; B) MWCNT/GF/Epoxy composites.

X-band (8.2 – 12.4 GHz). The coated GF and tricomponent nanocomposite samples under test were sandwiched between two X-band waveguide sections which were connected to separate ports of an Agilent Vector Network Analyzer 11644A. S_{11} (reflection) and S_{21} (transmission) parameters were obtained from the measurements, where the numbers indicate the port from which the wave exits and the port where the wave enters, respectively. SE effectiveness were calculated using the Equation 1. Pictures of the equipment and sample holder are shown in Figure 3.

3. Results and Discussions

3.1 Morphological analysis

The surface and fracture surface morphologies were assessed by SEM observation of MWCNT-coated GF. The micrographs in Figure 4 shows a GF fabric surface coated with a thin layer of MWCNT. There are some uncovered

regions on GF, caused by the space between the GF yarns. These regions were covered as the amount of CNT increased. This MWCNT/epoxy layer, which remained onto GF after acetone evaporation, can be better visualized in Fig. 4(b), showing an elevated coverage that is crucial to improve EMI shielding. Zooming in the SEM images of airbrushed surfaces (Figure 5), it can be noted that there are some regions with agglomerated MWCNT, Fig. 2(a), and others where the MWCNT are fairly well dispersed, Fig. 5(b). All nanocomposites presented this behavior. This indicates that conditions used to disperse MWCNT in acetone were, in general, good enough to promote an adequate dispersion of the nanoparticles^[23] over the GF fabric.

When in comparison with some other methods reported in the literature, as growth of CNT on GF surface^[11] and impregnation by paint brush^[24], the airbrushing method can be considered an easy way to impregnate CNT on GF in high concentration and could be a scalable alternative for fabric

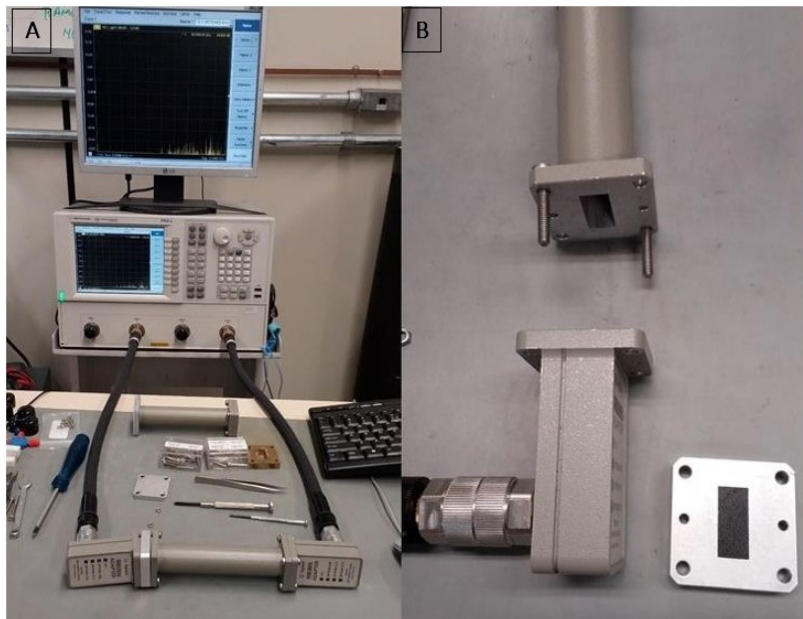


Figure 3. Pictures of A) Vector Network Analyzer for SE measurements; B) Sample holder.

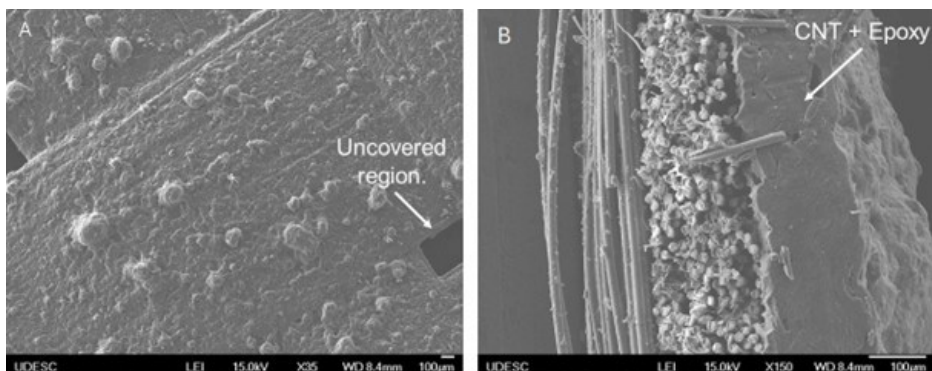


Figure 4. SEM images of GF coated with MWCNT by airbrushing: (a) surface; (b) fracture surface.

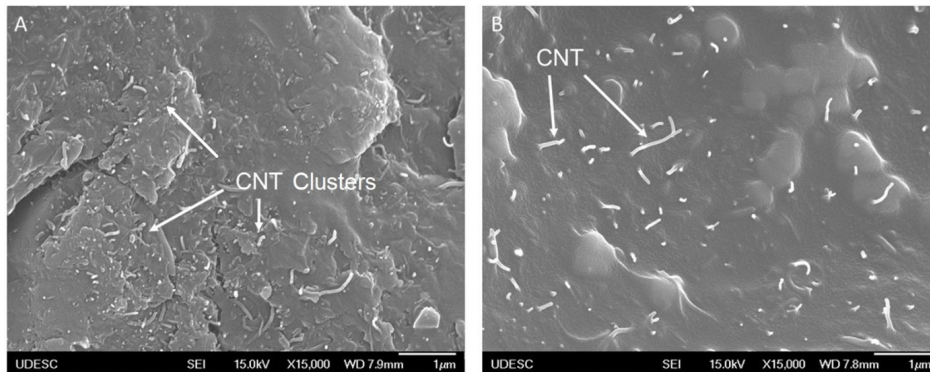


Figure 5. SEM images of GF coated with MWCNT by airbrushing: (a) CNT clusters and (b) dispersed CNT regions after deposition process.

nanocomposites, since there is no need of complex equipment usage for deposition, as *in situ* growth of CNT onto the fibers. In addition, the aspersation method can achieve deeper layers on the GF fabric due to the atomization of the formulation containing CNT, which is not possible using a paint brush.

3.2 Electrical conductivity

Table 1 shows the electrical conductivities of MWCNT/GF/epoxy nanocomposites measured by 4-point probe method. The conductivity values of 6.7×10^{-6} and 1.2×10^{-3} S/cm for the samples with 2.7% and 3.4% of deposited MWCNT, respectively, evidence the formation of a conductive path (percolation) network for higher MWCNT concentrations.

Electrical conduction is supposed to take place by electron hopping/tunnelling across the energy barrier gaps between conducting CNT in a epoxy matrix^[26]. EMI shielding is improved when electrical conductivity is higher, as it increases the barrier between air and the material surface, raising the impedance difference and thus preventing the wave penetration across the material^[11,12,16,27].

3.3 EMI shielding analysis of GF coated with MWCNT

EMI shielding values were measured in terms of reflectance, absorbance, and transmittance of electromagnetic waves (EW) through the nanocomposites. Table 2 summarizes the results obtained for GF coated with varied MWCNT content for an X-band range. The reflectance and absorbance increase with the MWCNT content, since the electromagnetic waves come into contact with more conductive particles and are reflected, and there are more active centers to interact with EW penetrating the material^[28]. On the other hand, the transmittance decreases with the amount of MWCNT since the reflections and absorbance increase, remaining a smaller fraction of the electromagnetic wave to be transmitted.

The effective absorbance (A_{eff}) calculated using Equation 3, at 10.5 GHz, increases more markedly with the MWCNT content, which means that there are more active centers to absorb a lower amount of incident EW, when considering the reflectance values, as shown in Table 3. The same behavior is observed for the other frequencies. A comparison between %R and A_{eff} measurements suggests that the overall mechanism is absorptive for GF coated with 1.3% MWCNT, and reflective for the other compositions.

Table 1. Electrical conductivities of MWCNT/GF/epoxy nanocomposites.

% MWCNT	Electrical conductivity (S/cm)
0 (neat epoxy)*	$\sim 10^{-7}$ *
1.3	8.5×10^{-7}
2.2	3.2×10^{-6}
2.7	6.7×10^{-6}
3.4	1.2×10^{-3}

* Ref. [25].

The shielding effectiveness (SE) can be written as a sum of attenuation by reflectance and absorbance of the EW, as shown in Figure 6. Shielding effectiveness is increased with the MWCNT concentration, and the best result was for 3.4% MWCNT, achieving an attenuation of 73.5% of the incident EW, which means a SE of 5.8 dB. The low SE of the nanocomposite with 1.3% MWCNT can be related to the low amount of nanotubes and voids in the GF surface coverage, which can be noted in Figure 4(a). This means that incident EW pass through the sample, since there are no barriers due to impedance differences between the air and these regions. As the amount of deposited MWCNT increases, the volume of void regions decreases, and the EMI shielding becomes more effective. Increasing the amount of MWCNT is equivalent to introduce more free electron charges that may arise from a percolated CNT-network, which ends up increasing the reflection.

3.4 EMI shielding analysis for tricomponent composites

Table 4 shows the EMI shielding characterization of the neat epoxy resin. The analysis showed that epoxy resin has a reflectance under 35%, due to the impedance mismatch with the air. The absorption is below 6% due to its insulating characteristic where there are no active centers to interact with EW, leading to the transmission of more than 60% of the incident EW.

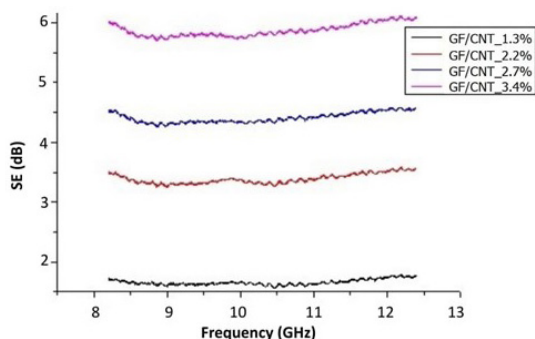
EMI shielding analysis for tricomponent composites (Table 5) showed that the addition of epoxy resin around the GF considerably increases %R, since there is one more barrier against the penetration of EW into the material due to the impedance mismatch. As shown in Table 4, epoxy resin reflects more than 30% of the incident EW,

Table 2. Reflectance (%R), Absorbance (%A) and Transmittance (%T) percentage values from shielding measurements of GF coated with MWCNT.

Frequency (GHz)	1.3% MWCNT			2.2% MWCNT			2.7% MWCNT			3.4% MWCNT		
	% R	% A	% T	% R	% A	% T	% R	% A	% T	% R	% A	% T
8.5	15.3	16.1	68.6	33.2	20.5	46.3	44.2	19.3	36.5	54.3	19.5	26.2
9.5	15.0	16.4	68.6	32.0	21.4	46.6	43.3	19.9	36.8	51.7	22.0	26.3
10.5	14.2	16.2	69.6	31.6	21.4	47.0	42.0	21.2	38.8	50.4	23.4	26.2
11.5	14.3	17.8	67.9	31.9	22.9	45.2	42.0	22.5	35.5	50.2	24.4	25.4

Table 3. Reflectance (%R), absorbance (%A), effective absorbance (A_{eff}) and transmittance (%T) measured at 10.5 GHz for GF coated with MWCNT.

MWCNT (%)	%R	%A	A_{eff} (%)	%T
1.3	14.2	16.2	18.9	69.6
2.2	31.6	21.4	31.3	47.0
2.7	42.0	21.2	36.5	38.8
3.4	50.4	23.4	47.2	26.2

**Figure 6.** Shielding effectiveness (SE) of GF coated with MWCNT.

consequently increasing %R on tricomponent nanocomposites. The attenuation by absorbance for the composites decreased due to the increase of %R in comparison with GF coated with MWCNT.

On the other hand, the effective absorbance (Table 6) was increased, since there is a smaller fraction of the incident EW that penetrates the composite, reaching 45% of effective absorbance at 10.5 GHz for the nanocomposite with 3.4% MWCNT. A comparison between %R and A_{eff} suggests that the overall shielding mechanism is reflective for all nanocomposites.

Shielding effectiveness was increased in MWCNT/GF/epoxy nanocomposites due to addition of epoxy resin (Figure 7). The best results were obtained for nanocomposites containing 3.4% MWCNT, with the attenuation of 83% of incident EW, which means 7.6 dB of EMI shielding effectiveness.

The variations observed in SE with the frequency can be explained by two factors: wave-current interaction and microstructure defects. When an incident wave packet hits the surface of the material, some waves will be reflected by the interaction with conductive particles and impedance mismatch. These reflected waves can interfere with incident

Table 4. Reflectance (%R), Absorbance (%A) and Transmittance (%T) percentage values from shielding measurements of neat epoxy resin.

Frequency (GHz)	%R	%A	%T
8.5	34.4	5.3	60.3
9.5	32.8	4.6	62.6
10.5	31.2	4.6	64.2
11.5	30.8	4.8	64.4

waves at the same spot, causing fluctuations in EMI shielding effectiveness. Presence of defects in microstructure as microcracks and pores within epoxy nanocomposites may also affect the EMI shielding effectiveness, causing multiple reflections^[29]. Other variations in absorption can be explained by differences in the local concentrations of MWCNT onto the surface of GF fabric, due to the manual process of deposition, causing different absorption levels.

Another relevant factor is the electrical conductivity values, as high conductivities magnify the differences between impedances and consequently increases the barrier against the penetration of electromagnetic waves. Only the highest value obtained in this work, 1.2×10^{-3} S/cm for the sample containing 3.4% MWCNT, is at the level considered reasonable for EMI shielding applications^[11,12,17]. This indicates that it is necessary to improve MWCNT concentration and/or its dispersion in order to reach a higher electrical conductivity.

The contribution of multiple reflections for the SE (SE_M) is also a factor to be considered since our SE values are below 10 dB. However, at GHz frequencies the skin depth (δ) for relatively good conductors (as is the case for the composites with 3.4% MWCNT, with conductivities in the order of 10^{-1} S/m) is considerably low and SE_M approaches to zero, indicating multiple reflection loss can be ignored for 2-4 mm thickness samples^[27,30].

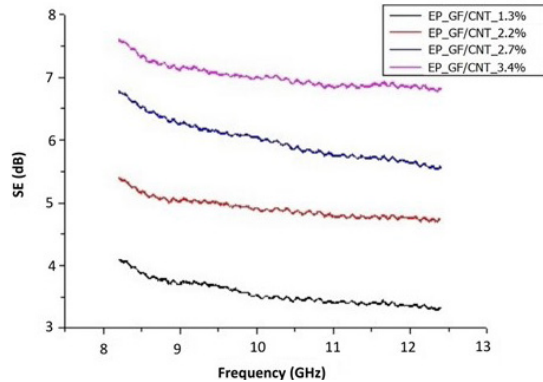
An additional factor to be considered is the thickness of the MWCNT/epoxy layer deposited onto the GF fabric (ca. 100 μm). Arjmand et al.^[27], for example, observed that the

Table 5. Reflectance (%R), Absorbance (%A) and Transmittance (%T) percentage values from shielding measurements of MWCNT/GF/epoxy nanocomposites.

Frequency (GHz)	1.3% MWCNT			2.2% MWCNT			2.7% MWCNT			3.4% MWCNT		
	%R	%A	%T	%R	%A	%T	%R	%A	%T	%R	%A	%T
8.5	48.8	10.4	40.8	55.2	14.4	30.4	64.2	13.6	22.2	67.3	14.3	18.4
9.5	47.3	9.8	42.9	52.8	15.6	31.6	62.7	13.0	24.3	65.6	14.7	19.7
10.5	44.7	13.0	42.3	51.4	15.8	32.8	60.1	14.0	25.9	62.7	16.9	20.4
11.5	44.0	10.2	45.8	51.7	14.9	33.4	58.9	14.3	26.8	62.4	17.0	20.6

Table 6. Reflectance (%R), absorbance (%A), effective absorbance (A_{eff}) and transmittance (%T) measured at 10.5 GHz for MWCNT/GF/epoxy nanocomposites.

MWCNT (%)	R %	A %	A_{eff} %	T %
1.1	44.7	13.0	23.5	42.3
2.2	51.4	15.8	32.5	32.8
2.7	60.1	14.0	35.1	25.9
3.4	62.7	16.9	45.3	20.4

**Figure 7.** Shielding effectiveness (SE) of MWCNT/GF/epoxy nanocomposites.

increase in thickness improves the SE since it is possible to have more interconnected CNT. Moreover, this leads to a possibility to have more active centers in the way of the incident wave, improving the SE^[26-29]. Higher MWCNT coating thickness would thus be important to achieve better EMI performance.

Finally, factors such as nanocomposite manufacturing techniques, dispersion techniques and nanoparticle type can lead to different SE values, demanding studies to find the best combination of these factors to improve the effectiveness of EMI shielding.

4. Conclusions

The results show that the airbrushing method has proved to be a promising option as a route to deposit carbon nanotubes on glass fiber fabrics, to obtain thin and light composites. Evaluations of reflectance, absorbance and effective absorbance showed a reflective value higher than effective absorbance, suggesting that the reflection mechanism was dominant in MWCNT/GF/epoxy composites. EMI shielding

measurements indicated that the thin tricomponent material can be potentially used as shielding for electromagnetic waves, achieving 83% of electromagnetic wave attenuation for composites with 3.4 wt% MWCNT.

5. Author's Contribution

- **Conceptualization** – Willian Rodrigo Schuster; Sérgio Henrique Pezzin; Fernando Humel Lafratta.
- **Data curation** – NA.
- **Formal analysis** – Willian Rodrigo Schuster.
- **Funding acquisition** – Sérgio Henrique Pezzin; Fernando Humel Lafratta.
- **Investigation** – Willian Rodrigo Schuster; Fernando Humel Lafratta.
- **Methodology** – Willian Rodrigo Schuster; Fernando Humel Lafratta.
- **Project administration** – Fernando Humel Lafratta.
- **Resources** – Sérgio Henrique Pezzin; Fernando Humel Lafratta.
- **Software** – NA.
- **Supervision** – Sérgio Henrique Pezzin; Fernando Humel Lafratta.
- **Validation** – Willian Rodrigo Schuster; Fernando Humel Lafratta.
- **Visualization** – Willian Rodrigo Schuster; Fernando Humel Lafratta.
- **Writing – original draft** – Willian Rodrigo Schuster.
- **Writing – review & editing** – Sérgio Henrique Pezzin; Fernando Humel Lafratta.

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