

Performance Prediction of Microwave Absorbers Based on POMA/Carbon Black Composites in the Frequency Range of 8.2 to 20 GHz

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ABSTRACT: This paper presents a comparative study involving experimental and numerical behaviors of radar absorbing materials (RAM), based on conducting composites of poly(o-methoxyaniline) (POMA) and carbon black (CB). Samples of POMA/CB in epoxy resin matrix were prepared. First, these samples were experimentally characterized by electric permittivity and magnetic permeability measurements in the frequency range of 8.2 to 12.4 GHz. Afterwards, a linear extrapolation of these electromagnetic parameters until 20 GHz was carried out. These amounts were used as parameters for a set of simulations, developed from numerical implementation of theoretical predictions. The main advantage of the performed simulations is to know the behavior of the POMA/CB/epoxy resin as RAM in a wide range of frequencies (8.2-20 GHz), previously to the experimental work. The validation of the simulations with experimental reflection loss measurements showed a good fit and allowed predicting the material behavior as RAM. The results show that the studied RAM presents good return loss values in different frequencies, for example, -32 dB (~99.95% of absorption) at 14.6 GHz and -18 dB at 19.2 GHz, for samples with 7 and 9 mm-thickness values, respectively. The simulation tool used in this study was adequate to optimize the RAM production, contributing to the reduction of development costs and processing time of this kind of material.

KEYWORDS: Poly(o-methoxyaniline), POMA, Microwave absorber, RAM.

INTRODUCTION

Associated with the increasing use of electromagnetic waves in the GHz range in equipment and devices used in telecommunication, military and medical areas, there is a need to monitor the effects of electromagnetic interference produced by the radiation generated (Feng *et al.* 2007; Folgueras *et al.* 2010). In seeking to eliminate or control these effects we observe the growing number of studies involving the use of so-called microwave absorbing materials or radar absorbing materials (RAM) (Gama *et al.* 2011; Dias *et al.* 2012).

RAM are so named because they have properties that allow them to exchange the energy of the incident electromagnetic radiation by thermal energy (Dias *et al.* 2012; Wang *et al.* 2017). For this phenomenon of energy exchange to take place, is required to set the proper values of impedances of such materials, in order to favor the propagation of the incident wave inside thereof, and not its reflection. Knowing that satisfies this condition promotes partial or almost total electromagnetic wave attenuation by physical and/or physico-chemical mechanisms (Feng *et al.* 2007). In pursuit of this condition, the impedance values of the absorbing materials, mainly at the interfaces, should be adjusted to approach the maximum

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values of the impedance of free space (377Ω) (Micheli *et al.* 2014). For this, the electric permittivity (ϵ), the magnetic permeability (μ) parameters and the thickness of a material are fundamental physical characteristics, determining the resulting attenuation of the electromagnetic wave incident on RAM (Gama *et al.* 2011).

The absorbing materials are divided into two major groups, namely, one that has dielectric losses and the one characterized by magnetic losses (Oh *et al.* 2004). Among the additives commonly used in dielectric absorbers processing, we have carbon black and intrinsically conductive polymers. In the case of magnetic absorbers, we have any ferrite and carbonyl iron (Dias *et al.* 2012). Among the conductive polymers studied as microwave absorbers, we have polyaniline and its derivatives, such as poly(o-methoxyaniline) (POMA) (Sanches *et al.* 2014).

It is worth mentioning that the impedance setting of a RAM involves much experimental work, in search of the best formulation of the components involved in this processing. In this case, it is necessary to define the additives which act as microwave absorbers and the polymer matrix used to both anchor the additives and confer the final shape of RAM (Balanis 2012). Moreover, this area still requires a thorough and complex experimental work on the electromagnetic characterization of the developed RAM. It is also known that the performance of these materials is also related to the frequency of the incident electromagnetic wave and the thickness of the processed materials, as shown in Eq. 1 (Balanis 2012):

$$Z = \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh\left(j \frac{2\pi}{\lambda} t \sqrt{\mu_r \cdot \epsilon_r}\right) \quad (1)$$

where: Z is the normalized input impedance of the material; t is the sample thickness; m is the magnetic permeability; e is the electric permittivity of the material, and λ is the wavelength of the incident plane wave in free space. The complex parameters ϵ and μ are expressed by Eqs. 2 and 3, respectively, considering the real and imaginary components of the permittivity (ϵ' , ϵ'' , respectively) and the permeability (μ' and μ'' , respectively).

$$\epsilon = \epsilon' - j\epsilon'' \quad (2)$$

$$\mu = \mu' - j\mu'' \quad (3)$$

Equation 4 gives the intrinsic impedance of the material, considering the normal incidence of the electromagnetic wave on the same, in a waveguide. By means of the normalized impedance, it can obtain the reflection loss (RL) and the impedance matching of the material positioned on a metal plate, Eq. 4 (Gama *et al.* 2011):

$$RL(dB) = 20 \log \left| \frac{Z-1}{Z+1} \right| \quad (4)$$

When the appropriate setting of the complex parameters of permeability and permittivity is reached, the RL is maximal for a given frequency and a given material thickness (Vinayasree *et al.* 2013).

The literature has shown some contributions in the performance prediction of RAM in wider ranges of frequencies and in different thicknesses supported by computational tools (Gama *et al.* 2011). This procedure is often based on values of permittivity and permeability extrapolated from experimental data collected in narrower frequency bands. These studies aim to reduce costs, processing time and the risk involved in the establishment of formulations attuned to the frequency range to be attenuated. However, it is important to mention that this procedure is also the subject of discussion among researchers, due to the complexity involved in the experimental process and the used parameters in the extrapolation of the complex parameters of permittivity and permeability for a wide range of frequencies (Dantas *et al.* 2015).

It is known that the complex parameters of permittivity and permeability exhibit a monotonic decrease with increasing frequency, approaching a linear behavior (Kao 2004). However, this assumption may not be as well behaved and the fit of the actual behavior of the material can deviate. Added to this, there are other factors that can contribute to other deviations of RAM behavior prediction, as the consideration of the material under study is homogeneous and that the calibration of equipment that measures the complex parameters (vector network analyzer), the adjustment of the waveguide flanges and the positioning of the sample in the waveguide are error-free. Thus, work prediction should always be considered with reservations and, whenever possible, be validated with experimental data, similarly to what occurs in other fields that use simulation and prediction.

In this sense, this study aims to present results of numerical predictions of the microwave attenuation performance of a composite based on POMA/carbon black in epoxy resin, in the frequency range of 8.2 to 20 GHz, correlating the behaviors numerically calculated with those experimentally determined.

MATERIALS AND METHODS

The materials used in this study were o-anisidine from Aldrich, with 99% of purity, ammonium persulfate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$) from Merck as oxidant, with 98% purity, $1.0 \text{ mol}\cdot\text{L}^{-1}$ HCl solution from Merck, with 37% minimum content of acid, carbon black type XC72R from Cabot company and a commercial bicomponent epoxy resin, type Araldite, attending the proportion 2:1 of resin:hardener (wt/wt).

The POMA used in this study was synthesized based on the work of Mattoso and Bulhões (1992) and presented in detail in a previous study (Pinto and Rezende 2012). This synthesis was performed in the presence of carbon black, based on the work of Wu *et al.* (2008), resulting in a composite named POMA/CB. For this, 10 wt% of carbon black was added to 200 mL of $1.0 \text{ mol}\cdot\text{L}^{-1}$ HCl solution containing 6.2 mL of o-anisidine freshly distilled and stabilized at 0°C . This mixture was left under agitation. Then, in a beaker were dissolved 2.88 g of ammonium persulfate in 50 mL of $1.0 \text{ mol}\cdot\text{L}^{-1}$ HCl solution. The reaction medium containing the o-methoxyaniline was maintained at 0°C during the dripping of the acid solution of ammonium persulfate. The total time of synthesis was 120 min (Mattoso and Bulhões 1992). Afterwards, the POMA/CB was washed and dried under vacuum.

The samples were processed in an epoxy resin matrix in the proportion of 20 wt% of the POMA/CB composite, respectively, through mechanical mixing of the components. This mixture was poured into a mold of dimensions of $23 \times 10 \times 9.0 \text{ mm}$ that corresponds to the exact dimensions of the sample holder used in the electromagnetic characterization. Epoxy resin curing was carried out at room temperature for a period of 24 h.

The real and imaginary values of ϵ and μ of the studied sample were obtained, in triplicate, based on ASTM D5568-01. Using a vector network analyzer from Agilent Technologies, model PNA-L N5230C, with four ports, a frequency generator between 300 kHz and 20 GHz, low loss cables, connectors and a high-precision rectangular waveguide adapter, being the adapter also from Agilent Technologies, model 00281-60016 OPTION 006. Figure 1 shows the apparatus and devices used in this work. The calculation of complex parameters of ϵ and μ was carried out with the aid of the software 85017E from Agilent, based on the Nicolson-Ross model (Nicolson and Ross 1970).

The method used for the extrapolation of the complex permittivity and permeability parameters in larger frequencies is based on a truncated Kramers-Kronig relation, based on finite frequency data (Dantas *et al.* 2015). Considering a few assumptions, such as the behavior of the loss tangent and the overall nature of corrections, the used method is robust within a few percentage of relative error, if the assumed hypotheses hold at the extrapolated frequency range. This method is described in the literature (Dantas *et al.* 2015).

A Java application was used to facilitate the mapping of the electromagnetic parameters of interest for the radar absorbing materials design. The computational tool developed, named "RFE" (an acronym in Portuguese for Reflectivity, Frequency and Thickness), implements directly Eqs. 1-4. In terms of computational time for the numerical solution of these equations, the application shows a very good performance, which in practical terms allows exploration in "real time" of several thickness settings dynamically.

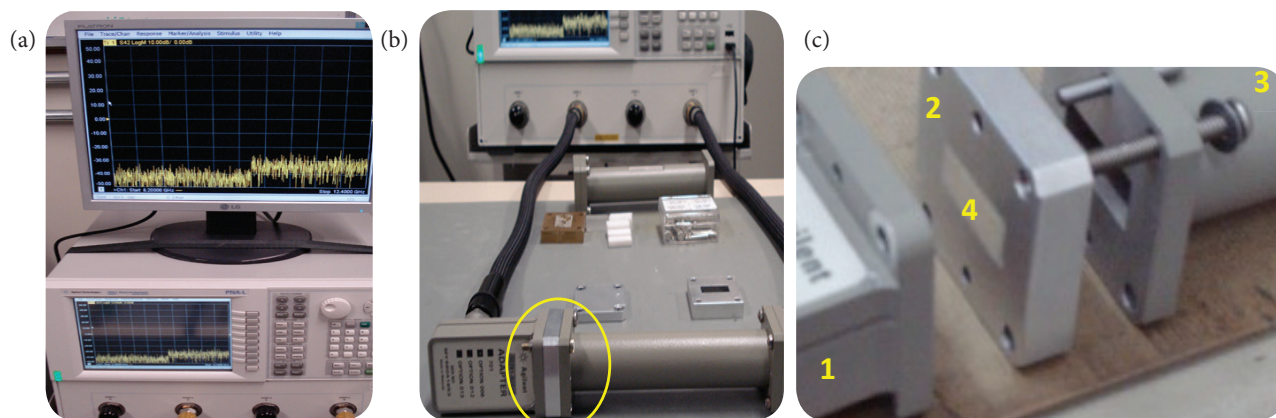


Figure 1. (a) Vector network analyzer. (b) Waveguide and adapters. (c) Details of the waveguide: (1) adapter of port 1; (2) sample holder; (3) waveguide section of port 2; (4) sample.

The RFE application outputs the attenuation as a function of the constitutive properties of the material and the incoming wave frequency. Specifically, the application receives as static input parameters the real and imaginary components of the electric permittivity (ϵ' and ϵ'' , respectively) and the magnetic permeability (μ' and μ'' , respectively), both as a function of frequency, and gives as output the reflectivity in dB, as a function of the range of frequencies of interest, according to the material thickness (in mm). Graphical user interface (GUI) windows were implemented in the code. The output plot of the attenuation behavior as a function of frequency is updated in “real time” from the thickness variation, which can be freely adjusted by the user. This feature facilitates a dynamic analysis of the attenuation behavior, allowing the user to compose a set of different scenarios for the RAM behavior.

RESULTS AND DISCUSSION

ELECTROMAGNETIC CHARACTERIZATION

Obtaining the complex components of the electric permittivity and magnetic permeability assists in understanding the phenomena of absorption of electromagnetic radiation by RAM, as mentioned in the literature (Singh *et al.* 1999). Figure 1 shows the values of real and imaginary components of the permittivity and permeability in X-band, for the POMA/CB composite (20 wt%) in epoxy resin. First, it is observed that the values of the complex electric permittivity are greater than the magnetic permeability; for example, at 10.2 GHz, the parameters ϵ' , ϵ'' , μ' and μ'' are equal to 4.4911, 0.3535, 1.0332 and 0.0791, respectively (Table 1). This behavior is expected, knowing that the absorbing material under consideration is the dielectric type, since the magnetic parameters are typical of this class of materials (μ' next to 1 and μ'' next to zero) (Vinoy and Jha 1996). Thus, the electric permittivity presents more significant values, with the storage component (ϵ') higher.

Figure 2 also shows that the four complex parameters experimentally measured are practically constant in the frequency range of 8.2 to 12.4 GHz. The observed behavior for the complex parameters is mentioned in the literature (Singh *et al.* 1999) and it is attributed to the fact that these quantities are less influenced in this frequency band (GHz). The opposite behavior is observed at lower frequencies (Hz e MHz), where the variations are more pronounced and significant (Hong *et al.* 2015). Since the values of the four components (ϵ' , μ' , ϵ'' and μ'') are presented on the same scale (Fig. 1 a,b), the differences become less obvious, especially for the imaginary components (ϵ'' and μ''). These values are proportionally smaller than those presented by the real components (ϵ' and μ'). Table 1 shows some values obtained experimentally for different frequencies, among the points collected for each of the real and imaginary components of ϵ and μ , in the frequency range of 8.2 to 12.4 GHz.

Figure 3 shows the extrapolated values of real and imaginary components of permittivity and permeability from 12.4 GHz to 20 GHz. The analysis of this figure and according to Table 1 it can be seen that the real and imaginary values of permittivity

suffer a decrease, in accordance to the literature (Hong *et al.* 2015), while the real and imaginary components of the magnetic permeability keeps nearly constant with a slight increase in higher frequencies.

Overall, Table 1 shows that the real components ϵ' and μ' show less variation between the measured values. The observed behavior shown by ϵ' , that is, its decrease with increasing frequency, is expected and described in the literature (Singh *et al.* 1999). On the other hand, it is observed a slight increase of μ' with the frequency increase. The literature has shown the variation of the magnetic and electrical properties of conducting polymers as a dependence of the conditions of synthesis and polymer doping (Sanches *et al.* 2013). The data obtained in this study show that these properties present a slight variation with the frequency increasing. However, the imaginary components ϵ'' and μ'' are smaller, although with the most significant variations mainly in ϵ'' . In the case of ϵ'' , it is observed a slight increase of this parameter. This behavior is described in the literature (Kao 2004) and it is attributed to losses due to dipole oscillation of the molecule in this frequency range. In a similar way, it is verified a slight increase of μ'' with the frequency increasing, suggesting that the magnetic property of conducting polymers changes with the frequency increasing in the GHz range.

The four components (ϵ' , ϵ'' , μ' and μ'') in the frequency range of 8.2 to 12.4 GHz (experimental data) and above 12.4 to 20 GHz (extrapolated data) were used in the performed simulations. Regarding that, complex parameters above 12.4 GHz until 20 GHz were extrapolated from experimental data (8.2-12.4 GHz), as previously described. This is the boundary condition adopted for this study.

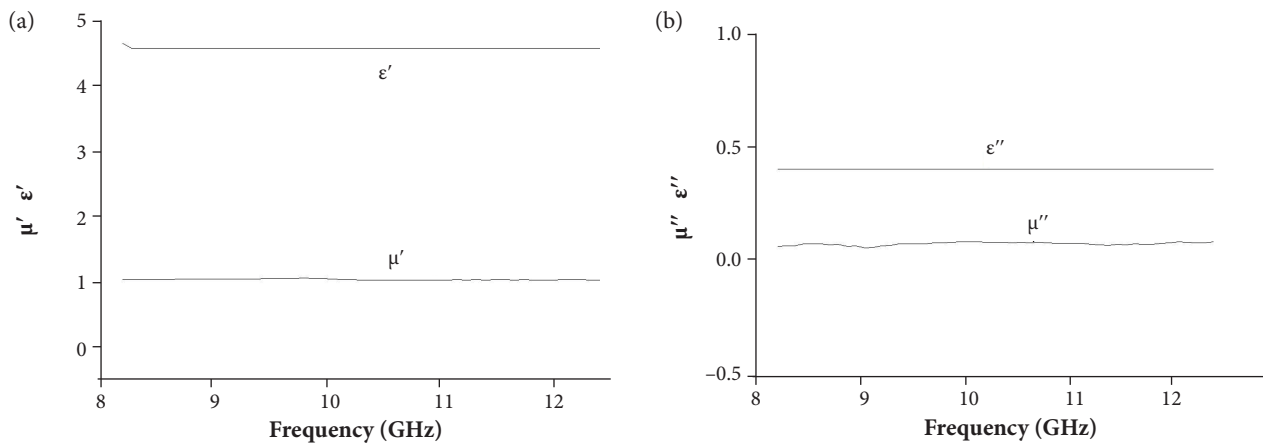


Figure 2. Experimental values of complex parameters ϵ and μ as function of frequency (8.2-12.4 GHz) of the POMA/CB (20% wt) in epoxy resin (a) real components [ϵ' and μ'] and (b) imaginary components [ϵ'' and μ''].

Table 1. Experimental and extrapolated values of real [ϵ' and μ'] and imaginary [ϵ'' and μ''] components of POMA/CB (20 wt%) in epoxy resin in different frequencies.

Data	Frequency (GHz)	ϵ'	ϵ''	μ'	μ''
Experimental	8.2	4.6773	0.5943	1.0386	0.0612
	10.2	4.4911	0.3535	1.0332	0.0791
	12.2	4.5961	0.2790	1.0316	0.0764
Extrapolated	14.2	4.5373	0.1901	1.0610	0.0849
	16.2	4.5241	0.1468	1.0721	0.0909
	18.2	4.5135	0.1164	1.0843	0.0983
	20.0	4.5029	0.0906	1.0935	0.1045

Figure 4 shows an experimental reflectivity curve for the sample containing 20 wt% of POMA/CB in epoxy resin, with 9.0 mm-thickness, in the frequency range of 8.2 to 12.4 GHz and others resulted from simulations using the RFE algorithm.

The reflectivity curve obtained experimentally (Fig. 4) shows that the RAM sample with 9.0 mm-thickness behaves as an efficient microwave absorber, with a maximum RL value of -24 dB ($> 99\%$ of absorption according to Lee, 1991) at 11.6 GHz. The comparison of this curve with that one simulated for a sample with the same thickness (Fig. 4) shows that the two curves present a good fit of experimental and simulated data. The small differences observed between these two curves are attributed to any error of the complex parameters experimentally measured in the X band, any possible irregularities in thickness and/or flatness of the sample, as cited in the literature (Gama *et al.* 2011).

Despite the small differences observed between the experimental and simulated curves (for the 9.0 mm-thickness specimen), it is possible to affirm that the tool used is robust and useful to predict the material behavior as a microwave absorber, given information related to the type of absorbing material (resonant or broadband), maximum frequency attenuation and its attenuation efficiency, previously to laboratory work. Thus, from the comparison of experimental and simulated curves in the

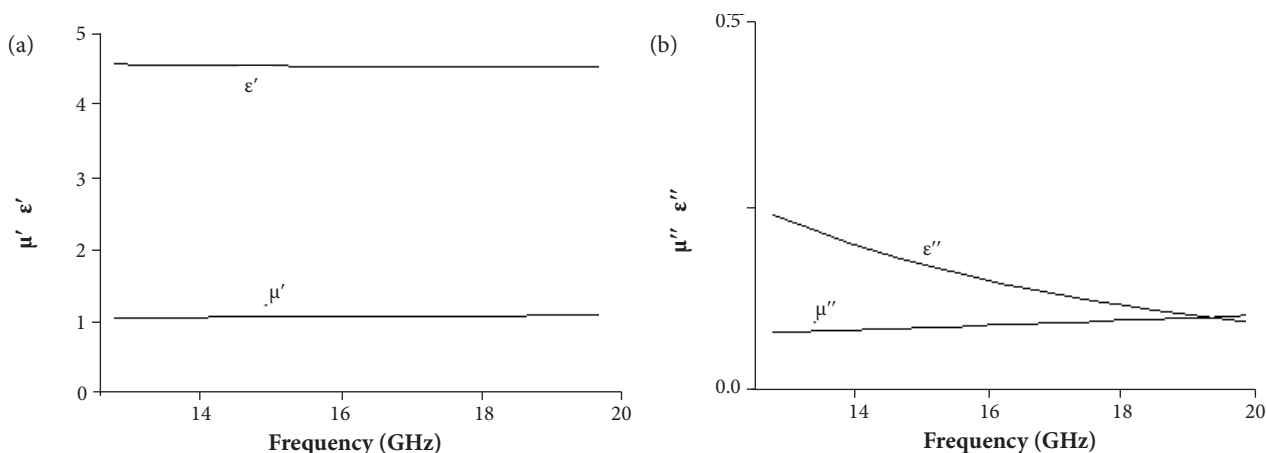


Figure 3. Extrapolated values of complex parameters of ϵ and μ as a function of frequency of POMA/CB (20 wt%) in epoxy resin (8.2 to 20 GHz): (a) real components (ϵ' and μ') and (b) imaginary components (ϵ'' and μ'').

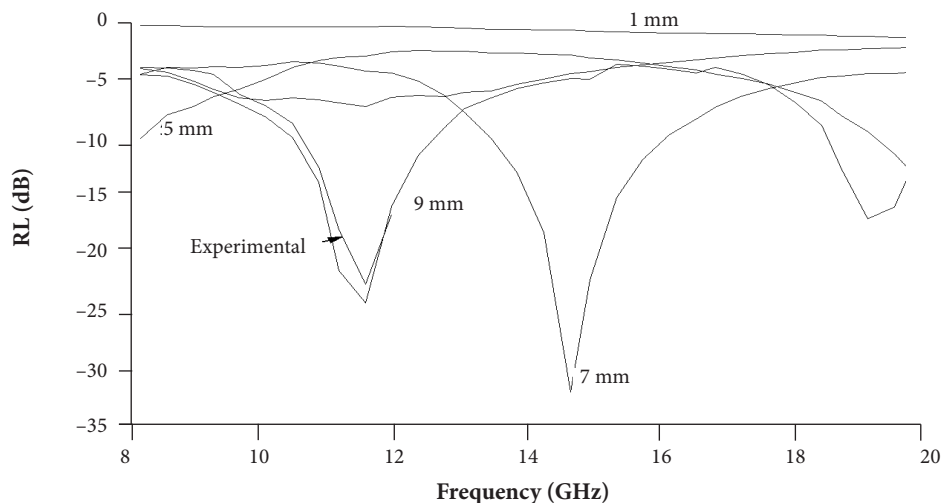


Figure 4. Experimental (in the frequency range of 8.2 to 12.4 GHz) and simulated RL curves for the sample of POMA/CB (20 wt%)/epoxy resin, with 1, 3, 5, 7 and 9 mm of thickness, in the frequency range of 8.2 to 20 GHz.

X band (8.2-12.4 GHz), it is possible to affirm that the algorithm used supports credible studies involving RAM prediction in other frequency bands and with different thicknesses, saving time and financial resources.

Extrapolated values of ϵ' , ϵ'' , μ' and μ'' in the frequency range of 12.4 to 20 GHz were obtained. From these data and those obtained experimentally (8.2-12.4 GHz), simulations were performed for the samples with thicknesses of 1, 3, 5, 7 and 9 mm, in the frequency range of 8.2 to 20 GHz (Fig. 4).

Table 2 summarizes the maximum RL values in dB and their frequencies of occurrence. From Table 2 and Fig. 4 we observe that a same RAM sample can present the maximum attenuation in different frequencies, with the thickness varying, as described in Eq. 1. Similar behavior is reported in the literature for magnetic absorbers based on carbonyl iron (Gama *et al.* 2011, Singh *et al.* 1999). Figure 4 and Table 2 show significant attenuation values (at least -10 dB, corresponding to 90% of attenuation, according to Lee 1991) over the frequency range of 8.2 to 20 GHz, for the sample thicknesses of 5.0, 7.0 and 9.0 mm. We can see, for example, RL values of -32 dB (> 99.9% of attenuation) at 14.7 GHz for the sample with 7.0 mm and -14 dB for the sample with 5.0 mm thicknesses at 20.0 GHz, respectively.

Table 2. Maximum attenuation and their frequencies of occurrence obtained from simulated RL curves for the sample of POMA/CB (20 wt%) in epoxy resin.

Thickness (mm)	Maximum attenuation (dB)	Frequencies of maximum RL (GHz)
3.0	-7.5	11.5
5.0	-14	20.0
7.0	-32	14.6
9.0	-24	11.6

Figure 4 also shows that the 9.0 mm thickness sample presents two peaks of resonance at 11.6 GHz and 19.2 GHz, respectively, with the periodicity of 7.6 GHz between these two maximum of attenuation. This phenomenon is attributed to the wave phase cancelling, associated with multiple wavelengths, which in turn is related to the physical thickness of the sample (Balanis 2012).

The results obtained in this work show that the sample of POMA/CB (20 wt%) in epoxy resin behaves as a microwave absorber in the frequency range of 8.2 to 20 GHz. The results obtained in the simulations support new experimental works of processing of microwave absorbers tuned in preselected frequencies.

CONCLUSION

In this study, we used an algorithm to simulate the behavior of microwave absorber based on POMA/carbon black in large frequency range (8.2–20 GHz) and with different thicknesses. For this, it was assumed the linearity of the experimental data of ϵ and μ in the frequency range of 8.2 to 12.4 GHz to obtain the extrapolated values in the frequency range of 12.4 to 20 GHz, which, in turn, were used in the predictions. The simulation validation was carried out with experimental measurements of reflection loss in the frequency range of 8.2 to 12.4 GHz and the results show a good convergence and fit between experimental and simulated RL curves. Thus, the use of this tool proved to be useful to predict the microwave absorber behavior with different thicknesses and in wide frequency range, minimizing costs and time devoted to process absorbers tuned to the selected frequencies. The results of this study also show that the composite based on POMA/carbon black presents an excellent potential as RAM in the frequency range of 8.2 to 20 GHz. Results of -32 dB at 14.6 GHz and -24 dB at 11.6 GHz are observed in the performed simulations, considering samples with 7.0 and 9.0 mm-thickness values, respectively.

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AUTHOR'S CONTRIBUTION

Experimental investigation, Discussion & Writing, Pinto SS; Discussion & Review, Rezende MC.

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