Study of the influence of Carbonyl iron particulate size as an electromagnetic radiation absorbing material in 12.4 to 18 GHz (K_u) Band

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***Abstract*— This paper presents the influence of different sizes of carbonyl iron particles on the reflectivity measurements of Radar Absorbing Material (RAM). The electromagnetic characterization was performed with a vector network analyzer and a rectangular waveguide in the frequency range of 12.4 to 18GHz (Ku Band). The influence of different parameters such as thicknesses, particle sizes and concentration of carbonyl iron were evaluated. Reflectivity results showed the influence of these parameters on the performance of the RAM. The best reflectivity values (~ -18 dB) were obtained for samples with 60 wt% concentration and 5 mm thickness. We provide information about significantly reflection loss improvement by simply controlling carbonyl iron particulate size.**

***Index Terms*—** carbonyl iron filler, Ku band, microwave absorber.

I. INTRODUCTION

Radar Absorbing Material (RAM) is a type of material designed to attenuate electromagnetic radiation on specific frequencies. Many researches have been made about RAM due its countless applications in electromagnetic compatibility and interference reduction. These materials can be applied on communication systems, modern electronic devices, anechoic chamber, military stealth technology, and so on [1]. RAMs can be produced in different forms, such as paints or thin films [2]. Usually, RAMs are composite materials made with polymer (matrix) and absorptive material (mean). In the literature, materials with dielectric and/or magnetic losses are commonly used as means. In this matter, materials like ferrite, carbonyl iron (CI), carbonaceous materials and conductive polymers has advantages over lossless materials [2],[3]. Despite their high specific mass, composites made with ferrites or CI have advantages like thin thickness and broadband frequency absorption because of iron on their compositions [4]. Carbonyl iron has a relatively low electrical conductivity, a high Curie temperature, and a high saturation magnetization. These properties make CI a good candidate to be used as absorption mean, especially in the frequency range between 2 -18 GHz [5].

The effectiveness of the absorptive material contributes to the reflectivity losses that is determined

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through the obtained values of the complex permittivity and permeability. However, the control of the complex permittivity and permeability are obtained by the addition of magnetic and dielectric additives. Understanding the effects caused by RAM processing require the investigation of several parameters, but the main ones are absorbent mean and matrix properties [6]. Thus, this paper presents the influence of CI particle size on RAM absorption parameter over the frequency range from 2 to 18 GHz. Here, we propose the reflectivity control using different CI particle sizes, where we can significantly improve the absorption properties.

II. EXPERIMENTAL

A. Material and sample preparation

Composites were prepared with commercial CI powder from BASF GmbH as additive. Commercial bi-component silicone was used as matrix.

Carbonyl iron powder was separated into different particle sizes (Pe) using sieve shaker with three different sieves: 25 < Pe <53 μm; 53 ≤ Pe <63 μm and; Pe> 63. After 30 minutes sieving, CI powders were separated following the mass ratio of 40:60, 50:50 and 60:40 of CI concentration over silicone. Sample thickness ranged from 1 mm to 5 mm, but since the best results were obtained for 2, 3 and 5 mm, our discussions were based on these thicknesses. Table 1 summarizes all samples discussed in this paper.

TABLE I. SIZE OF THE PARTICULATES

Thickness of samples (mm)	**Size of CI particles Pe (μm)**			**CI Concentration**		
2	Pe >63	53 < Pe < 63	25 < Pe <53	40%	50%	60%
3	Pe >63	53 < Pe < 63	25 < Pe <53	40%	50%	60%
5	Pe >63	53 < Pe < 63	25 < Pe <53	40%	50%	60%

Composites were manually homogenized. Each sample of silicone-CI composite was mixed with ~0.6 ml catalyst until the beginning of reaction, which could be noticed by bubble formation and viscosity increasing. Before sample hardening, mixture was moved to K_u-band mold with 15.7 mm width and 7.9 mm height. Full catalyst reaction of composite mixtures were about 30 minutes.

B. Morphological and electromagnetic characterization

Crystalline phases of samples were investigated through X-Ray Diffraction (XRD). A Panalytical X'Pert Powder system equipped with a CuKα (λ = 0.154 nm) was used. Composites were scanned from 20° to 90° with sampling intervals of 0.02°. Carbonyl iron particulate sizes were analyzed with a Field Emission Gun Scanning Electron Microscope (FEG-SEM). The equipment used was a TESCAN

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– Vega 3 operating with secondary electron detection.

Electromagnetic characterization was performed with a K_u-band rectangular waveguide (Agilent WR-62 P11644A) coupled on a 50 GHz PNA-L vector network analyzer (Keysight N5232A). Electromagnetic properties were measured in the K_u band, i.e., from 12.4 to 18 GHz. Through scattering parameters (S parameters) it was possible to comprehend the interaction of electromagnetic wave on samples and calculate the permittivity and permeability of materials. The method used to obtain the electromagnetic properties of samples was Nicolson Ross Weir (NRW), which is also called Transmission and Reflection (TR) method [7]. Fig. 1 illustrates the transmitted (S21 and S12) and reflected (S11 and S22) waves that can be measured with PNA-L. It is through these parameters that permittivity and permeability are extracted, allowing the material characterization over a frequency range.

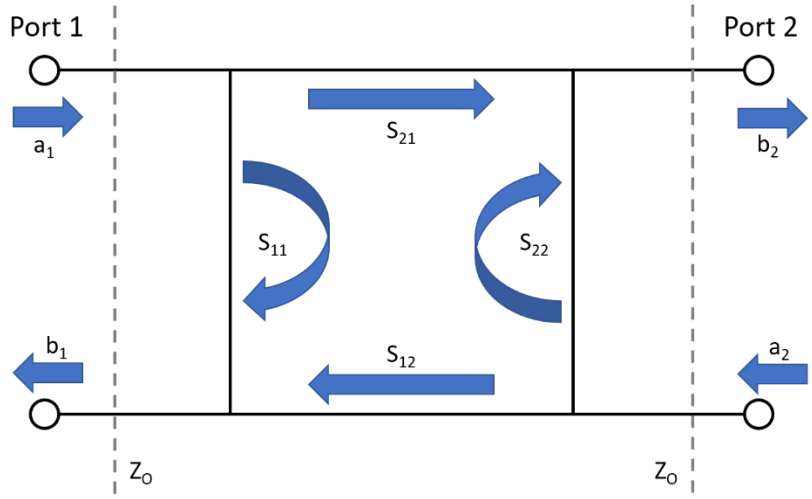

Fig.1 Schematic representation of a device with two ports [8]. The symbols a and b represent the amplitude of the incident wave and the response.

The electromagnetic properties of a material, i.e., the complex relative permittivity (**εr = ε '- jε''**) and permeability (**μr = μ' - jμ''**) are determined by S-parameters measured over a frequency range [9]. The real part of permittivity and permeability (**ε'** and **μ'**) represents the capacity of energy storage in the sample, while the imaginary parts (**ε''** and **μ''**) represent the electric and magnetic energy losses [10].

III. RESULTS AND DISCUSSIONS

A. Field Emission Gun Scanning Electron Microscopy

Particulate sizes of pure carbonyl iron analyzed with FEG-SEM are presented in Fig. 2. All particulate sizes obtained after sifting presents spheres of different sizes. Although spheres apparently have similar sizes, there are different agglomeration of them over sifted powder. It is interesting to note that particulate sizes between 25 μm and 53 μm, Fig. 2a, presents grains more dispersed than

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particulates sizes bigger than 63 µm. This agglomeration caused the particle separation through the sieves, where the average size of spheres clusters was estimated based on the sieve weft spacing.

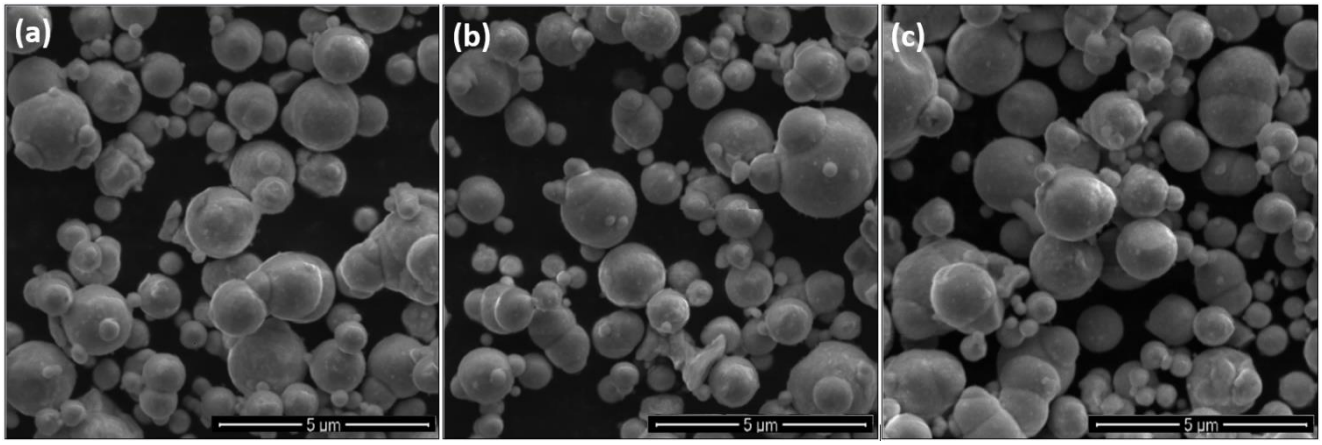

Fig.2 FEG-SEM images obtained for the three particulate sizes with 15.000 of magnifications of (a) 25 < Pe < 53, (b) 53 < Pe < 63, and (c) Pe > 63 µm.

B. X-Ray Diffraction

XRD analysis of CI confirmed the same cubic crystalline lattice for all three separated particulate powder. The same result was obtained with CI powder with no sifting treatment, which means that crystalline structure was maintained after sifting and there was not a specific element responsible for agglomeration. Well defined peaks associated with (110), (200) and (211) planes were detected at 2θ = 44.6º, 64.9º and 82.3º, respectively. All peaks are related to the α-Fe phase of the CI [9][10].

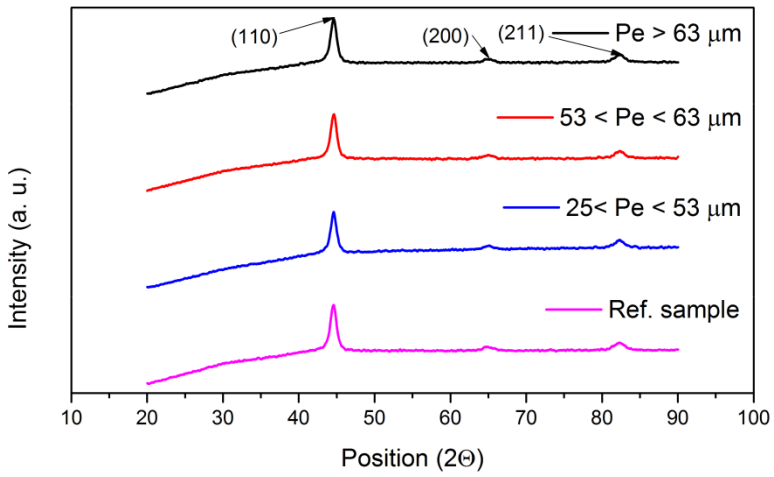

Fig.3 X-ray diffractograms of CI samples as a function of particle size (a) 25 <Pe <53 µm (b) 53 <Pe <63 µm (c) Pe> 63 µm (d) without sieving

C. Electromagnetic Characterization - Electrical Permittivity and Magnetic Permeability

Fig. 4a – 4c represent the real permittivity (**ε'**) of composites with CI concentrations of 40%, 50%

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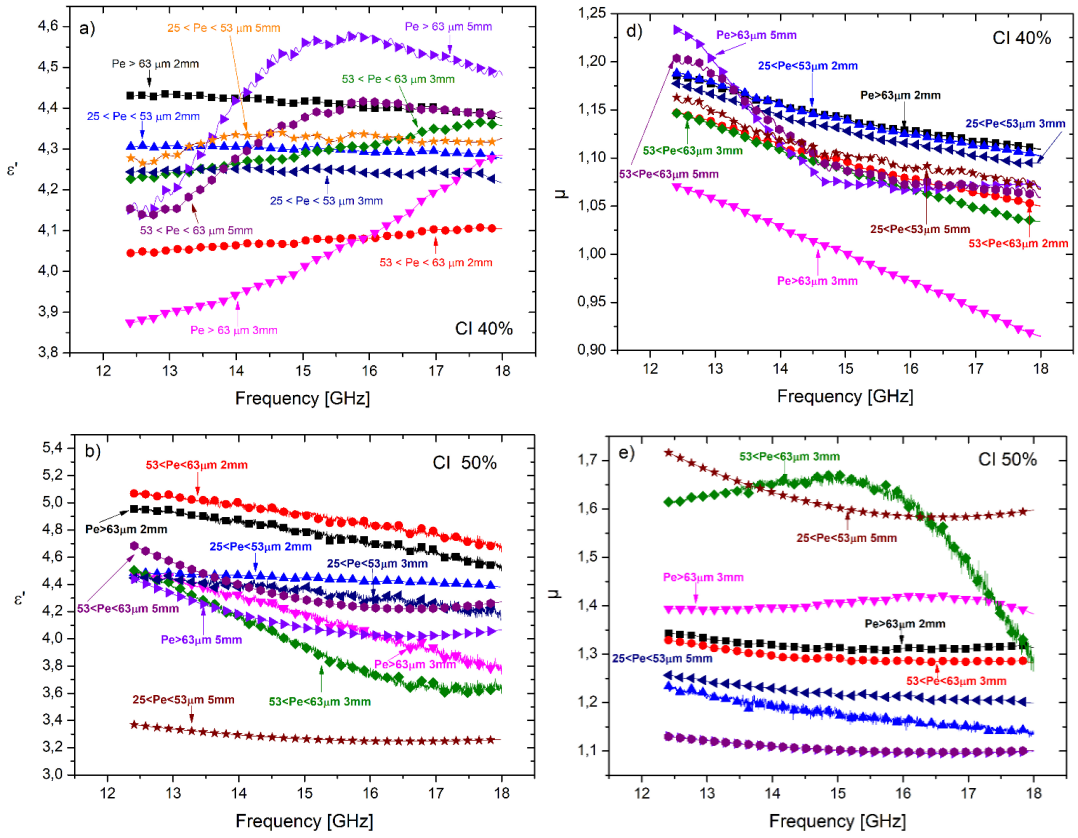
and 60%, respectively. Fig. 4d – 4f are the real permeability (**μ'**) of composites with CI concentrations of 40%, 50% and 60%, respectively. It is possible to observe the effects of CI concentration and particle size on permittivity and permeability analysis, where some measurements also presented a frequency dependency over the analyzed range. It is important to notice that CI concentration has a strong dependence on permittivity, as well as on the boundary conditions between additive and interface. Increasing the CI concentration affects the real permittivity due the interaction between CI particles. The internal structure arrangement and the volume fraction of CI in the silicone is directly related to the interaction of the electromagnet wave into the composite, having influence on the storage charging in the presence of an applied electric field [11]. It is possible to observe that at a 40% CI concentration causes a variation on permittivity from 3.85 to 4.6, Fig. 4a. For concentration of 60% this variation on permittivity is about 4.75 and 5.35, Fig. 4c. However, for 50% CI concentration, Fig. 4b, the permittivity ranges from 3.2 to 5.1, which is the biggest permittivity variation observed. The highest values of **ε'** are associated with the highest proportion of CI inside silicone matrix, which increases the number of CI/silicone interfaces. Permeability of samples with 40% and 60% CI concentration have a slightly decreasing as a function of frequency for all particle sizes and thicknesses. However, carbonyl iron concentration of 50% has no frequency dependence like the others. Similar behavior was observed by Zhu et al over the frequency range of 2 - 18 GHz [12].



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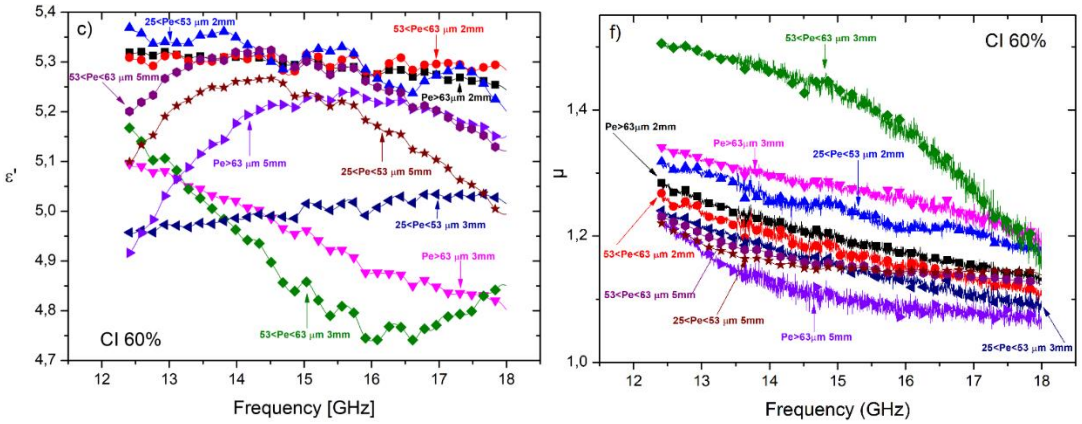

Fig.4 Experimental values of the real part of the permittivity (a) (b) and (c) and permeability (d), (e) and f as a function of frequency.

D. Reflectivity Measures

According to equations (1) and (2), the Reflection Loss (RL) of a RAM have influence of sample thickness and may have a frequency dependence inherited from complex permittivity and permeability [13].

$$\boldsymbol{Z_r} = \sqrt{\frac{\boldsymbol{\mu_r}}{\boldsymbol{\varepsilon_r}}} \tan \boldsymbol{h}\left(j\frac{2\pi}{\boldsymbol{\lambda}}\boldsymbol{t}\sqrt{\boldsymbol{\mu_r} \times \boldsymbol{\varepsilon_r}}\right) \qquad (1)$$

$$RL(dB) = 20 \log \left|\frac{\boldsymbol{Z_r} - 1}{\boldsymbol{Z_r} + 1}\right| \qquad (2)$$

Here, $\boldsymbol{Z_r}$ is the relative input impedance of material, **t** is the sample thickness, $\boldsymbol{\mu_r}$ is the material relative magnetic permeability, $\boldsymbol{\varepsilon_r}$ is the material relative dielectric permittivity and $\boldsymbol{\lambda}$ is the wavelength of the incident wave in the free space.

Fig. 5a – 5d present the experimental reflectivity of evaluated samples. From Fig. 5a to Fig. 5c, reflectivity is highlighted for samples with 3 and 5 mm thickness, where all three particulate variations and CI concentrations are plotted. Since samples with 2 mm thickness presented the best RL results, all samples with this thickness is plotted in Fig. 5d for comparison.

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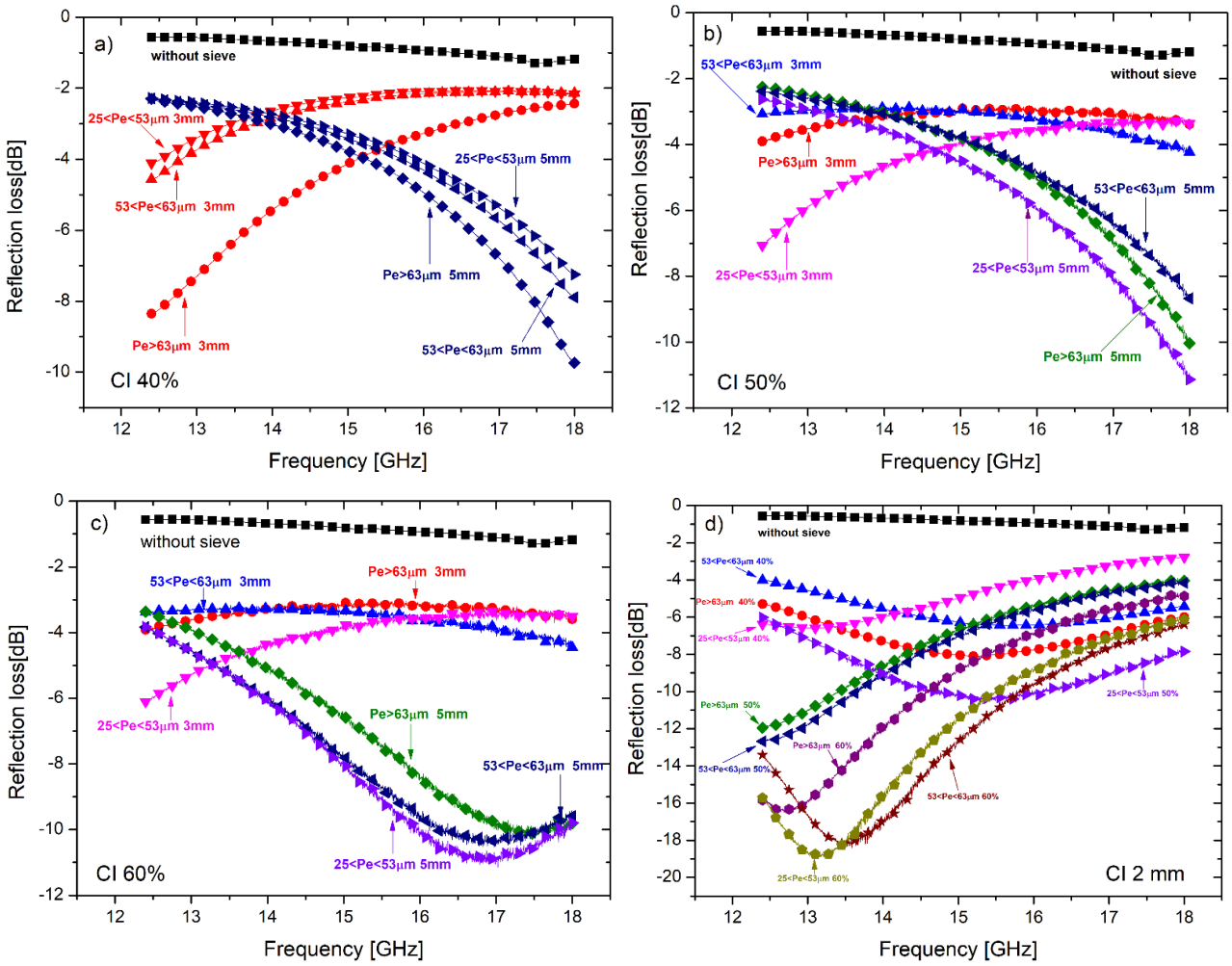

Fig. 5. (a), (b) and (c) reflectivity for samples of thicknesses of 3 mm and 5 mm with variation of the particulate, and (d) reflectivity for samples with variation of the particulate at 2 mm of thicknesses.

In Fig. 5a, which have RL plots of 40% concentration, it is possible to verify that the highest reflectivity value was obtained for sample with 5 mm thickness and particulate size bigger than 63 μm. The attenuation in this sample is -9.5 dB at 18 GHz, i.e., 90% of the incident wave was attenuated [14]. It is also observed that RAM with the same particle size, but with thickness of 3 mm, tends to act as a microwave absorber in the X-band frequency range (8.2 - 12.4 GHz). For these samples, the maximum reflectivity loss was - 8.5 dB at 12.4 GHz. Thus, RAM with 40% CI concentration and 3 mm and 5 mm thicknesses tends to attenuate the incident wave in the X-band and K-band (18 – 26.4 GHz), respectively.

This behavior is not observed for samples with no sieving. In other words, granulometric separation made possible the choice of the frequency band to work. This effect can be attributed exclusively to particle size, since no structural changes that could justify such behavior was noticed, as it could be observed in XRD graphs in Fig. 3.

It is possible to observe that 5 mm thickness samples trends to attenuate K-band frequencies when samples present concentrations of 40% and 50%, Fig. 5b. However, a minimum RL value is observed

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at ~17 GHz for CI concentration of 60%, Fig. 5c.

Samples with thickness of 2 mm have greater attenuations, which occurs mainly with CI concentration of 60%. The best RL attenuation results are close to 13 GHz, Fig. 5d.

In summary, these results highlight different behaviors as a function of sample thickness and concentration. For the samples with 3 mm thickness, reflectivity presents a trend to have a better performance in the frequency band that precedes the K_u-band, i.e., X-band. For samples with 5 mm thickness there is a trend to great reflectivity attenuation in the K-band. These results show that the thickness and the concentration have a strong influence on frequency range division. These results are very important since controlling particulate size and thickness enable controlling of frequency ranges. For the same CI concentration (60%) and two different thickness (2 and 5 mm) it is possible to shift the RL from 13 GHz to 17 GHz. The reflectivity curves show the predominant influence of the particle sizes resulting in more efficient absorbers with attenuation values > 90%.

IV. CONCLUSIONS

We demonstrated the possibility to attenuate electromagnetic wave over a frequency range by controlling sample thickness, particle size and CI concentration of RAM. Values obtained for reflectivity (RL) showed that it is possible to have an efficient RAM (RL < -10 dB) in the frequency range of 12-18 GHz using a thin sample (2 mm) by simply controlling particulate size and concentration. Based on these results it is possible to design and manufacture different electromagnetic absorbers with the same material by simply controlling parameters of concentration particle size. Carbonyl iron separated in different particulates have great potential to be used as absorber materials in the K_u-band.

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