Effects of Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ Nanoparticles Concentration in a Silicone Matrix on Complex Permeability and Permittivity in the 1-10 GHz Range

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Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ (0 ≤ x ≤ 0.65) nanoferrite particles were used as filler in a silicone polymer matrix in order to obtain a composite material with the purpose of absorbing electromagnetic radiation. The composite was characterized by X-ray diffraction, transmission electron microscopy and force modulation microscopy (FMM) and the electromagnetic properties by means of network and impedance analyzers. It was observed that the composite with higher concentrations of Zn$_x$ (x = 0.5 and 0.65) presented the lowest dispersion, due to the size of the zinc particles. This paper provides a framework for the practical application of absorbent composite materials of MnZn nanoferrite particles combined with silicone rubber to be used in 1-10 GHz frequency bands, with reflectivity values of -24 dB in the 6 GHz frequency range.

Keywords: electromagnetic absorption, nanoferrite particles, silicone rubber, composite materials.

1. Introduction

Spinel-type ferrites are ferromagnetic materials used in the field of magnetic materials due to their excellent electromagnetic properties. They can be applied in transformers$^{1-3}$, transmission devices$^{4-6}$, biomedical applications$^{7-11}$, for electromagnetic radiation absorption$^{12-15}$, among others. These materials can be synthesized by different preparation methods, such as: conventional oxide blending$^{16-18}$, coprecipitation$^{19-21}$, sol-gel$^{22-24}$ and combustion$^{25-27}$. However, low purity, heterogeneity, as well as aggregation are factors to be observed in the choice of the technique, especially in applications for electromagnetic absorption.

The combustion reaction technique has been standing out among the synthesis methods, since it is a safe, fast and relatively low-cost method, besides presenting adequate reproducibility for production of ceramic powders. Moreover, it is also able to produce objects with the desired structure and composition for a wide range of applications and still offers the possibility of large-scale production$^{28,29}$. Electromagnetic radiation absorbing materials have attracted the attention of industry due to the growing demands of government legislation to control spurious electromagnetic radiation levels emitted by electronic equipment, that are associated with industrial standards for electromagnetic compatibility and interference. That study area has been reported by several authors$^{30-35}$. The materials Electromagnetic radiation absorbing are composed of compounds that provide energy losses from electromagnetic radiation. In certain frequency ranges, these materials attenuate the incident electromagnetic radiation wave and dissipate the absorbed energy in the form of heat$^{36}$. In order to supply the growing demand for electromagnetic absorbing films and coatings, composite materials consisting of ferrite particles embedded in polymeric matrices have been the subject of numerous researches$^{37-39}$. Among these matrices we can highlight the silicone, which has very attractive original features in terms of optical and mechanical properties as well as good retention and flexibility, that are ideal for these applications$^{40}$. The interest in polymer nanocomposites has rapidly been increasing in industry and in academia, due to their effect of altering many properties, such as absorbance, ion exchange capacity, thermal and solvent resistance, gas barrier properties, flame retardance, mechanical properties (hardness and stiffness) or simply to reduce the cost price$^{41}$.
Based on this context, this work aims to analyze the microstructural and electromagnetic properties of Mn-Zn ferrites incorporated in a silicone matrix for application as electromagnetic radiation absorbing materials. The absorption, permeability and permittivity properties were evaluated in the frequency range of 1-10 GHz, with ferrite nanoparticles of different granulometric bands dispersed in silicone rubber.

2. Experimental

2.1. Materials and methods

In order to carry out the synthesis of Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrites, with $x = 0, 0.35, 0.5$ and $0.65$, high purity reagents (nitrates) were used, with glycerin being the fuel. The initial composition of the solution was based on the total valence of oxidizing and reducing reagents, using concepts from the propellants and explosives chemistry, with the purpose of establishing the stoichiometry of the interest phase. The redox mixture of metal nitrates and fuel was subjected to direct heating in a reactor with a production capacity of 200 g batches of product, illustrated in the Figure 1. The Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrite particles were dispersed in a silicone matrix in a mass ratio of 2:1.

2.2. Characterization

The samples were characterized by X-ray diffraction (XRD) on a Shimadzu XRD-6000 model diffractometer using a monochromatic radiation source (Cu-Kα of $\lambda = 1.5418$). Regarding the characterization of particle size distribution, a Horiba SZ-100 series zeta nanoparticle analyzer was used. The morphological aspects of the ferrite samples were analyzed using a Shimadzu Superscan SS-500 scanning electron microscope (SEM) and a Philips model EM420 transmission electron microscope (TEM), with a voltage of 120 kV. Each sample was analyzed by force modulation microscopy (FMM), with an atomic force microscope Agilent 5420 SPM. The electromagnetic evaluation was performed on an Agilent 4294A impedance analyzer by the SOL (Short-Open-Load) method, with the 85054D Agilent calibration kit, and on an Agilent Network Analyzer model N5231A, with the 85052D Agilent calibration kit, also by the SOL (Short-Open-Load) method, that uses short, open and load patterns.

3. Results and Discussion

3.1. X-ray diffraction (XRD)

Figure 2 illustrates the X-ray diffractograms of the pure Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ($x = 0, 0.35, 0.5$ and $0.65$) ferrites obtained by large scale combustion reaction and when they are incorporated into silicone matrices. It can be seen from Figure 2 the formation of cubic inverse spinel phase of single MnFe$_2$O$_4$ ferrite and mixed Mn-Zn, which were identified by means of crystallographic records JCPDF 74-2403 and JCPDF 89-7556, respectively. All samples of pure ferrite showed peaks with high intensity and high basal width for all reflections, demonstrating their crystallinity and nanostructural characteristics. This indicates the efficiency of the combustion technique in making it possible to obtain these single-phase systems, with characteristics of nanoscale particles, in addition to the possibility of obtaining on a large scale.

Regarding the ferrites incorporated in the silicone matrix, a lower peak intensity was observed in relation to the ferrite of the single MnFe$_2$O$_4$ ferrite and mixed Mn-Zn. This behavior is probably associated with the presence of amorphous silicone rubber. However, its presence did not interfere with the formation of spinel structure of ferrites, as confirmed by the presence of characteristic peaks related to the cubic spinel phase.

Figure 3 illustrates Rietveld’s refinement of Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ($x = 0, 0.35, 0.5$ and $0.65$). The experimental points (blue points) were adjusted to intensity (red line) with the TOPAS program whose standard record is (ICDD 89-7554 for Mn-Zn ferrite and ICDD 74-2403 for MnFe$_2$O$_4$ ferrite).

Through the refinement data, it was possible to observe that the amounts of phases, for all samples obtained by large-scale combustion reaction, were 100%, so it can be concluded that presented a single phase, proving the results reported in the XRD analysis. The refined structural parameters and the factors related to progress, the quality of the refinement and the agreement between the observed and calculated profile, Rwp and GOF, are described in Table 1.

3.2. Particle size distribution

Figure 4 illustrates the diameter distribution of equivalent particles as a function of the cumulative volume of Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrites, with compositions $x = 0, 0.35, 0.5$ and $0.65$, obtained by large scale combustion reaction in the range of 10 to 10.000 nm. The SZ-100 uses the light dynamic dispersion technique to determine particle size. Dynamic light scattering is the measurement of fluctuations.
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Figure 2. X-ray diffractograms of Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrite samples with compositions $x = 0, 0.35, 0.5$ and $0.65$ (a) in powder and (b) inserted into the silicone.

Figure 3. X-ray diffratograms with Rietveld refinement with compositions (a) $x = 0$, (b) $0.35$, (c) $0.5$ and (d) $0.65$. 
in the intensity of light dispersed over time. The reading occurs through the Brownian movement of the particles in an appropriate dispersant. Considering the median diameter (50%), the sample \( \text{Mn}_{0.5} \text{Zn}_{0.5} \text{Fe}_2 \text{O}_4 \) presented the largest particle size, with a value of 54.5 nm, which coincided with the narrower distribution curve, indicating the reduction of the average separation distance between the particles. The \( \text{MnFe}_2 \text{O}_4 \) sample had the smallest median diameter (50%), with a value of 39.78 nm, and a wider distribution curve, which can be attributed to the fact that it is the only sample that does not have zinc in its composition.

### Table 1. Rietveld refinement data of \( \text{Mn}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4 \) samples (\( x = 0, 0.35, 0.5 \) and 0.65).

| Samples                  | \( R_{wp} \) | \( R_{exp} \) | GOF | Phases (%) | Parameters of Network (Angstron) |
|--------------------------|--------------|---------------|-----|------------|----------------------------------|
| \( \text{MnFe}_2 \text{O}_4 \) | 1.67         | 0.94          | 11.76 | \( \text{MnFe}_2 \text{O}_4 \)-100% | \( a=b=c=8.498 \) |
| \( \text{Mn}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4 \) | 1.61         | 1.21          | 1.33 | (\( \text{ZnMnFe} \)(\( \text{MnFe}_2 \text{O}_4 \))) 100% | \( a=b=c=8.497 \) |
| \( \text{Mn}_{0.35}\text{Zn}_{0.65}\text{Fe}_2\text{O}_4 \) | 1.64         | 0.94          | 1.74 | (\( \text{ZnMnFe} \)(\( \text{MnFe}_2 \text{O}_4 \))) 100% | \( a=b=c=8.462 \) |
| \( \text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4 \) | 1.65         | 1.18          | 1.39 | (\( \text{ZnMnFe} \)(\( \text{MnFe}_2 \text{O}_4 \))) 100% | \( a=b=c=8.455 \) |

**Figure 4.** Particle size distribution of (a) \( \text{MnFe}_2 \text{O}_4 \), (b) \( \text{Mn}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4 \), (c) \( \text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4 \) and (d) \( \text{Mn}_{0.35}\text{Zn}_{0.65}\text{Fe}_2\text{O}_4 \).

**Figure 5.** Transmission Electron Microscopy (TEM) micrographs observed in a bright field and their respective refractive patterns of \( \text{Mn}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4 \) ferrites, with compositions \( x = 0, 0.35, 0.5 \) and 0.65, obtained by large scale combustion reaction. From the micrographs obtained by TEM, it was possible to observe, in general, the formation of agglomerates consisting of hexagonal and spherical shaped nanoparticles with varying sizes. It is possible to note that the sample for \( x = 0 \) had an average particle size of 75 ± 0.9 nm, for \( x = 0.35 \) it was 71.5 ± 0.4 nm, for \( x = 0.5 \) it was 78 ± 1.2 nm and for sample \( x = 0.65 \) it was 63 ± 0.5 nm. Overall,

### 3.3. Transmission electron microscopy (TEM)

Figure 5 illustrates the micrographs observed by Transmission Electron Microscopy (TEM) in a bright field and their respective refractive patterns of \( \text{Mn}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4 \) ferrites, with compositions \( x = 0, 0.35, 0.5 \) and 0.65, obtained by large scale combustion reaction. From the micrographs obtained by TEM, it was possible to observe, in general, the formation of agglomerates consisting of hexagonal and spherical shaped nanoparticles with varying sizes. It is possible to note that the sample for \( x = 0 \) had an average particle size of 75 ± 0.9 nm, for \( x = 0.35 \) it was 71.5 ± 0.4 nm, for \( x = 0.5 \) it was 78 ± 1.2 nm and for sample \( x = 0.65 \) it was 63 ± 0.5 nm. Overall,
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all the systems presented particle sizes less than 100 nm, indicating that they can be considered as materials with nanostructural characteristics. It was noted that besides the samples with $x = 0$ and $x = 0.5$ present a larger particle size, they also present an aspect of greater agglomeration, with the presence of grain outlines of the pre-sintered particles, which are highlighted in Figures 5a and 5c.

In relation to the increase in the zinc content in the composition, a non-linear behavior was observed, since as the zinc content of the sample Mn$_{0.65}$Zn$_{0.35}$Fe$_2$O$_4$ is added to Mn$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ there was an increase of approximately 8% followed by a 20% decrease from the sample Mn$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ to Mn$_{0.35}$Zn$_{0.65}$Fe$_2$O$_4$.

In the case of polycrystalline ferrites, the permittivity is a response attributed to the spaces between the loads at the grain boundaries, while the permeability is related to the magnetizing mechanisms: the spin rotations and the size of the magnetic domains, that is, the particle size$^{45}$.

3.4. Scanning electron microscopy (SEM)

Figure 6a-d illustrates the sample morphology of Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrites, with compositions $x = 0, 0.35, 0.5$ and 0.65, embedded in a silicone matrix. From the micrographs, it was possible to observe, in general, that the samples presented a uniform distribution of the ferrite load within the silicone matrix, however with the formation of irregularly dispersed agglomerates throughout the matrix. There are no significant changes in the composite morphology with the change in compositions.

3.5. Force modulation microscopy (FMM)

Figure 7a-d shows the FMM oscillation phases of Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrites, with compositions $x = 0, 0.35, 0.5$ and $x = 0.65$, embedded in a silicone matrix. It is noted through the FMM that the increase in the Zn content provided less dispersion of the particles, which tended to agglomerate inside the composite, since Zn presents smaller particles in relation to Mn, and then promote a greater state of agglomeration mainly due to the mutual attraction of the nanoparticles$^{46}$. However, one should point out that the oscillation phase of FMM is only a relative indicator of homogeneity, since it shows only the contrast between dispersion and particle agglomeration for each case, not being used as a comparison to calculate the average stiffness of each sample, for which the oscillation amplitude value is required.

3.6. Magnetic Permeability and magnetic permittivity

Figure 8 illustrates the measurements of complex real magnetic permeability ($\mu'$) and imaginary magnetic permeability ($\mu''$) versus frequency for Mn$_{1-x}$Zn$_x$Fe$_2$O$_4$ ferrites, with
Figure 6. Morphological characteristics obtained by SEM for the samples of (a) MnFe$_2$O$_4$, (b) Mn$_{0.65}$Zn$_{0.35}$Fe$_2$O$_4$, (c) Mn$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ and (d) Mn$_{0.35}$Zn$_{0.65}$Fe$_2$O$_4$ (2.000 increase).

Figure 7. Phase angle of the FMM for the samples of (a) MnFe$_2$O$_4$, (b) Mn$_{0.65}$Zn$_{0.35}$Fe$_2$O$_4$, (c) Mn$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ and (d) Mn$_{0.35}$Zn$_{0.65}$Fe$_2$O$_4$. 

compositions $x = 0$, $0.35$, $0.5$ and $0.65$, embedded in a silicone matrix. The complex permeability spectra for all samples are plotted as a function of frequency. Both the real ($\mu'$) and the imaginary ($\mu''$) parts of the permeability were apparently constant and with close values ($\mu' = 3.4$) for the blankets with $x = 0.5$ and 0.0, respectively, in the frequency range 1 to 10 GHz. For blankets with $x = 0.65$ and 0.35, which has a lower concentration of Zn, the values were approximately $\mu' = 2.7$ and 2.9.

These responses of permeability as a function of increased frequency are attributed to the combinatorial magnetic relaxations of domain wall movements and spin rotations in the nanoferrites incorporated in the silicone. This finding is in accordance with several data reported in the literature$^{47-49}$.

Figure 9 illustrates that the permittivity versus Frequency for blankets with $\text{Mn}_1\text{Zn}_x\text{Fe}_{2}\text{O}_4$ ferrites with compositions $x = 0$, $0.35$, $0.5$ and $0.65$.

This behavior is compatible with its real part (\(\varepsilon'\)), which remains very low across the measurement range for all investigated samples. The variation in the Zn concentration did not represent major distortions in the electrical values, which were around $\varepsilon' = 1.2$ for all samples but with a decay at high frequencies. The reduction of the permittivity value for materials at high frequencies can be justified mainly because of the atomic and electronic polarization within the ferrite nanoparticles. The electrical polarization mechanism is considered to be the cause of the jump of electrons between Fe$^{2+}$ and Fe$^{3+}$ at octahedral sites. According to Nasir et al.$^{50}$ it is known that polarization can be maintained due to the longer relaxation time at low frequencies, and not be maintained with increasing frequency, since the relaxation time decreases in greater frequency. This behavior has been reported by several authors$^{51,52}$.

According to transmission line theory, the reflection loss (RL) can be calculated from the material impedance value, $Z_m$, and of the characteristic air impedance, $Z_0$. The material impedance is calculated based on the permittivity and complex relative permeability, according to Equation 1$^{53}$:

$$Z_m = Z_0 \sqrt{\mu_r \varepsilon_r \tanh \left[ \left( \frac{\omega \cdot d}{c} \right) \sqrt{\mu_r \varepsilon_r} \right]}.$$  

Thus, the reflectivity is calculated in decibels using Equation 2:

$$RL = 20 \log_{10} \left| \frac{Z_m - Z_0}{Z_m + Z_0} \right|.$$  

Figure 10 illustrates the loss of reflection (dB) for blankets of $\text{Mn}_1\text{Zn}_x\text{Fe}_{2}\text{O}_4$ composition, with $x = 0$, $0.35$, $0.5$ and $0.65$, with a thickness of 20 mm. For obtaining an electromagnetic radiation absorbing material, the real part of the permeability must be maximized and the real part of the permittivity minimized, so that these parameters are close to each other to achieve harmonization conditions. From Figure 10 it can be seen that the blanket with equal concentrations of Zn and Mn presented the minimum reflection loss, which was -24 dB, at a frequency around 6 GHz. With $x = 0.35$ in mass of ferrite in the absorbing material, the reflectivity is -10 dB at the frequency of 7 GHz. It was observed that the samples X= 0 and 0.5 showed similar reflectivity values in the 6 GHz range, this can be justified by the fact that the samples present the larger particle sizes.
The technique of phase cancellation of the electromagnetic wave seeks to adjust the thickness of the absorber, in order to cause the phase cancellation of the incident wave with those of the wave emerging from the absorber, at the interface of the absorber material with the air. This technique is widely used in the production of the resonant absorbers. Resonant absorbers represent the majority of the market. These absorbers perform relatively well, but are effective only in very narrow frequency ranges.

The high absorption of this sample is due to its permeability values of the real and imaginary parts, which were the highest in relation to the other compositions.

The data obtained show promising data to be used in the frequency range between 6 to 8 GHz, the composites obtained show promising data to be used in the camouflage of structures, in order not to occur detection by radars, in narrow band.

4. Conclusions

The synthesis by combustion reaction was a favorable technique for single-phase obtaining of Mn-Zn Fe$_2$O$_4$ (x = 0, 0.35, 0.5 and 0.65) ferrites on a large scale and with high crystallinity, without significant changes when incorporated into the silicone. The average particle sizes were around 45 nm. The blanket with the highest Zn concentrations (x = 0.5 and 0.65) presented the lowest dispersion, due to the smaller zinc particles, which ended up promoting a high state of agglomeration. Regarding the absorptive character of the blankets, the composition x = 0.5 presented the reflectivity value of -24 dB in the 6 GHz frequency range. From the results obtained, it is important to highlight how promising is the incorporation of nanoparticles into polymeric blankets, being possible to achieve optimal absorption values which allows its application, for example, in the camouflage of structures, in order not to occur the detection by radars, in narrow band.

From these results it is possible to manage the obtaining of hybrid absorbers, by combining magnetic additives with different concentrations in mass and thickness, in obtaining broadband absorbers with a reduced thickness, in the range of 8.0 – 10.0 GHz, with superior attenuation of ~ 10 dB, that is, 90% absorption of incident radiation.

5. References

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