Study the Microwave absorption characteristics of U-type barium hexaferrites prepared using chemical method

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Abstract

U-type Barium hexaferrite with stoichiometric composition Ba₄Co₀.3Zn₀.6Cu₁.1Fe₃₆O₆₀ has been prepared using citrate precursor method at low temperatures (1000, 1050, and 1100°C). The XRD patterns of the prepared powders at temperatures (1000 and 1050°C) show the presence of spinel, M and Y-type phases, while U-type ferrite phase(with crystallite size 40 – 90 nm) obtained at 1100 °C. Radar absorbing nanocomposite samples were prepared by mixing, molding and curing mixtures of Ba₄Co₀.3Zn₀.6Cu₁.1Fe₃₆O₆₀ powders (prepared at 1100°C) with epoxy resin (ER),(80:20% weights). The microwave attenuation measurements were based on the Transmission/Reflection method using rectangular waveguides as the confining medium for the samples at 8.0–12 GHz frequencies.

The complex dielectric constant, complex permeability (εᵣ', εᵣ, μᵣ' and μᵣ") and losses(tan δₑ and tan δµ ) of hexaferrite-epoxy resin composites were measured in the range of 8 -12 GHz. Measured results show that the parameters εᵣ', εᵣ, μᵣ' and μᵣ" exhibited very small change as the measuring frequency increasing at (9.2–11.2 GHz). The results also indicate that the content of copper and zinc ions closely affects the microwave properties of composite samples, resulting in microwave absorption greater than 90.0 % (reflectivity ≤ -10 dB) at 9.2-11.2 GHz with 2.0 mm thickness. Attenuation matching results show that the electromagnetic wave propagation in ferrite is slower than that in free space by six times, this result may help us to design microwave absorber at x-band frequencies with 2 mm thickness.

Keywords: RAM, U-type ferrite, XRD, x-band, microwave properties, hexaferrites, and reflection loss.

1. Introduction

The development of microwave absorber continues to attract much attention because of the increasing environmental pollution from wireless telecommunication systems, high-frequency circuit devices and the essential part of a stealth defense system for all military platforms because it can transform undesired electromagnetic waves into heat [1]. Also, microwave absorbers have been widely used to prevent or minimize electromagnetic
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reflections from large structures such as aircraft, ships and tanks and also to cover the walls of anechoic chambers [2]. An effective electromagnetic wave absorber must fulfill the following requirements [3]:

1. maximum absorption of electromagnetic waves with minimum reflection,
2. dissipation of incident wave energy changes into heat.

Since microwave permeabilities of known magnetic materials do not exceed several units above GHz frequency, other approaches to broaden bandwidth of radar absorbers attract great attention. Conventional methods of creating broadband wave absorbers employ multilayer absorbing structures with impedance-graded composites or the parameters (permittivity and permeability) with frequency dispersion [4 and 5].

As magnetic materials, barium hexaferrites are not generally replaced by any other magnetic material, because they are relatively inexpensive, stable, and have a wide range of technological applications. Barium hexaferrites have been classified, according to their structures, into five main classes: BaFe$_{12}$O$_{19}$, BaMe$_2$Fe$_{16}$O$_{27}$ (W-type), Ba$_2$Me$_2$Fe$_{28}$O$_{46}$ (X-type), Ba$_2$Me$_2$Fe$_{12}$O$_{22}$ (Y-type), Ba$_3$Me$_2$Fe$_{24}$O$_{41}$ (Z-type), and Ba$_4$Me$_2$Fe$_{36}$O$_{60}$ (U-type), where Me represent a divalent ion from the first transition series [6].

U-type barium hexaferrite is a promising material for applications such as RAM at GHz frequencies, which requires high permeability, great resistivity, and good chemical and thermal stability. These ferrites absorb microwaves due to various interactive loss mechanisms related to the magnetization and electric polarization of the material. However, only few studies have been reported on the microwave performance or magnetic properties of U-type hexaferrite since it was developed by Jonker et al. [7].

Various techniques have been developed to prepare nanosized hexaferrite for these Purposes. They include the glass crystallization method [8], the wet method [9], the liquid mix technique [10], colloidal methods [11], and so on. In conventional ceramic methods, a high sintering temperature is necessary to obtain this U-type hexaferrite. By using chemical methods, this temperature can be reduced. In the present work, the citrate precursor process in air was used to obtain Ba$_4$Co$_{0.3}$Zn$_{0.6}$Cu$_{1.1}$Fe$_{36}$O$_{60}$ hexaferrite; this process permits the formation of the U-type phase at lower sintering temperatures. The introduction of Cu$^{2+}$ and Zn$^{2+}$ ions in the structure of Ba$_4$Co$_{0.3}$Fe$_{36}$O$_{60}$ can also reduce the sintering temperature, because it can act as a flux due to its low melting point (1050 ºC). X-ray diffraction (XRD), with Cu K$_\alpha$ radiation was used to characterize the synthesized material.

The relative complex permeability and permittivity parameters of ferrite-polymer composites may depend on the type, composition and concentration of ferrite besides the temperature and frequency of operation [12]. The microwave measurements were based on the transmission/reflection method (T/R) using rectangular waveguides as the confining medium for the samples. The materials were analyzed in the frequency range 8.0–12.4 GHz (X-band).
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2- Matching conditions and Loss Mechanisms

For a microwave-absorbing layer terminated by a short circuit, the normalized input impedance related to the impedance in free space, \( Z_{\text{in}} \), and reflection loss (R.L.) related to the normal incident plane wave are given by theory of the absorbing wall,[13]

\[
Z_{\text{in}} = \left( \frac{\mu_r^*}{\varepsilon_r^*} \right)^{1/2} \tanh \left[ j \left( \frac{2\pi fd}{c} \right) \left( \frac{\mu_r^*}{\varepsilon_r^*} \right)^{1/2} \right] \quad \text{(1)}
\]

\[
\text{R.L. (dB)} = 20 \log_{10} \left[ \frac{(Z_{\text{in}} - 1)/(Z_{\text{in}} + 1)}{10} \right] \quad \text{(2)}
\]

, where \( f \) is the frequency, \( c \) is the speed of light and \( d \) is the thickness of specimen. \( \varepsilon_r^* \) is the relative complex permittivity = \( \varepsilon'_r - j\varepsilon''_r \), where \( \varepsilon'_r \) and \( \varepsilon''_r \) is the real and imaginary parts of \( \varepsilon_r^* \), respectively. \( \varepsilon''_r \), associated with electric field loss, results mostly from electric-dipole polarization at microwave frequencies. \( \mu_r^* \) is the relative complex permeability = \( \mu'_r - j\mu''_r \), where \( \mu'_r \) and \( \mu''_r \) is the real and imaginary parts of \( \mu_r^* \), respectively. \( \mu''_r \), associated with magnetic field loss, results from magnetic-dipole magnetization. Further, the loss tangent of the dielectric/magnetic can be expressed as \( \tan \delta_e = \frac{\varepsilon''_r}{\varepsilon'_r} \) and \( \tan \delta_\mu = \frac{\mu''_r}{\mu'_r} \), respectively. To achieve a reflection coefficient of zero, the impedance matching condition is given by \( Z_{\text{in}} = 1 \) to represent the perfect absorbing properties would therefore have \( \mu_r \) equal to \( \varepsilon_r \) as large as possible to achieve absorption in thinnest layer. [5]

The loss mechanisms are divided into dielectric loss and magnetic loss in microwave frequency. These loss tangents can be summarized as follows:

\[
\tan \delta = \tan \delta_e + \tan \delta_\mu \quad \text{.................................................. (3)}
\]

There are three important matching conditions (\( Z_1, Z_2 \),and \( Z_3 \)) to evaluate the microwave absorption of ferrite: epoxy composite samples with (80:20) as weight ratio and 2mm thickness;

\[
Z_1 = \frac{\varepsilon'_r}{\mu'_r} \quad \text{,} \quad Z_2 = \frac{\tan \delta_e}{\tan \delta_\mu} \quad \text{,} \quad \text{and} \quad Z_3 = \frac{1}{n} = \frac{d}{\lambda} \quad \text{......... (4)}
\]

, where \( n \) is represents the refractive index and equals to \( n = (\varepsilon_r^*\mu_r^*)^{1/2} \), \( d \) is sample thickness(mm), and \( \lambda \) is free space wavelength.

3. Experimental

Nanosized \((\text{Co-Cu-Zn})_2\) U-type powders were synthesized by the citrate precursor method, the following reagents: \( \text{Fe(NO}_3\text{)}_3\cdot9\text{H}_2\text{O} \) (98%) , \( \text{Ba(NO}_3\text{)}_2 \) (99%), monohydrate citric acid (99.5%) , \( \text{Co(NO}_3\text{)}_2\cdot6\text{H}_2\text{O} \) (98%), \( \text{Zn(NO}_3\text{)}_2\cdot6\text{H}_2\text{O} \) (98.5%), and \( \text{Cu(NO}_3\text{)}_2\cdot3\text{H}_2\text{O} \) (98.5%) of Merck make, used in stoichiometric molar ratios to achieve the \( \text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60} \) hexaferrite.

The preparation of the solutions was carried out by weighting the reagents and placing them into adequate vessels. Distilled water was added to each one, until total dissolution of the solids and then mixed. The resulting mixture was heated under reflux up to 80 °C to complete the reaction, in order to allow the addition of \( \text{NH}_4\text{OH} \), drop by drop into the solution to make it neutral or slightly alkaline (pH: 7- 8 ), for subsequent precipitation of the organo-metallic complex. The key metal cations reacted with citric acid, under controlled pH conditions, to give the respective metal citrate, making up a homogeneous joint metallic citrate precursor complex.
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Distilled water was then added under vigorous stirring, into the reacting mixture to promote the precipitation of a complex citrate gel of barium, iron, zinc, copper and cobalt. After that, the remaining aqueous solution was removed by drying it at 80 °C. The remaining desired solid phase was a highly viscous residue. The gel was submitted to a calcinations performed at different temperatures 1000, 1050, and 1100 °C in air (with a residence time at the calcinations temperature of 4 hours). Then, it was cooled down to room temperature. The calcined products were submitted to X-ray diffraction in order to assure the formation of the magnetic crystalline phase of the U-type barium hexaferrites (Ba₄Co₀.₃Zn₀.₆Cu₁.₁Fe₃₆O₆₀), which only occurs at 1100 °C. The composite specimens for measuring microwave absorber properties were prepared by mixing, molding and curing mixtures of barium hexaferrite powders with epoxy resin. This resulted in a barium hexaferrite: epoxy resin concentration of 80:20% by weight.

The microwave measurements were based on the transmission/reflection method (T/R) using a rectangular waveguide as the confining medium for the samples. The measurements were made with a HP 8510 network analyzer system. The material was analyzed in the frequency ranges of 8-12 GHz. From the data obtained, a prediction of the microwave reflectivity levels for the sheet absorbers (ferrite: epoxy resin=80:20% weight) dimensions (23x11 mm²) with other thickness values in the range (1.25-4.0 mm) was made to estimate the variations of reflection loss (dB) vs. frequency (GHz).

4- Results and discussion
4-1- X-ray diffraction patterns

The nanostructure of Ba₄Co₀.₃Zn₀.₆Cu₁.₁Fe₃₆O₆₀ (U-type) were performed by means of X-ray diffractometer (Philips X’pert MPP), (CuKα radiation, λ = 0.154056 nm) under a voltage of 40 kV and a current of 100 mA. The XRD patterns at room temperature for the investigated composition of U-type barium hexaferrite samples synthesized by the citrate precursor method are shown in figure (1). The U-type phase powder can not be produced so easily due to the complexity of its structure, which imposes progressive transformation through intermediate ferrites before achieving the final required structure. The experimental results of XRD pattern indicate that the intermediates with different crystalline phases are a function of the calcining temperatures. Results also showed these transformations as being the following:

a) at 1000°C, Ba₂Co₂Fe₁₂O₂₂(Y-type) and spinel (BaFe₂O₄) phases were found
b) at 1050 °C, the product consisted of Y and M phases; c) for the samples calcinized at 1100 °C for 4 hours, U-type phase was clearly the majority phase.

The average crystallite size (D=40 to 90 nm) for prepared ferrite Ba₄Co₀.₃Zn₀.₆Cu₁.₁Fe₃₆O₆₀ powders was calculated employing the Scherrer formula:

\[ D = \frac{0.94 \lambda}{\beta \cdot \cos \theta} \]

………………………………………… (5)
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where $\lambda$ is the X-ray wavelength (0.51417 nm), $D$ is the crystallite or particle size, $\beta$ is the breadth (rad) at half-maximum intensity of the reflection, and $\theta$ is the Bragg angle.

4-2- Complex dielectric constant & relative permeability ($\mu_r^*$ and $\varepsilon_r^*$)  
The frequency dependence of $\varepsilon_r'$, $\varepsilon_r''$ for Ba$_4$Co$_{0.7}$Zn$_{0.5}$Cu$_{1.1}$Fe$_{36}$O$_{60}$ composites is shown in figure (2) and (3). The real part of dielectric constant $\varepsilon_r'$ shows insignificant variation, while the imaginary part values of $\varepsilon_r''$ show a little increase as the measuring frequency 8 - 12 GHz increase for all the composites (calcined with different temperatures). The values of $\varepsilon_r'$ and $\varepsilon_r''$ are highest for ferrite calcined at 1100 °C and lowest for those at 1000 °C, which may be ascribed to the formation of Fe$^{+2}$ ions with the calcined temperature increasing. Figure (4) and (5) illustrates the frequency dependence of the $\mu_r'$ and $\mu_r''$ for all samples in the range from 8-12 GHz. It can be observed that the permeability ($\mu_r'$ and $\mu_r''$) increases with frequency increase for the entire sample, however, this increase is more rapidly at low frequencies than that at high frequencies, the values of $\mu_r'$ and $\mu_r''$ of the sample calcined at 1100 °C are higher than that of other samples below 1100 °C in low frequencies. The enhancement of $\mu_r'$ and $\mu_r''$ with calcined temperature increasing is also observed in figures (4) and (5), this can be ascribed to: (1) increasing in temperature results in the increasing of the samples density which facilitates the movement of the spins, (2) increasing in temperature also results in the number of pores decreasing which is favorable to the wall motion.

Figure (6) shows the reflection loss values as a function of frequency for ferrite composites calcined at different temperature (1000, 1050, and 1100 °C) with 2.0mm thickness using network analyzer system. As seen in figures (6), the sample of 2.0mm thickness calcined at 1100 °C showed better performance than that calcined below 1100 °C as RAMs. These samples having reflection loss lower than (-10 dB) over the frequency band (9.2 – 11.2 GHz), and its peak value are (-40 dB) at 10.2 GHz, while other calcined samples at temperatures bellow 1100 °C having reflection loss more than (-10 dB) over (x-band) frequencies.

Figure (7) shows frequency dependence of $\mu_r^*$ and $\varepsilon_r^*$ for samples under the test. It is probable that the absorber does not match a free space in the high frequency region because the parameters ($\varepsilon_r', \varepsilon_r''$) and ($\mu_r', \mu_r''$) has no frequency dispersion. It can be seen from figure(7) that ferrites calcined at 1100 °C shows larger electric and magnetic properties over (x-band) frequencies ,which can caused by the presence of Cu$^{+2}$,Co$^{+2}$ and Zn$^{+2}$ ions and formation of Fe$^{+2}$ ions in the U-type hexaferrite compositions.

4-3- Microwave losses and matching conditions  
The U-type hexaferrite material examined is having both dielectric and magnetic losses. The matching condition for perfect electromagnetic wave
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absorption is given by Z=1, we discuss briefly the matching conditions (Z₁, Z₂, and Z₃ in equation 4) for a single layer ferrite absorber with 2.0 mm thickness. Figure (8) illustrate the frequency dependence of dielectric (εᵣ/ε₀) and magnetic losses (μᵣ/μ₀) for U-type ferrite: EP composite at the frequency range of 8-12 GHz.

As can be seen, the dielectric losses are (about 0.03 - 0.3) and the magnetic losses are higher than dielectric ones (about 0.2-0.6) at frequencies of 8-12 GHz. It is also illustrates that the dielectric loss increases with rising frequency in the beginning until it reaches (0.3) at 10 GHz after which the value of (εᵣ/ε₀) very small increases with frequency increase and keeps as a constant at higher frequency, however, the magnetic loss (μᵣ/μ₀) is almost fluctuate around the constant values (about 0.6) over frequency range (10–12 GHz). Also it can be seen that the total losses increases at high frequencies may explain the performance of the ferrite sample absorbers, illustrated by the reflection loss curves in Figure (6).

Figure (9) shows frequency dependence of the matching conditions (Z₁= εᵣ/μ₀, Z₂= tanδₑ/μ₀, and Z₃= n = λ/d), it can be observed that the Z₁ decreases with frequency increase in the beginning until it reaches the value (2.2) at 10 GHz and keeps as a constant at frequency range (10-12 GHz), while, magnitude of Z₂ is less than unity and keeps as a constant at frequency range (8-12 GHz).

In the figure (9) the ratio (λ/d) is called matching thickness and takes the value (about 5.8). The electromagnetic wave propagation in ferrite is slower than that in free space by six times, this results can help us to design microwave absorber with thickness 2.0mm of U-type hexaferrite :epoxy(80:20% weight) composite.

4-4- Reflection loss of ferrite with different thickness

Figures (10) and (11) shows frequency dependence of reflection loss for U-type hexaferite epoxy (80:20% weight) composite at different thickness (1.25–4.0 mm), these figures illustrates that the reflection loss varies with varying absorbing thickness, and the absorbing frequencies is increase with decreasing thickness of ferrite samples. The substitution of Co⁺² by Cu⁺² and Zn⁺² ions, resulted in microwave absorption greater than 99.0%(reflection loss≤ -10 dB) at 9.2 – 11.2 GHz and 9.5 – 10.4 GHz for x-band, with 2.0 and 2.25 mm thickness, respectively.

5- Conclusion

U-type hexaferite with stoichiometric composition Ba₄Co₀.₂Zn₀.₆Cu₁.₁Fe₃₆O₆₀ has been prepared using citrate precursor method at calcinations temperatures (1100°C) lower (about 300 °C) than that of conventional method with good magnetic properties. Structure of the prepared samples has been characterized by the X-ray diffraction (XRD) technique. The XRD patterns of the prepared powders at temperatures (1000 and 1050°C) show the presence of spinel, M, and Y-type phases, while single phase of U-type ferrite(with crystallite size 40 – 90 nm)
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was obtained at 1100 °C. The partial substitution of Co$^{+2}$ for Zn$^{+2}$ and Cu$^{+2}$ ions in Ba$_4$Co$_2$Fe$_{36}$O$_{60}$ ferrite improved the microwave attenuation properties illustrated by the increase in the complex permeability, dielectric constant, losses (magnetic and electric) and microwave absorption (or reflection loss). The Ba$_4$Co$_{0.3}$Zn$_{0.6}$Cu$_{1.1}$Fe$_{36}$O$_{60}$: ER (80:20 weight percent) nanocomposite can be used as a potential magnetic loss material for X-band frequencies with microwave absorptions greater than 90% (reflection loss ≤ –10 dB) at the frequency range of 9.2-11.2 GHz with 2.0 mm thickness.

As a result, the (80:20 weight percent) nanocomposite of Ba$_4$Co$_{0.3}$Zn$_{0.6}$Cu$_{1.1}$Fe$_{36}$O$_{60}$: ER at 2.0 and 2.25 mm thickness showed the best performance as a RAM for the X-band, with a microwave absorption of 99.9% (reflection loss= –38 dB) and 99.8% (reflection loss= -32 dB) at 10.6 GHz and 9.8 GHz, respectively.

The electromagnetic wave propagation in ferrite is slower than that in free space by six times, this result may help us to design microwave absorber with thickness 2.0 mm of U-type hexaferrite: epoxy (80:20 weight percent) composite.

References
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Fig. (1): x-ray diffraction patterns of \( \text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60} \) U-type Precursors calcined at various temperatures: (a) at 1000 °C. (b) at 1050 °C. (c) at 1100 °C. (M: \( \text{BaFe}_{12}\text{O}_{19} \), S: \( \text{BaFe}_2\text{O}_4 \), Y: Y-type, and U: U-type)

Fig. (2): Frequency dependence of \( \varepsilon'_\text{r} \) of \( \text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60} \) ferrite calcined at different temperatures for 4 h.
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Fig. (3): Frequency dependence of $\varepsilon''$ of $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite calcined at different temperatures for 4 h

Fig. (4): Frequency dependence of $\mu''$ of $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite calcined at different temperatures for 4 h

Fig. (5): Frequency dependence of $\mu''$ of $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite calcined at different temperatures for 4 h
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Fig. (6): Frequency dependence of reflection loss of $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite calcined at different temperatures for 4 h.

Fig. (7): Frequency dependence of parameters($\mu_r'$, $\mu_r''$, $\varepsilon_r'$ and $\varepsilon_r''$) for $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite samples calcined at 1100 °C for 4 h.

Fig. (8): Frequency dependence of electric and magnetic tangent loss for $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite samples calcined at 1100 °C for 4 h.
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Fig. (9): Matching conditions $Z_1$, $Z_2$ and refractive index (n) as a function of frequency for $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite samples calcined at $1100^\circ\text{C}$ for 4 h.

Fig. (10): Frequency dependence of reflection loss of $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite samples with different thickness calcined at temperatures $1100^\circ\text{C}$ for 4 h.

Fig. (11): Frequency dependence of reflection loss of $\text{Ba}_4\text{Co}_{0.3}\text{Zn}_{0.6}\text{Cu}_{1.1}\text{Fe}_{36}\text{O}_{60}$ ferrite samples with different thickness calcined at temperatures $1100^\circ\text{C}$ for 4 h.
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يمكنني التعرف على صور الأوراق المكتوبة بشكل طبيعي.