

Preparation of Superparamagnetic

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⁴ Preparation of Superparamagnetic $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ Particle by Coprecipitation-Sonochemical Method for Radar Absorbing Material

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Abstract. One of many applications of spinel ferrite nanoparticles is related to their performance as radar absorbing materials. In this work, we report developing synthesis method through combined coprecipitation-sonochemical routes in preparing $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle from iron sand in Indonesia as a vital raw material. The structure, size, morphology, and elements of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle were investigated via X-Ray diffractometry and Transmission/Scanning Electron Microscopy (TEM/SEM) combining Energy Dispersive Spectroscopy (EDS). The magnetic properties of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle were characterized by using Vibrating Sample Magnetometer (VSM). Furthermore, the reflection loss character of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle was determined via Vector Network Analyzer (VNA). From the qualitative and quantitative analysis of the XRD data, it can be identified that the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle formed a spinel cubic structure in a single phase with the lattice parameter of approximately 8.401 Å. It is known from the TEM image that the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle had a size of about 9.7 nm and tended to agglomerate. Furthermore, the data analysis of the $M(H)$ curve presented that the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle has a superparamagnetic behavior with the saturation magnetization of approximately 43 emu/g. Finally, the data analysis of the reflection loss as a function of frequency showed that the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle performs as a radar absorbing material with the absorption performance of approximately -11.0 dB at the frequency of 10.8 GHz



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Keywords: $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$, nanoparticle, coprecipitation-sonochemical method, superparamagnetic, radar absorbing material.

1. Introduction

Spinel ferrite nanoparticles have general formula AB_2O_4 and form spinel crystal structure, where A represents the metallic cations located at the tetrahedral site, and B represents the metallic cations positioned at the octahedral site [1]. Spinel ferrite nanoparticles are a group of functional magnetic materials that have attracted significant attention due to their high performance in various applications. Nowadays, several researchers have extensively reported that the spinel ferrite nanoparticles have excellent application performances in many fields. The applications include for hyperthermia treatment [2], energy storage [3], core, switching and MLCI's [4], as a catalyst to eliminate organic pollutants [5], electromagnetic shielding [6], microwave absorption and high-frequency [7], and so forth.

Numerous efforts have been conducted intensively and continuously by many researchers to obtain the high-performance spinel ferrite nanoparticles as radar absorbing materials. It is imperative because radar absorbing materials have an essential role in fulfillment in civil and military purposes [8]. The efforts have been conducted through developing synthesis method, expanding characterization technique, and also developing a theoretical explanation to achieve the thin, flexible and light absorbers with high reflection loss performance.

For mass production, it is essential to develop the synthesis method efficiently and inexpensively in preparing spinel ferrite nanoparticles as radar absorbing materials. However, many papers reported that the ferrite nanoparticles as radar absorbing materials are prepared from commercial precursors which are relatively expensive. Therefore, to reduce production cost, in this experiment, we propose the utilization of iron sand from natural resources in Indonesia in preparing $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{O}_4$ nanoparticle. Among various synthesis methods, the coprecipitation approach has advantages due to its simplicity, effectivity, low price, and ability in producing spinel ferrite nanoparticles [9]. Furthermore, the sonochemical approach also is also superior regarding its high performance in preparing the nanoparticles in various shapes and sizes with high homogeneity. Therefore, we also propose a combination of the two approaches in synthesizing the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{O}_4$ nanoparticle. Finally, we complete this works by discussing in detail the structural, magnetic, and microwave absorption characteristics of the prepared $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle.

2. Experimental Method

2.1 Sample Preparation

The $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle was prepared by using a combined coprecipitation-sonochemical method using natural iron sand as a primary raw material. The iron sand, was taken from Sine Beach Tulungagung - East Java Indonesia, was extracted and purified by using a magnetic separator to obtain the Fe_3O_4 powder following our previous works [10,11]. The process of the powder was reacted with HCl (Merck, PA) to form a FeCl_2 and FeCl_3 solution tracking our previous experiments [12–14]. The solution was then mixed with ZnCl_2 (Merck, PA) and $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (Merck, PA) by using a magnetic stirrer. The process was continued by wisely dropping NH_4OH (Merck, PA) and combined with the sonochemical process by using an Ultrasonic Batch also at room temperature to obtain the black precipitated sample. The precipitated sample was washed several times by using distilled water until it reached a normal pH condition. Finally, the precipitated sample was filtered before drained at 100 °C to obtain the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle.

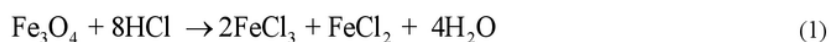
2.2 Characterizations

The crystal structure was investigated using X-ray Diffractometry (XRD) with Cu-K α radiation in the range of 10° – 80°. The XRD data were analyzed by using Rietica program [15] based on Rietveld

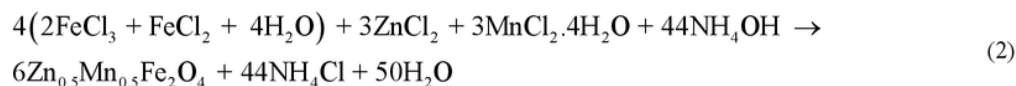
refinement approach [16] to obtain the phase purity, crystal structure, lattice parameters, and crystallite size of the sample. The particles size, morphology and elemental composition of the sample were investigated by means of Transmission/Scanning Electron Microscopy (TEM/SEM) combined with energy-dispersive spectroscopy (EDS). The magnetic properties of the sample, as well as saturation magnetization, coercive field, and remanent magnetization, were characterized by using Vibrating Sample Magnetometer (VSM) at room temperature. Finally, the performance of the sample as a radar absorbing material was investigated by using vector network analyzer (VNA) at a frequency in the range of 8 - 12 GHz.

3. Results and Discussion

The chemical reaction of the Fe_3O_4 from iron sand dissolved in HCl tracks the following equation of reaction [17].



From reaction (1), the metal salt solution containing Fe^{3+} and Fe^{2+} can result only through one step of reacting natural Fe_3O_4 with HCl. The prepared solution from the first reaction was then mixed with ZnCl_2 and $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$, and then reacted with NH_4OH by dropping process to form the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle. The sonochemical route was also applied to reducing the agglomerated particles of the sample during the precipitating process. The detail of the chemical reaction is presented in equation of reaction (2). The remained or unwanted product in this reaction was removed by a washing process using distilled water for several times to achieve a normal pH condition and then followed by a heating process at 100 °C.



The obtained $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle was characterized using XRD to investigate the phase purity, crystal structure, as well as lattice parameter and crystal volume, and also crystallite size. The XRD pattern of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle is presented in Figure 1.

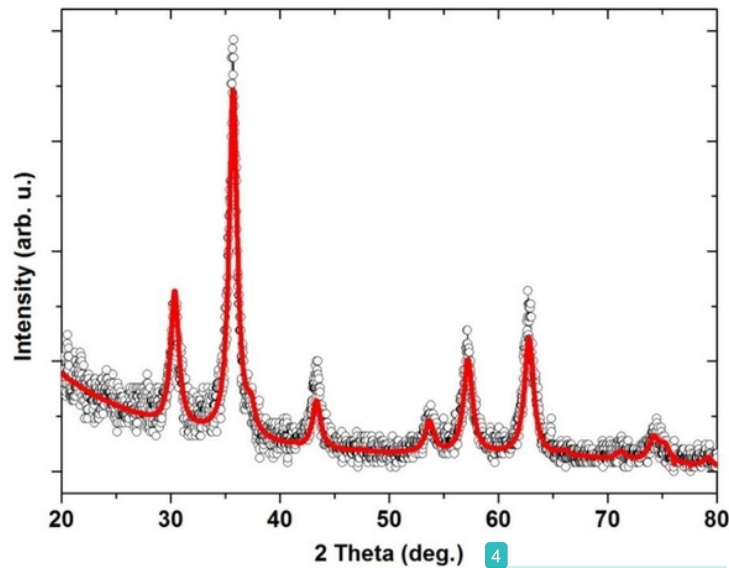


Figure 1. X-ray diffraction pattern and of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle synthesized by the coprecipitation-sonochemical method

The XRD data as illustrated in Figure 1 were analyzed qualitatively and quantitatively. From the qualitative analysis, the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle formed a single phase without impurity. Meanwhile, from the quantitative analysis, the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle built a spinel crystal structure in the cubic system. The measured XRD data and the Rietveld refinement plot are, respectively, shown by the circles and solid line. The lattice parameter of the crystal can be calculated using the following equation [18].

$$a = \frac{\lambda}{2} \sqrt{\left[\frac{(h^2 + k^2 + l^2)}{\sin^2 \theta} \right]} \quad (3)$$

Where a , λ , (hkl) , and θ represent the lattice parameter, wavelength of X-ray, Miller indices, and Bragg angle, respectively. For this case, the lattice parameter is $a = b = c$, and the crystal volume is $V = a \times b \times c$ as the cubic structure with the space group of $Fd\bar{3}m$. From the quantitative analysis, the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle has the lattice parameters and crystal volume of approximately 8.401 Å and 592.9 Å³, respectively. These are in excellent agreement with the results reported by other researchers [19,20].

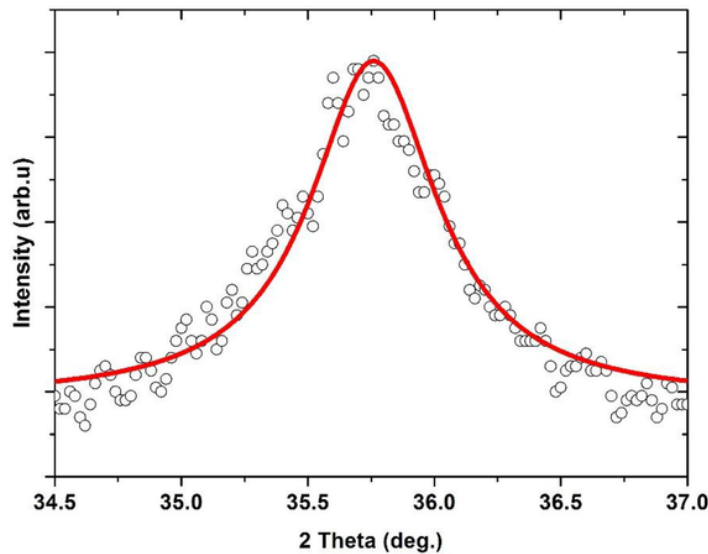


Figure 2. FWHM fitting (solid line) of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ XRD pattern (circle) synthesized by the coprecipitation-sonochemical method

Based on Figure 2, the crystallite size of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle was calculated by using Debye-Scherrer's formula to ensure that the particle formed in nanometric size. The fitting result of the FWHM for the highest peak is presented in Figure 2. The equation (4) represents the Debye-Scherrer's formula [21].

$$D = \frac{K \cdot \lambda}{\beta \cos \theta} \quad (4)$$

Where D is the crystallite size (nm), K is the constant of 0.9, θ is the Bragg angle (degree), β is the excessive line broadening (rad), and λ is the wavelength of X-ray (0.15406 nm). From the data analysis in this experiment, the crystallite size of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle prepared by the coprecipitation-sonochemical method is 10.7 nm. This result is quite similar to the crystallite size of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ prepared by precursor-based combustion method [22]. It means that the combined method is effective and efficient to prepare $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particle in nanometric size from local natural iron sand. Furthermore, the particle size is also compared to the TEM characterization of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle as shown in Figure 3.

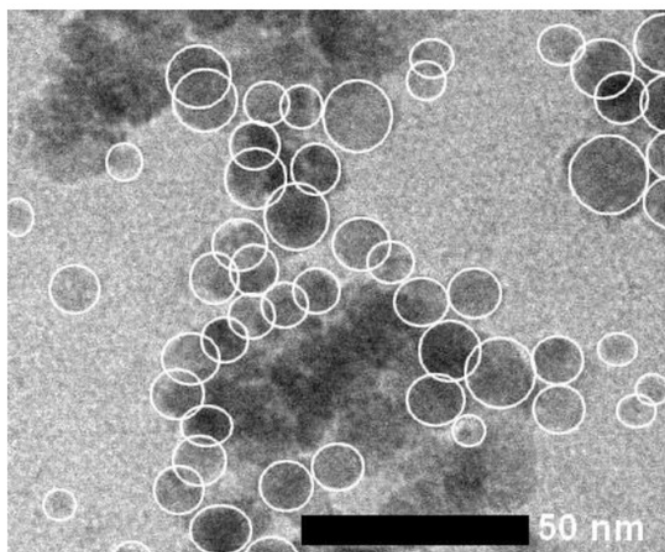


Figure 3. TEM image of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle synthesized by the coprecipitation-sonochemical method

Based on Figure 3, it is clear that the prepared $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle in this experiment has nanometric size. Quantitative analysis of the TEM image presented that the average particle size of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle is 8.7 nm with a standard deviation of 2.4 nm. This result is close to the size calculated from the XRD data. In this work, the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle from iron sand has smaller particle size than that of $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle from commercial precursors prepared by microwave induced urea nitrate and coprecipitation processes [23,24]. The $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle had a tendency to form a spherical shape with aggregation. The small particle size can contribute the phenomenon of agglomeration in the magnetic nanoparticles.

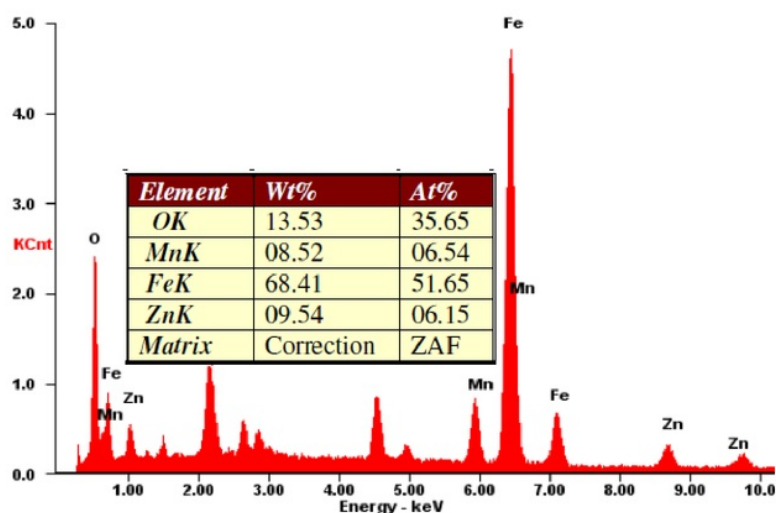


Figure 4. EDS Spectra and elemental quantification of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$

nanoparticle synthesized by the coprecipitation-sonochemical method

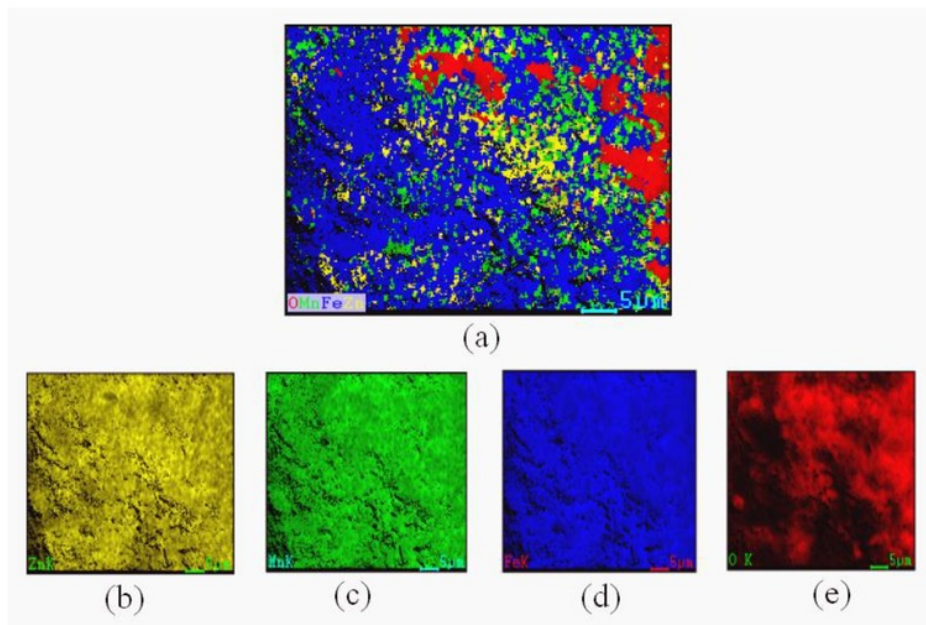


Figure 5. EDS Mappings of (a) all elements, (b) Zn, (c) Mn, (d) Fe, and (e) O for the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle synthesized by the coprecipitation-sonochemical method

The result of Rietveld analysis of the XRD data for the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle became physical evidence that the Mn and Zn ions successfully substituted Fe ion in the crystal structure. In order to enhance our knowledge about the composition and distribution of the metallic elements and oxygen, further investigation was also carried out by using SEM combined with the EDS. The investigation resulted in EDS spectra, elemental quantification as shown in Figure 4, and also elemental mapping for all elements and individual element as presented in Figure 5. From the data, the selected elements such as iron, zinc, manganese, and oxygen tended to distribute randomly in all space of the material. Furthermore, the distribution of iron was dominant because it has the highest composition. Interestingly, the atomic composition of zinc and manganese was quite similar representing that the theoretical calculation (Mn : Zn = 0.5 : 0.5) was appropriate for the experimental data.

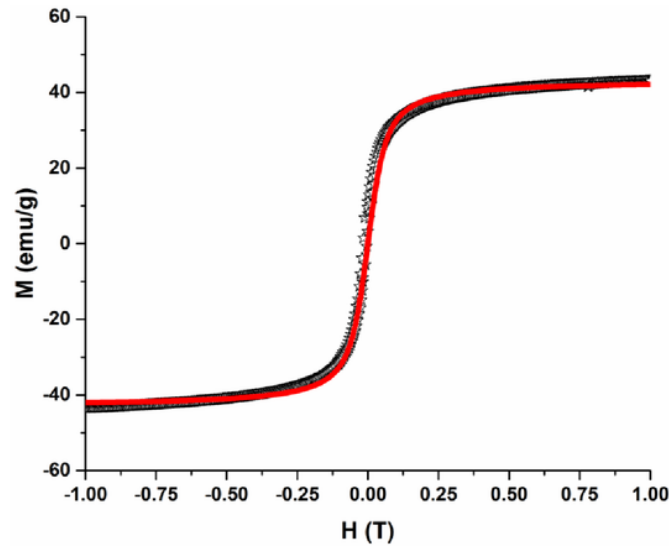


Figure 6. Magnetization curve of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle synthesized by the coprecipitation-sonochemical method

Figure 6 presents the magnetization curve $M(H)$ of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle at room temperature with the magnetic field (H) in the range of -1 to 1 T. Qualitatively, the $M(H)$ curve of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle displays S profile with the remanent magnetization (M_r) and the coercive force (H_c) values which are nearly negligible. Therefore, these characters pointed out that the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle demonstrates a superparamagnetic character [25]. Moreover, to study the magnetic properties comprehensively, the quantitative analysis of the $M(H)$ curve was conducted via fitting analysis. The data analysis was done by employing Langevin function according to the following equation [26].

$$M = M_s \left(\coth \left(\frac{\mu H}{k_B T} \right) - \frac{k_B T}{\mu H} \right) \quad (5)$$

Where M is the magnetization, M_s is the saturation magnetization, μ is the magnetic moment, H is the magnetic field, k_B is the Boltzmann constant, and T is the temperature. The solid line and star represent the fitting model and experimental data, respectively. The theoretical calculation fits the experimental data well with the R -square of 99.8%. From the result of data analysis, the M_s and $\mu H/k_B T$ values were approximately 43 emu/g and 40, respectively. The saturation magnetization value of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle prepared in this work is higher than that of $\text{Mn-ZnFe}_2\text{O}_4$ nanoparticles reported by other researchers [23,27]. The saturation magnetization of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ in the nanoparticle is lower than that of the bulk as ascribed to the particle size. The saturation magnetization of the spinel-structured materials such as $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle can be determined from the magnetic moments of individual ion, the ions distribution in the spinel crystal structure, and the exchange interaction between tetrahedral and octahedral positions.

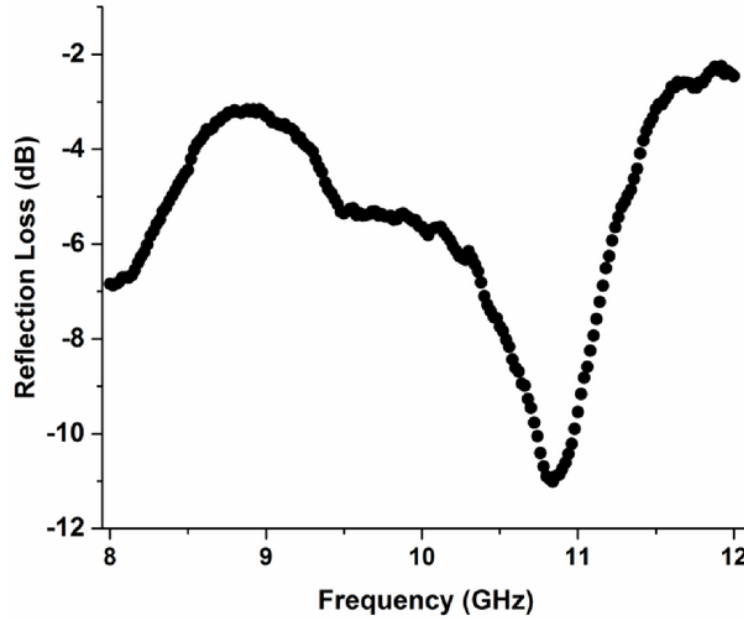


Figure 7. The reflection loss of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle synthesized by the coprecipitation-sonochemical method at the frequency ranged from 8 – 12 GHz

Figure 7 exhibits the reflection loss character of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle at a frequency ranging of 8 – 12 GHz. The reflection loss of the material which represents the ability to absorb electromagnetic wave can be determined by employing the following mathematical expression [28].

$$RL = 20 \log \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right| \quad (6)$$

Where RL , Z_0 , and Z_{in} are the reflection loss (dB), the characteristic impedance of free space, and input impedance, respectively. The characteristic impedance and the input impedance are presented in equation (7) and (8).

$$Z_0 = \left(\frac{\mu_0}{\epsilon_0} \right)^{\frac{1}{2}} \quad (7)$$

$$Z_{in} = \left(\frac{\mu_r}{\epsilon_r} \tanh \left\{ j \frac{2\pi f t}{c} (\mu_r \epsilon_r) \right\} \right)^{\frac{1}{2}} \quad (8)$$

Where μ_r and ϵ_r represent the complex permeability and permittivity, f represents the frequency of operation, t represents the thickness of the sample, and c is the light velocity.

Based on Figure 7, there were two reflection losses of the absorption peaks at frequencies of approximately -7.0 dB and -11.0 dB at 8 GHz 10.8 GHz, respectively. The electromagnetic wave absorption after calculating reaches about 80 % at the highest peak frequency of 10.8 GHz with a thickness of 1.0 mm. Low reflectivity of the material makes excellent absorption performance. It means that the strongest performance of the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle in term of absorbing electromagnetic wave is -11.0 dB at the frequency of 10.8 GHz. Therefore, the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle prepared by a coprecipitation-sonochemical method in this experiment exhibits its sustainability as a radar absorbing material, especially for military purposes. Usually, the radar absorbing materials for military applications are maintained at a frequency in the range of 8 to 12 GHz [8]. The materials should have magnetic and dielectric characters with proper impedance and interaction with the electromagnetic energy to give a high performance in absorbing electromagnetic wave.

4. Conclusion

The $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle was successfully prepared by employing a mixed coprecipitation-sonochemical method using iron sand as the core precursor. From the XRD data analysis, the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ particles formed a spinel cubic structure in a single phase with the particle size in nanometric scale and a lattice parameter of approximately 8.401 Å. Furthermore, the data analysis of the $M(H)$ curve presented that the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle has a superparamagnetic behavior with the saturation magnetization of approximately 43 emu/g. Finally, the data analysis of the reflection loss as a function of frequency showed that the $\text{Zn}_{0.5}\text{Mn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticle performs a radar absorbing material with the absorption performance of -11.0 dB at the frequency of 10.8 GHz.

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