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Barium M-Hexaferrite $BaFe_{12}O_{19}$ Based on Magnetic Properties and Microwafe Absorption

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Abstract

Barium M-hexaferite is a material which has the potential to be developed as a magnetic material which is needed in various fields of application. Various methods have been used to produce this material, but in general these methods require heating at relatively high temperatures. This research was conducted to characterize the magnetic properties and microwave absorption of barium M-hexaferrite which was synthesized using the coprecipitation method by sintering at low temperatures. Synthesis of barium M-hexaferite was carried out using $BaCO_3$, $FeCl_3$. $6H_2O$, HCl, NH_4OH. The results of the coprecipitation were dried using a light bulb at a temperature of 85^0C, then sintered at a 300^0C with variations in holding times of 1 hour, 3 hours and 5 hours. The magnetic properties of the synthesized materials were tested using VSM, while the properties of microwave absorption were characterized using VNA. From the VSM test results, it can be determined that the highest remanence magnetization and coercivity values were found in the samples sintered at 300^0C for 3 hours, namely Mr 0.103 emu/gram and Hc 0.058 Tesla. Meanwhile, from testing the absorption properties of the material for microwaves using VNA, it was found that the largest reflection loss value was found in the sample which was calcined for 3 hours, which was 52.4 dB or equivalent to an absorption of 99% at a frequency of 11 GHz. From the research it was found that barium M-hexaferite as a result of coprecipitation can be developed into a good microwave absorbent material.

Keywords: barium M-Hexaferrites, coprecipitation, magnetic properties, microvwave absorption

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INTRODUCTION

Nanotechnology has produced many smart magnetic materials, one of wich is a microwave absorbing material. Radar Absorber Material (RAM) is a material used in sealth technology to disguise the position and structure of vehicle from radar. The effectiveness of radar camouflage depens on how much electromagnetic wafe energy can be absorbed by absorber material used. One of the dominant factors influencing the performance of the absorber material is the magnetic properties of the material. Barium hexaferrite is a material that has a high magnetic loss factor (Sulistyo et al., 2012).

Barium M-hexaferrite is one of a type of barium hexaferrite material which has the potential to be developed. Barium M-hexaferrite material has been widely developed as a magnetic material which is needed in various fields of application, because this material has characteristics including relatively high Curie temperatures, coercivity values, magnetic saturation, and high magnetic anisotropy, as well as very good chemical stability (Simbolon et al., 2013; Sahlam, et al., 2018; and Purnama et al., 2015). Barium hexaferrite as a ferrite magnet, besides having relatively high permeability, permittivity and spontaneous magnetization, is also composed of oxide components so that it also has high electrical

resistivity or a good insulator. The combination of the intrinsic properties of the magnetic properties and the electrical properties of such ferrite places the ferrite magnetic material as an absorber of microwaves including waves with frequencies used in radar (Sulistya et al., 2012; Kanagesan et al, 2014).

The physical properties of barium M-hexaferite are greatly influenced by the synthesis process, chemical composition, temperature and sintering period, type and number of substitutions (Zhang et al., 2013). Various methods have been used to produce hexaferrite powder, namely: solid state method, wet chemistry method, high energy milling method, sol-gel method, and hydrothermal method. However, these methods require heating at relatively high temperatures (Johan, 2011; Priyono & Manaf, 2007; Purnama et al., 2015). In this research, the synthesis and characterization of nanocrystalline barium hexaferrite powder will be carried out using the coprecipitation method, it is hoped that by relatively low heating temperatures a barium hexaferite phase can be obtained. This is supported by the statement of Permana et al. (2017) that several studies have successfully synthesized magnetic nanoparticles using the coprecipitation method. The results of the synthesis of magnetic powders using the coprecipitation method at room temperature have produced Fe3O4 magnetic nanoparticles (Permana et al., 2017).

The coprecipitation method is a method of synthesizing inorganic compounds which is based on the deposition of more than one substance together when passing through the saturation point. Coprecipitation is a promising method because the process uses low temperatures and it is easy to control particle size, besides the time required is relatively short. By using the coprecipitation method, the crystal structure and magnetic properties of the synthesized material can be optimized by controlling synthesis parameters such as temperature, solvent, pH of the solution, stirring speed, stirring time, metal salt concentration, and surfactant concentration (Muflihatun et al., 2015). Some of the substances commonly used as precipitating agents in the coprecipitation method are hydroxides, carbonates, sulfates, and oxalates.

The magnetic properties of a material are determined by the magnitude of the remanent induction value or remanence magnetization value (Mr) and the coercivity field (Hc). The Mr value indicates the magnetization remaining in the material when the external field H has been removed. This shows the ability of the material to magnetize when given an external field. The greater the Mr value of a material, the greater its magnetic properties. The coercivity field is the magnitude of the field required to make the magnetism of a material equal to zero. So the coercivity field is the external field needed to remove the residual magnetism of the material. The greater the Hc value, the greater the magnetic properties (Jatiutoro et al., 2007).

This research was conducted with the aim of characterizing barium M-hexaferite powder which was synthesized using the coprecipitation method using sintering at low temperatures. The characterization of the magnetic properties of barium M-hexaferrite used a vibrating sample magnetometer (VSM) while the characterization of the microwave absorption properties used a vector network analyzer (VNA).

METHOD

In this research, barium M-hexaferrite powder was synthesized using the coprecipitation method. The main materials used in this study were barium carbonate ($BaCO_3$), $FeCl_3.6H_2O$, HCl (12.063 M), NH_4OH (6.5 M) and distilled water. $FeCl_3$ powder was dissolved in distilled water until completely dissolved (indicated by the absence of precipitate in the solution). $BaCO_3$ powder was reacted with HCl solution using a magnetic stirrer, then added distilled water until completely dissolved. The two solutions are mixed while continuously stirring and heated with a magnetic stirrer. Then add NH_4OH little by little until a brownish yellow precipitate forms. The precipitate is rinsed with distilled water and precipitated using a permanent magnet. The precipitated powder is dried using a light

bulb until completely dry, then sintered at 300^0C with variations in holding time of 1 hour, 3 hours and 5 hours.

Identification of the synthesized sample phase was carried out by conducting a Philips XPert MPD (Multi Purpose Diffractometer) x-ray diffraction test at the RC X-Ray Diffraction Laboratory (Research Center) LPPM ITS Surabaya. Measurement characterization takes an angle of 2θ from 20^0 to 70^0 with a step of 0.04^0. The phase identification process is based on matching the data of measured diffraction peak positions with a database. Phase identification is also called search-match which can be done manually or computer-based. Data from the test results with XRD were analyzed using Rietica and MAUD software. Rietica software is used to identify the phases formed in the synthesized material, while MAUD is used to identify the phase composition and estimate crystal size.

Furthermore, samples that have been sintered at 300^0C are characterized using a Vibrating Sample Magnetometer (VSM) and a Vector Network Analyzer (VNA). VSM measurements are carried out to determine the magnetic properties of materials through the resulting hysteresis curves, such as saturation magnetization, remanent magnetization, and coercivity fields. VSM is a magnetization measuring instrument that works based on the induction method. Data from the VSM test results will be analyzed using the origin software. The VNA test can be used accurately to measure the scattering parameters of a material in the frequency range of 10 MHz to 110 GHz. VNA measures materials over a specific frequency range. At a given frequency, a signal is sent out from section 1 of the VNA to the device under test (DUT) section. It is possible that some of the signal is reflected back in section 1, some may be dissipated in the DUT (or called irradiation), and some may be transmitted in section 2. Accurate measurement of the VNA is a complex comparison of the reflected signals (reflections) with the initial signal (S_{11}) as compared between the transmitted signal (transmitted) and the initial signal (S_21). Likewise the sending signal out of section 2 can be used to measure S_{12} and S_{22}.

RESULTS AND DISCUSSION

The results of sample testing using XRD were then analyzed using Rietica and MAUD software. Figure 1 below is a graph showing the diffraction pattern of powder sintered at 300^0C for 1 hour, 3 hours and 5 hours. From the figure it can be seen that the synthesized sample contains barium M-hexaferite ($BaFe_{12}O_{19}$) and hematite (α-Fe_2O_3) phases. From the phase identification using Rietica software, it was found that among the coprecipitated powders which had been sintered at 300^0C which produced the $BaFe_{12}O_{19}$ phase with the highest purity was the sample sintered for 3 hours which was 53.02%. Of the three samples showing the same diffraction pattern, what is different is the intensity or peak height of each sample.

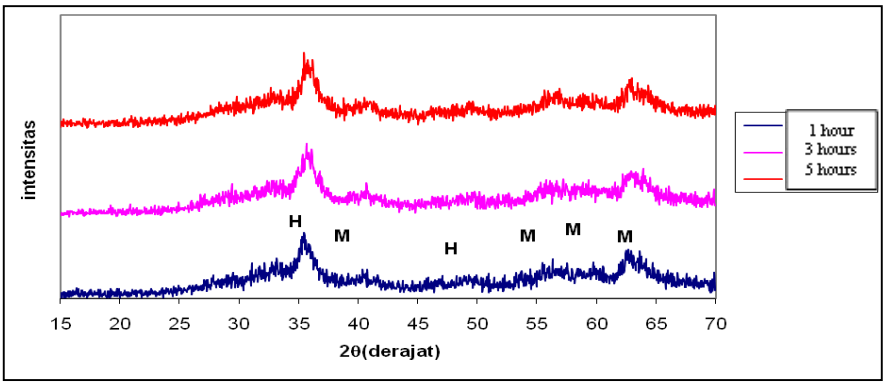

Figure 1. X-ray diffraction patterns of samples sintered at 300^0C with various holding times of 1 hour, 3 hours and 5 hours.

Table 1 below shows the phase composition of the samples sintered at 300^0C with various holding times of 1 hour, 3 hours and 5 hours.

Table 1. The phase composition of the results of diffraction data analysis using Rietica software

Sintering Temperature	GoF	$BaFe_{12}O_{19}$ Phase (% Wt)	α-Fe_2O_3 Phase (% Wt)
300^0C, 1 hour	3.73	49.32	50.68
300^0C, 3 hours	3.95	53.02	46.98
300^0C, 5 hours	3.84	51.91	48.09

Based on the refinement results using MAUD software, the estimated crystal size value of the synthesized powder was obtained at 300^0C. Table 2 shows the estimated crystal size of the refined results using MAUD.

Table 2 Estimation of crystal size from diffraction data analysis using MAUD software

Sintering Temperature	Rw (%)	Rb (%)	Rexp (%)	Sigma	Crystal Size (nm)
300^0C, 1 hour	26.53	20.97	21.09	1.25	88.90
300^0C, 3 hours	24.95	19.72	20.59	1.21	88.33
300^0C, 5 hours	29.74	23.20	23.14	1.28	72.26

Based on Table 2, it is known that the approximate size of the barium M-hexaferite crystal powder was synthesized by the coprecipitation method and then sintered at 300^0C on average below the order of 100 nm. This is in accordance with the statement put forward by Permana et al. (2017) that the coprecipitation method is a synthesis method that has been widely used by researchers because it is quite effective for producing nanoparticle materials (Permana et al., 2017).

VSM measurements were carried out to determine the magnetic properties of materials through the resulting hysteresis curves, such as saturation magnetization (Ms), remanent magnetization (Mr), and coercivity fields (Hc). Measurement of the magnetic properties of materials is carried out as a function of magnetic field, temperature, and time. The resulting hysteresis curve gives the relationship between the magnetization (M) and the applied external magnetic field (H). Figure 2 shows the hysteresis curve of the synthesized samples. From the hysteresis curve of the test results with VSM, the parameters of the magnetic properties of the material that can be determined are only the remanence magnetization value (Mr), the coercivity field (Hc), and the magnetization value of the material when the external field is given is 1 Tesla (M_{1T}) which is shown in Table 3. The saturation magnetization value of the material cannot be measured because barium M-hexaferite is a hard magnet that has a large saturation magnetization value, while the VSM used to test magnetic properties is only able to work in a magnetic field of up to 1 Tesla.

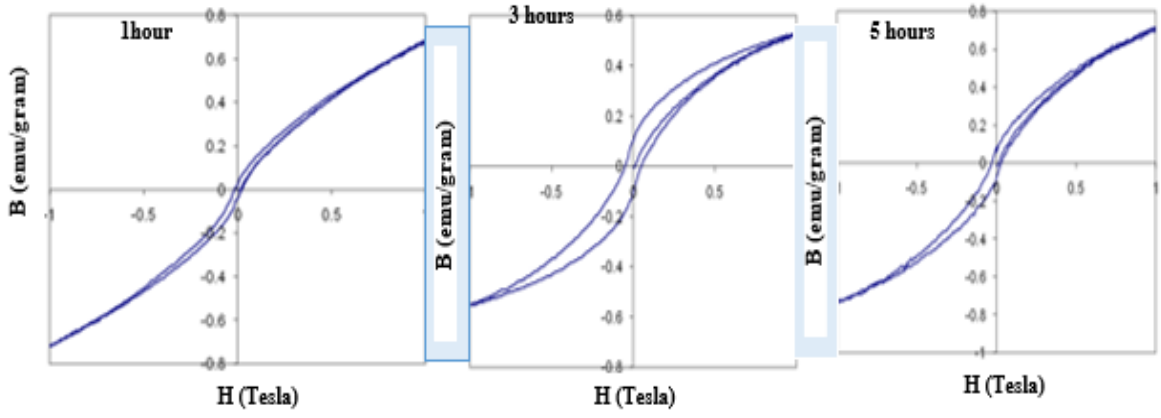

Figure 2. Hysteresis curve of the $BaFe_{12}O_{19}$ wich was sintered at 300^0C

Table 3. Magnetization values at 1 Tesla, remanence magnetization, and coercivity of sintered samples

Sintering Temperature	M_{1T} (emu/gram)	Mr (emu/gram)	Hc (Tesla)
300^0C, 1 hour	0,68	0,024	0,018
300^0C, 3 hours	0,53	0,103	0,058
300^0C, 5 hours	0,69	0,058	0,026

The remanence magnetization value of each successive sample is 0.024; 0.103; and 0.058 emu/gram. The remanence magnetization shows the magnetization that remains when the external H field has been removed. This shows the ability of the material to magnetize when given an external field. The greater the Mr value of a material, the greater its magnetic properties.

The coercivity field is the magnitude of the field required to make the magnet equal to zero. So the coercivity field is the external field needed to remove the residual magnetism of the material. The greater the Hc value, the greater the magnetic properties. From the samples tested, the Hc values were 0.018 each; 0.058; and 0.0257 Tesla.

There are several possibilities that cause the low magnetic properties of the material when judged by the low value of the remanence magnetization and coercivity field. The first possibility is the low magnetic properties of the material due to the results of phase identification by X-ray diffraction indicating that the sample produced is not a single phase of barium M-hexaferite but still contains a hematite phase. Barium M-hexaferrite is ferrimagnetic while the α-Fe_2O_3 phase is diamagnetic, that is, it repels magnetic fields. This of course has a big effect on the magnetic properties of the sample produced. This is reinforced by the results of research conducted by Muflihatun et al. (2015) that the presence of the α-Fe_2O_3 phase which has anti-ferromagnetic properties can reduce the magnetization value (Muflihatun et al., 2015). The structural form of barium M-hexaferite is hexagonal, because the resulting sample contains other phases so that the crystalline form of the resulting particles is not a perfect hexagonal shape. This allows for defects in the material which can reduce the value of the magnetism parameter in the material.

The low values of the remanence magnetization and coercivity field as well as the narrow area of the hysteresis curve strengthen the crystal size estimation obtained through the MAUD software (approximately 80 nm). Some theories say that the particle surface effect, small crystal size, and sample inhomogeneity will affect the low remanence magnetization value of the material. The smaller the crystal size, the smaller the number of domain walls. The domain wall contributes to the magnitude of the remanence value and the coercivity field of the material. The more domain walls, the higher the required coercivity field value, conversely the fewer domain walls have an impact on decreasing the coercivity value. The lack of domain walls is also indicated by the area of the curve which describes the energy level required for magnetization as well as for demagnetization in the material. Domain walls inhibit domain growth so that the energy required for magnetization becomes greater. The narrow area of the curve illustrates that the energy required for magnetization is also small (Mashuri, 2012).

Ferrite materials that have crystal sizes on the order of micrometers have limitations in being able to be used at high frequencies (GHz). This limitation is caused by the low resonant frequency (of the order of MHz). The resonant frequency that occurs is due to the movement of the domain wall. This problem can be overcome by synthesizing ferrite with sizes in the order of nanometers. The coprecipitation method is preferred because it is capable of producing ceramic powders with crystal sizes below 100 nm.

From the hysteresis curve, the highest values of remanence magnetization and coercivity field among the three samples were powder sintered at 300^0C for 3 hours. This relates to the results of phase identification resulting from x-ray diffraction using Rietica software, which showed that the sample sintered for 3 hours contained the highest barium M-

hexaferite phase composition, namely 53.02%. If seen from the shape of the flat curve and the small value of the coercivity field, the synthesized sample is ferrimagnetic towards superparamagnetic. Superparamagnetic materials have a coercive field (Hc) of zero value.

The next step is the measurement of VNA which is carried out to characterize the absorption properties of the material against microwaves. Return Loss can be obtained by measuring the scattering parameters using a Vector Network Analyzer (VNA). From testing the absorption properties of microwaves using VNA, it was found that the S_{11} value data illustrates the energy value that is reflected back by the material. S_{11} consists of real and imaginary values. The value of S_{11} can be obtained using Equation (1), where α and β are the real and imaginary values of S_{11} (Nam, I.W. et al., 2011).

$$RL = S_{11}(dB) = 20 \log_{10} \sqrt{\alpha^2 + \beta^2} \qquad (1)$$

To determine the percentage of absorbed power, generally use the following equation (2):

$$RL(dB) = 10 \log_{10} \frac{P_i}{P_r} \qquad (2)$$

where RL (dB) is the return loss in dB units, P_i is the power of the incident wave and P_r is the power of the reflected wave.

Measurements were made in the 6-18 GHz frequency range. Figure 3 shows the characteristics of the microwave absorption of the synthesized samples sintered at 300^0C. From the figure it can be seen that the sample sintered at 300^0C for 3 hours has a more obvious effect on microwave absorption than the other two samples. At a frequency of around 11 GHz, samples that were calcined for 3 hours had a reflection loss or return loss of up to 52.4 dB or the equivalent of an absorption value of 99.99%, while samples sintered for 1 hour and 5 hours respectively has a reflection loss of up to 39.1 dB and 24.4 dB. Table 4 shows the return loss values and the percentage of absorption of microwaves from the three samples at several frequencies.

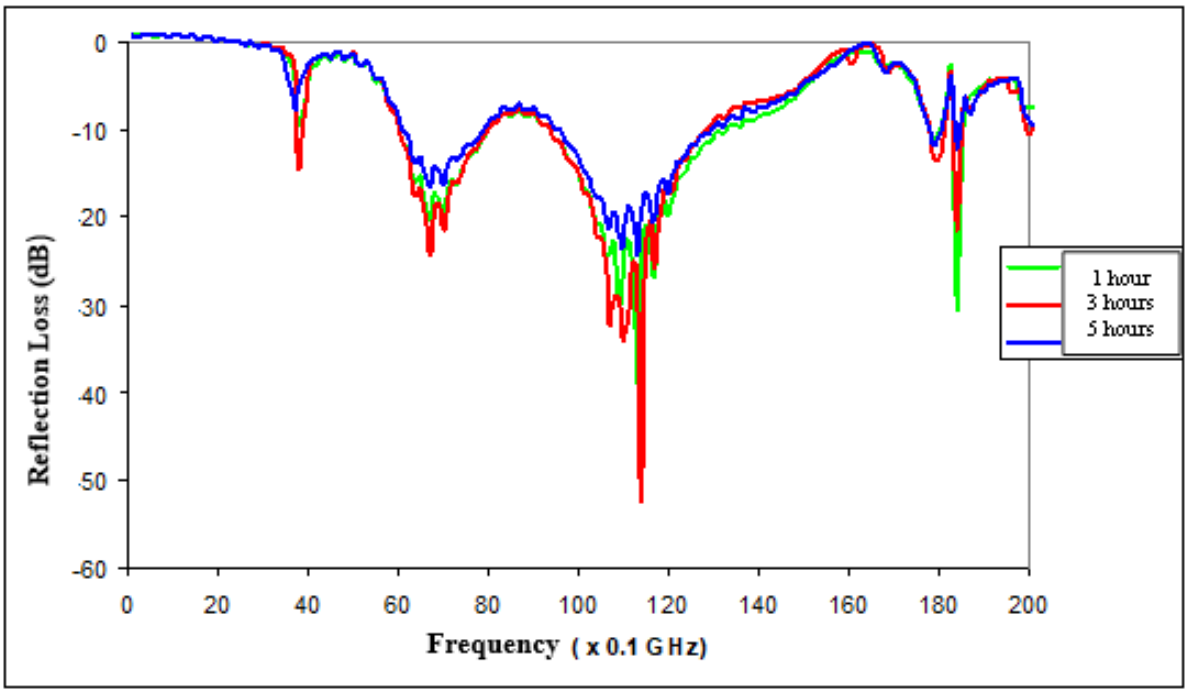

Figure 3. Characteristics of the microwave absorption properties of the samples sintered at 300^0C with variations in holding time for 1 hour, 3 hours, and 5 hours.

Tabel 4. The value of the reflection loss or return loss (RL) and the percentage of absorption from the sample sintered at 300^0C

Sintering Temperature (^{0}C)	Frequecy RL (GHz)	RL (-dB)	Absorption (%)
300, 1 hour	4	9.04	87.50
300, 3 hours	4	14.3	96.28
300, 5 hours	4	7.82	83.40
300, 1 hour	6	19.6	98.90
300, 3 hours	6	24.3	99.62
300, 5 hours	6	16.3	97.66
300, 1 hour	11	39.1	99.98
300, 3 hours	11	52.4	99.99
300, 5 hours	11	24.4	99.63
300, 1 hour	18	30.7	99.91
300, 3 hours	18	21.5	99.29
300, 5 hours	18	13.5	95.53

The magnitude of the material's ability to absorb microwaves is highly dependent on the phase content in the sample. The sample which was calcined at 300^0C for 3 hours had the largest $BaFe_{12}O_{19}$ phase content compared to the other two samples, so it had the greatest ability to absorb microwaves. The two phases ($BaFe_{12}O_{19}$ and α-Fe2O3) contained in the three samples, the $BaFe_{12}O_{19}$ phase has the ability to absorb microwaves. Barium M-hexaferrite is classified as a hard magnetic material that has a high coercivity field value, so that in its application barium M-hexaferite is synthesized by substituting other non-magnetic metal cations that are almost the same size (Al^{3+}, Ga^{3+}, Co^{2+}, Ti^{4+}, Zn^{2+}). The addition of Co and Zn showed a reduction in the axial anisotropy of Barium M-Hexaferrite. However, in this study barium M-hexaferrite was produced which had a relatively low remanence magnetization (Mr) value of 0.103 emu/gram for samples sintered at 300^0C for 3 hours. This remanence magnetization (Mr) value is lower than that of a study conducted by Purnama et al (2015), barium hexaferrite produced by the same method but sintered at 800^0C, 900^0C and 1000^0C has Mr value of 28.34 respectively; 27.20; and 26.14 emu/gram. Likewise, with a lower coercivity (Hc) field value compared to research conducted by Rosyidah (2013), the resulting barium M-hexaferite experienced a decrease in Hc value from 0.1734 Tesla to 0.058 Tesla. So even though it has not been substituted for non-magnetic metal ions, the resulting sample has a large absorption value or return loss. So the synthesized material can be used as a good microwave absorbent material, which has a relatively low coercivity value but has a high absorption value.

CONCLUSION

From the data analysis that has been done, it can be concluded that the coprecipitation method has been able to produce barium M-hexaferite powder. From the analysis of diffraction data using Rietica and MAUD software, it is known that samples sintered at 300^0C for 3 hours contain the highest barium M-hexaferrite ($BaFe_{12}O_{19}$) phase, which is equal to 53.02% with an estimated crystal size of 88.33 nm. The results of sample testing using VSM, it is known that of the three samples that have the largest remanence magnetization (Mr) and coercivity (Hc) values are the samples which were sintered at 300^0C for 3 hours, namely Mr 0.103 emu/gram and Hc 0.058 Tesla . The sample that was sintered for 3 hours had the highest return loss value compared to the other two samples, which was 52.4 dB or equivalent to an absorption of 99.99% at a frequency of 11 GHz.

RECOMMENDATION

Based on the experience in this research, there are several things that need to be considered, namely: During the synthesis, the environmental conditioning (pH, temperature)

is made the same for each experiment. The use of the furnace in each calcination is endeavored to use the same furnace. Sample preparation for VSM testing is still not good, because it uses mica plastic which can affect the results of the material's microwave absorption, therefore it is necessary to think about a better form of sample preparation.

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