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Synthesis of Barium M-Hexaferrite Using Co-precipitation Method with Zn Doping Based on Natural Iron Sand at Tebing Beach, North Lombok as Microwave Absorbent Material

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Abstract: This study aims to synthesize barium M-hexaferite using the coprecipitation method with Zn doping based on natural iron sand at the Tebing Beach of North Lombok as a microwave absorbent material. This study used 3 variations of doping concentration (x), namely x = 0.000; 0.400; and 0.800. In addition, variations in the calcination temperature were also carried out, namely 400°C and 1100°C. After the calcination stage, the sample is ground to obtain smaller or nano-sized particles. The results showed that the sample has a brick red color for a temperature of 400°C and black for a temperature of 1100°C. This shows that the higher the calcination temperature, the darker the color of the sample.

Keywords: Barium M-hexaferrite; Co-precipitation; Iron sand; Microwaves

Introduction

Indonesia is a country with abundant natural resources, one of which is natural iron sand (Susilawati et al., 2021). Iron sand is a source of natural magnetic material that can be obtained along the coastline (Elinda et al., 2022; Rahmawati et al., 2013; Susilawati et al., 2018). Lombok Island has a coastline of 2,333 km, this certainly makes Lombok Island's iron sand a potential for development, one of which is at the Tebing Beach of North Lombok (Susilawati et al., 2018).

Iron sand has the main components of iron oxide (Fe_2O_3) and silica (SiO_2) (Rahmi et al., 2019) and contains magnetic minerals such as magnetite (Fe_3O_4) and hematite (a-Fe_2O_3) (Fatari et al., 2022). Applicatively, natural iron sand can be used as Radar Absorbing Material (RAM) (Fatimah et al., 2022). RAM is a microwave absorbing material. The waves on the RADAR (Radio Detection and Ranging) tool use microwaves because they have a super high frequency. RAM is generally applied to the field of defense and state security. More specifically, RAM can be applied to anti-radar aircraft or submarines so that they cannot be detected by enemy radar (Susilawati et al., 2021).

The material being developed as a microwave absorber is Barium M-Hexaferite (BaM) (Susilawati et al., 2018). BaM is a complex oxide ceramic with the chemical formula $BaFe_{12}O_{19}$. BaM is included in ferromagnetic oxide materials with dielectric and magnetic properties which are widely used in radio frequency and microwave applications. Theoretically, BaM has characteristics that qualify as an absorber, namely having large crystalline anisotropy, high coercivity (6700 Oe = 0.67 Tesla), relatively high Currie temperature (450°C), relatively large magnetic saturation (Ms = 78 emu / gram), good chemical stability and resistance to corrosion (Simbolon et al., 2013), and has a very high melting temperature of 1390°C (Pullar et

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al., 2012). BaM has a very large coercivity field which causes the anisotropic properties of the material to increase. Therefore, the absorption properties become weaker, so it is necessary to reduce the value of the coercivity field. Decreasing the value of the coercivity field of this magnetic material results in a decrease in its magnetocrystalline anisotropy field (Ambarwanti et al., 2014).

It is necessary to do an engineering of BaM so that it meets the criteria as a microwave absorbing material or Radar Absorbing Material (RAM). One way to engineer this magnetic property is to add ions that have ionic radii almost the same as those of Fe metal. Some metals that can be used are Co (Khaerunnisa et al., 2018), Cu (Wardani et al., 2022), Zn (Halik et al., 2016), Ni (Munib et al., 2016), and Zn-Mn (Susilawati et al., 2017). Zn ions are ions that have almost the same ionic radius as Fe ions. Zn ions are diamagnetic and generally have an oxidation state of +2. The ionic radius of Zn^{2+} is 0.074 nm, while the ionic radius of Fe^{3+} is 0.065 nm, so it is possible that the presence of Zn^{2+} ions will replace Fe^{3+} (Silvia et al., 2013).

Synthesis of $BaFe_{12}O_{19}$ using Zn doping ($BaFe_{12-x}Zn_xO_{19}$) can be carried out using the coprecipitation method (Doyan et al., 2015). The coprecipitation method is a method for wet chemical synthesis that is simple and inexpensive in operational costs. This method has advantages such as high purity, simple deposition process making it easier to separate, relatively fast time required, and can produce very small grain-sized particles or nanoparticles (Rahmawati et al., 2013).

Based on the description above, the researcher intends to synthesize BaM using the coprecipitation method with Zn doping based on natural iron sand at the Tebing Beach of North Lombok as a microwave absorbent material.

Method

Synthesis of BaM with Zn doping ($BaFe_{12-x}Zn_xO_{19}$) using coprecipitation method based on natural iron sand at the Tebing Beach of North Lombok as a microwave absorbent material has been successfully carried out. The materials used in this study were magnetite (Fe_3O_4) obtained from iron sand, $BaCO_3$ as a holder, Zn as a doping, HCl as a solvent, distilled water (H_2O) as a diluent and NH_4OH as a precipitant (Susilawati et al., 2022). BaM synthesis was carried out using 3 variations of doping concentration (x), namely x = 0.000; 0.400; and 0.800. In addition, variations in the calcination temperature were also carried out, namely 400°C and 1100°C. After the calcination stage, the sample is ground to obtain smaller or nano-sized particles (Susilawati et al., 2020). The series of stages of the synthesis of BaM coprecipitation method can be seen in Figure 1.

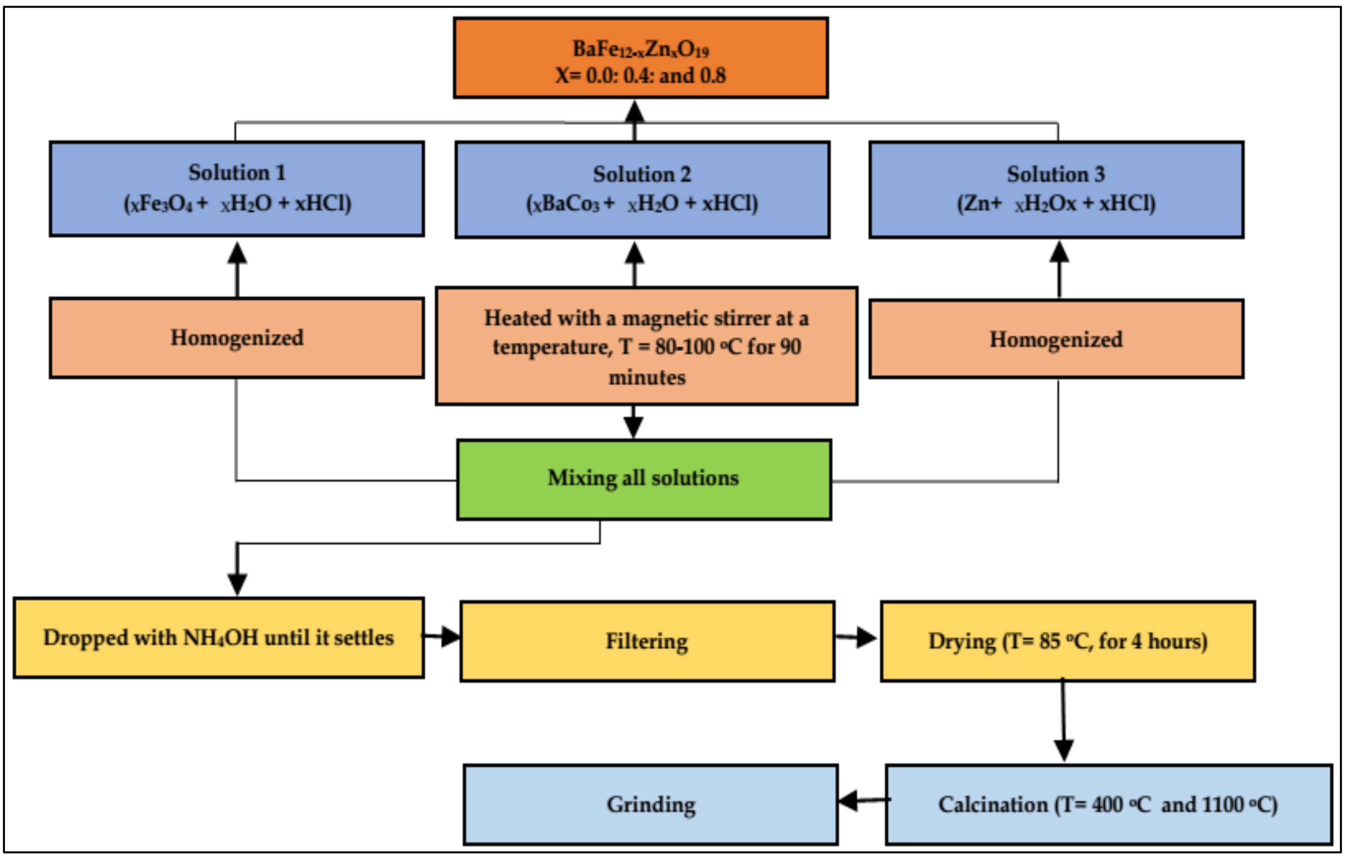

Figure 1. The series of stages of the synthesis of $BaFe_{12-x}Zn_xO_{19}$ with coprecipitation method

Result and Discussion

Natural iron sand as the basic material for the synthesis of $BaFe_{12-x}Zn_xO_{19}$ comes from the sand of Tebing Beach in North Lombok Regency. Based on the results of an initial study conducted by researchers on several beaches in North Lombok, it was found that Tebing Beach with the blackest sand color indicates the high magnetic content.

Figure 2. Natural iron sand Tebing Beach, North Lombok

Table 1. Elements/Compounds Needed in the Synthesis of $BaFe_{12-x}Zn_xO_{19}$

Nilai x	Fe_3O_4 (gram)	HCl (ml)	$BaCO_3$ (gram)	HCl (ml)	Zn (gram)	HCl (ml)	NH_4OH (ml)
0.000	13.320	38.153	2.839	2.384	0.000	0.000	70.000
0.400	12.810	36.707	2.829	2.375	0.375	0.950	90.000
0.800	12.350	35.370	2.819	2.367	0.747	1.895	110.000

The synthesis of $BaFe_{12-x}Zn_xO_{19}$ was carried out in several steps, namely first calculating the mass and volume of the material. Mathematically it can be calculated the mass and volume of material needed to make solution 1, solution 2 and solution 3 at each doping concentration as shown in Table 1. Then, the results of these calculations will become a reference in the stages of the synthesis of $BaFe_{12-x}Zn_xO_{19}$.

The next step is to make solution 1 by dissolving Magnetite (Fe_3O_4) and HCl using a hot plate, and stirring using a magnetic stirrer at 80–100^{o}C for 1 hour (Susilawati et al., 2021). Solution 2 was made by dissolving $BaCO_3$ and HCl using a hot plate, and stirring using a magnetic stirrer at 80–100^{o}C for 90 minutes. Solution 3 was prepared by dissolving Zn using a hot plate, and stirring using a magnetic stirrer at 80–100^{o}C for 30 minutes. After that, solution 1, solution 2 and solution 3 were mixed into a beaker container and stirred using a magnetic stirrer on a hot plate until a homogeneous solution was formed. A homogeneous solution is a solution that has been thoroughly mixed and has become a single unit. The composition of a homogeneous solution is so uniform that different parts cannot be observed, even with an optical microscope (Suparwati, 2017).

The homogeneous sample was then dripped with NH_4OH little by little to obtain a precipitate with high homogeneity and a weak acid solution pH. The sample is then cooled and filtered using filter paper and diluted with distilled water until the sample pH is neutral and the filtered water is clear.

The filtered sample that settles is then dried using an oven at 85^{o}C for 4 hours and then crushed to obtain a finer powder. These steps were then repeated for each different doping concentration. This is intended to determine the effect of doping concentration on the absorption properties of microwaves

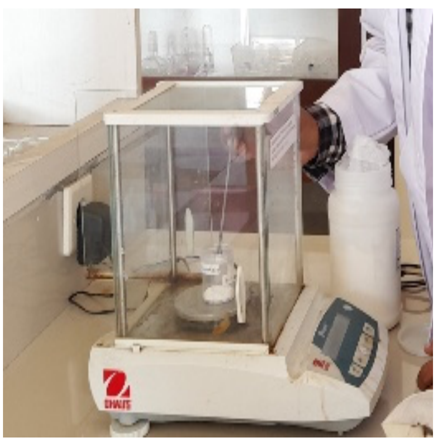

Figure 3. The process of weighing the materials needed

Figure 4. The reaction process for solutions 1 and 2 and dripping with NH_4OH

Figure 5. Sample filtering and waiting for the neutralization of the sample until the pH is neutral

Figure 6. Sample baking process

Figure 7. Sample calcination process at $400^{o}C$

Figure 8. Sample calcination process at $1100^{o}C$

Table 2. Calcination Results for Each Variant of Doping Concentration and Temperature

Calcination Temperature (^{o}C)	Value of x		
	0.000	0.400	0.800
400			
1100			

Finally, the samples were calcined using a furnace with a temperature variation of 400°C and 1100°C, then crushed until smooth. Calcination is the process of heating the sample at high temperatures to remove the volatile content in the sample (Susilawati et al., 2019). Calcination also affects the size of the sample particles which are expected to have nano or nanoparticle sizes (Susilawati et al., 2018). The different temperature variations are intended to determine the effect of calcination temperature on the absorption properties of microwaves (Susilawati et al., 2020). The results of the synthesis of Barium M-Hexaferrite using the coprecipitation method show different colors for each variation of calcination temperature (Khaerunnisa et al., 2018). Dark brick red for a temperature of 400°C and black for a temperature of 1100°C. This shows that the higher the calcination temperature, the darker the color of the sample (Susilawati et al., 2020). This is because the solvent used has evaporated due to the calcination process (Doyan et al., 2020).

Conclusion

Synthesis of $BaFe_{12-x}Zn_xO_{19}$ based on Tebing Beach iron sand has been successfully synthesized using the coprecipitation method. The results showed that the sample has a brick red color for a temperature of 400°C and black for a temperature of 1100°C. This shows that the higher the calcination temperature, the darker the color of the sample.

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