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Synthesis $BaFe_{12}O_{19}$ based on natural iron sand with Cu and Ni doping using coprecipitation method

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Abstract. Synthesis of $BaFe_{12}O_{19}$ based on natural iron sand with Cu and Ni doping using the coprecipitation method has been done. This research was conductedat the Chemistry Laboratory, Faculty of Teacher Training and Education, Mataram University. The synthesis was carried out through the variation of doping ions and calcination temperature. The concentration of doping ion used is x = 0.0; 0.4; 0.8; and 1.0 with variations in calcination temperature of 200, 600, and 1000 ^{0}C. The materials used are $BaCO_3$, Fe_3O_4, $CuSO_4$, and $NiCl_2.6H_2O$ powder. This synthesis process aims to obtain $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder that will later be used as a microwave absorbent material. To obtain sample powder of $BaFe_{12-2x}Cu_xNi_xO_{19}$, thisresearch is carried out through three stages: natural sand separation, magnetic minerals separation, and synthesisand calcination. The separation is to attach magnetic materials from the sand using a permanent magnet. After that, the magnetic mineral is detached from the separated sand to obtain Fe_3O_4 powder, which will later be used for synthesis. The results of synthesis and calcination showed that the more doping ions applied, the more concentrated the resulting powder. Similarly, according to the temperature variation, the higher the temperature, the more black the sample powder is.

1. Introduction

Electromagnetic wave (EM)-based technology devices are currently widely used in various fields, including military defense. The application of EM waves in this field is Radio Detection and Ranging (Radar) [1]. In military defense, there is a technology used to make ships or aircraft undetectable by radar, namely stealth technology. Stealth technology has two methods that are the design method and the coating method. In the device's design, the aircraft is modified in an angular shape. This modification aims to engineer the reflected wave (scattering) not to lead to the radar detector so that the plane cannot be detected [2]. However, this method requires an enormous cost. Therefore, another efficient alternative technique to develop is the coating method [3].

The coating method with Radar Absorbing Material (RAM) can reduce reflections and absorb microwaves (absorber) so that the material coated with RAM is not detected by radar [4]. The material used as an absorber must have high permeability (μ), high permittivity (ε), low coercivity (Hc) [5], and high magnetic saturation (Ms) [6]. Generally, the material used as an absorber is Barium M-Hexaferrite (BaM) [7]. BaM is known as a permanent magnet that has hardmagnetic properties [8]. Because BaM is hardmagnetic, this will cause the absorption of BaM to be weak, making it less practical to implement as an absorber. However, the material used as an absorber must be soft-

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magnetic [9]. Therefore, for this reason, doping with other metal cations with ionic radii quite similar to Fe, such as Co, Cu, Zn, Ni, and Mn, is required. [10]. The doping used in this research is Copper (Cu) and Nickel (Ni) because they have almost the same ionic radius as iron (Fe). In addition, based on their electron configurations, Cu and Ni have 1 and 2 unpaired electrons, which means that the doped metal has fewer electrons than Fe metal. Fe metal has four unpaired electrons, so it will be doped using a metal with fewer unpaired electrons. As it is known that the more unpaired electrons a metal has, the stronger its magnetism.

The synthesis of $BaFe_{12}O_{19}$ using Cu and Ni doping unveiled by the formula $BaFe_{12-2x}Cu_xNi_xO_{19}$ can be carried out using several methods: the coprecipitation method [11], the sol-gel method [12], the mechanical alloying method [13], and the mechanical milling method [14]. $BaFe_{12-2x}Cu_xNi_xO_{19}$ was synthesized using natural iron sand from the Ijobalit Beach, Labuhan Haji District, East Lombok. Barium Hexaferrite is classified according to its structure into six types, namely $BaFe_{12}O_{19}$ (type M), $BaMe_2Fe_{16}O_{27}$ (type W), $Ba_2Me_2Fe_{28}O_{46}$ (type X), $Ba_2Me_2Fe_{12}O_{22}$ (type Y), $Ba_3Me_2Fe_{24}O_{41}$ (type Z) and $Ba_4Me_2Fe_{36}O_{60}$ (type U) where Me is a divalent ion from the first transition series [15]. Type W-, X-, Y-, Z- is a type that is less economical because the process is relatively intricated. Thus, type M is preferable to use because it has uniaxial magnetic properties at room temperature and has an anisotropic field of 17.5 KOe. The synthesis in this study was carried out to produce $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder, which would then be calcined to reduce the moisture content of the sample powder.

2. Methods

The method used in this research is a laboratory experiment. This experiment is conducted at the Chemistry Laboratory, Faculty of Teacher Training and Education, the University of Mataram. The fundamental ingredients are $BaCO_3$, Fe_3O_4 taken from natural sand from Ijobalit beach, East Lombok, $CuSO_4$, $NiCl_2.6H_2O$, 37% HCl, NH_4OH, and H_2O. In the forming of barium ferrite, $BaFe_{12-2x}Cu_xNi_xO_{19}$ was carried out using four mole fractionvariations i.e. x = 0.0; 0.4; 0.8; and 1.0.

The process to obtain $BaFe_{12-2x}Cu_xNi_xO_{19}$ sample powder is through three stages, the first stage is to separate natural sand, then the second stage is to separate magnetic minerals, and the third stage is synthesis. The separation is done by attaching magnetic materials from natural iron sand using apermanent magnet, as shown in Figure 1. After that, separation and purification are carried out to obtain Fe_3O_4 powder. Afterward, Fe_3O_4 powder will be synthesized using the coprecipitation method by settling more than one substance together through the steps shown in Figure 2 [16]. The following process is calcination, as shown in Figure 3.

(a) (b) (c)

Figure 1. Natural sand separation stage. (a) Natural sand; (b) Separation using permanent magnet; (c) Magnetic minerals

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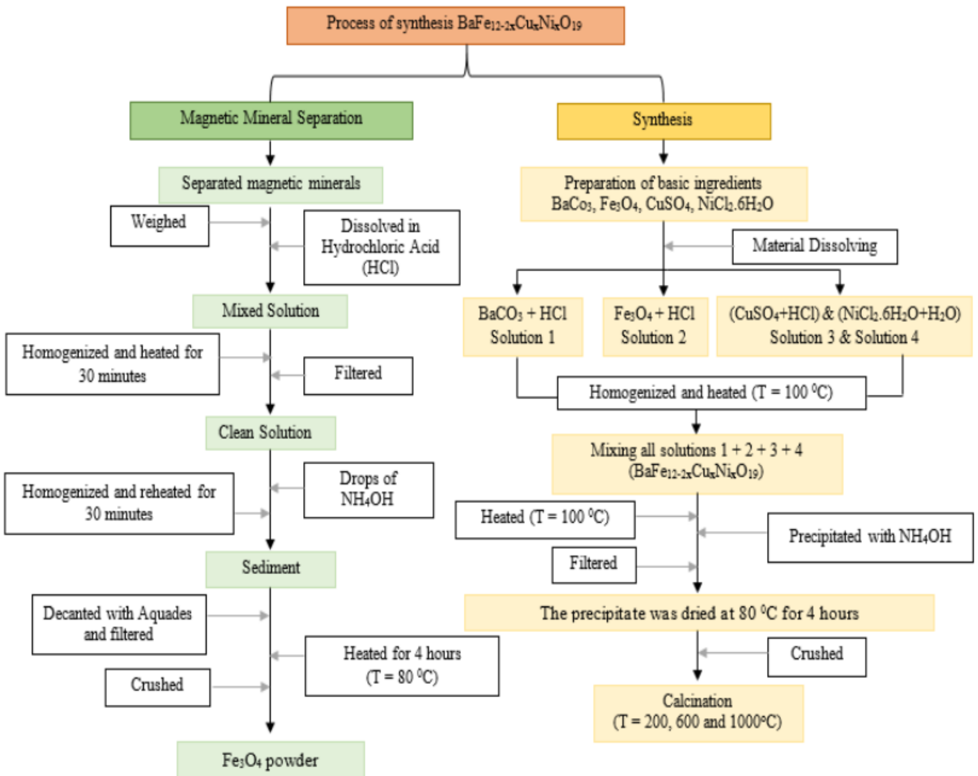


Figure 2. The process of synthesis $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder

(a) (b) (c)

Figure 3. Calcination stage. (a)put the sample into the kiln; (b) heated at various temperatures; (c) calcination results

3. Result and Discussion

The synthesis of Barium M-Hexaferrite begins with the chemical reaction equalization of the materials used to adjust the composition required in this process. The reaction equation is calculated based on the amount of doping ion concentration used, namely x = 0.0; 0.4; 0.8 and 1.0.

The reaction equation for the compound without doping

Equation A

$$BaCO_3 + 2HCl \rightarrow BaCl_2 + H_2CO_3$$

Equation B

$$4Fe_3O_4 + 32HCl \rightarrow 4FeCl_2 + 8FeCl_3 + 16H_2O$$

Equation A + B

$$BaCl_2 + H_2CO_3 + 4FeCl_2 + 8FeCl_3 + 16H_2O + 34NH_4OH \rightarrow BaFe_{12}O_{19} + 34NH_4Cl + 32H_2O + 2H_2 + CO_2$$

The reaction equation for the compound with doping x = 0.4 ($BaFe_{11.2}Cu_{0.4}Ni_{0.4}O_{19}$)

Equation A

$$BaCO_3 + 2HCl \rightarrow BaCl_2 + H_2CO_3$$

Equation B

$$3.733Fe_3O_4 + 29.866HCl \rightarrow 3.733FeCl_2 + 7.467FeCl_3 + 14.933H_2O$$

Equation C

$$0.4CuSO_4 + 0.8HCl \rightarrow 0.4CuCl_2 + 0.4H_2SO_4$$

Equation D

$$0.4NiCl_2.6H_2O + H_2O \rightarrow 0.4NiCl_2 + 7H_2O$$

Equation A+B+C+D

$$BaCl_2 + H_2CO_3 + 3.733FeCl_2 + 7.467FeCl_3 + 14.933H_2O + 0.4CuCl_2 + 0.4H_2SO_4 + 0.4NiCl_2 + 7H_2O + 33.4NH_4OH \rightarrow BaFe_{11.2}Cu_{0.4}Ni_{0.4}O_{19} + 33.4NH_4Cl + 40.03H_2O + CO_2$$

Based on the equation above, it can be seen that the required compound is accordingto the value of Mr (relative atomic mass), M (Molarity), and n (molality) of each compound used. The following table shows the results of the compound calculation needed in the synthesis process.

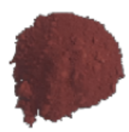
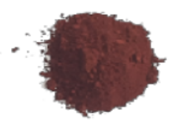
Table 1. Calculation results of compounds for synthesis

No	Compound	X	Fe_3O_4	$BaCO_3$	$CuSO_4$	$NiCl_2.6H_2O$
1	$BaFe_{12}O_{19}$	0	20,8354	4,43448		
2	$BaFe_{11,2}Cu_{0,4}Ni_{0,4}O_{19}$	0,4	19,3724	4,41800	1,42934	2,12858
3	$BaFe_{10,4}Cu_{0,8}Ni_{0,8}O_{19}$	0,8	17,9202	4,40165	2,84810	4,24141
4	$BaFe_{10}Cu_{1,0}Ni_{1,0}O_{19}$	1,0	17,2007	4,39351	3,55355	5,29196

After gaining the required compound composition, the next step is to carry out the synthesis process according to the steps shown in Figure 2.

The powder produced from the synthesis process will then be calcined at different temperatures: 200, 600, and 1000 ^{0}C. Calcination aims to reduce the water content in the sample and shrink the pores during the calcination process. The results of the calcination of the $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder obtained underwent a color change, as shown in Table 2.

Table 2. Effect of doping ion variation and temperature variation of $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder calcination

$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.0 (T = 200^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.4 (T = 200 ^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.8 (T = 200 ^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 1.0 (T = 200^{0}C)
			
$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.0 (T = 600^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.4 (T = 600^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.8 (T = 600 ^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 1.0 (T = 600^{0}C)
			
$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.0 (T = 1000 ^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.4 (T = 1000^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 0.8 (T = 1000^{0}C)	$BaFe_{12-2x}Cu_xNi_xO_{19}$ X = 1.0 (T = 1000 ^{0}C)
			

As elaboratedin Table 2, it can be seen that the color of the sample powder produced varies with the increase in the calcination temperature and the addition of doping use. The more doping ions mixed, the more concentrated the color of the sample powder. At 200 ^{0}C, the sample powder appears to produce a similar color because at 100-300 ^{0}C only H_2O is released, and the sample will be bound to O-H [17]. Next, at 600 ^{0}C, the sample has been seen a color difference from brick red to dark brown. This occurrence happens because, at 600 ^{0}C, there is a release of CO_2 gas and a significant reduction in mass weight. Finally,at the last temperature of 1000 ^{0}C, a color change from brown to black indicated that a crystal structure had formed. Dark brown to black powder demonstrates that all the H_2O and HCl content of 100% has been used up [18]. This reveals that in the heating process, the basic ingredients are completely dissolved [19].

The calcination process on the color change of $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder indicates crystallization, influenced by temperature in the calcination process. The $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder will form

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hexagonal crystals at a temperature of at least 600 ^{0}C. Based on this research, it was found that the color change of the $BaFe_{12-2x}Cu_xNi_xO_{19}$ powder will be darker as the calcination temperature increases and the doping ion is used.

4. Conclusion
Based on the synthesis of $BaFe_{12-2x}Cu_xNi_xO_{19}$ using the coprecipitation method with variations in doping ions and calcination temperature. The more doping ions used, the more concentrated the resulting powder. Likewise, with the temperature variations used, the higher the temperature,the blacker the sample powder is. The color of the sample indicates that the sample has formed crystals or not.

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