Influence of Micro Structure Based Magnetic Material BaNi\textsubscript{X}Al\textsubscript{6-X}Fe\textsubscript{6}O\textsubscript{19} as Material Absorber Wave Magnetic Structure

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Abstract. Material have researched electromagnetic wave absorber based barium hexaferrite BaNi\textsubscript{X}Al\textsubscript{6-X}Fe\textsubscript{6}O\textsubscript{19} (x = 0; 0.5; 1; 2; 3) of the local raw materials sand iron Sukabumi. This material is synthesized by coprecipitation method and mixing it using solid state reaction. Material in milling for 5 hours, and heated (sintering) at a temperature of 100 °C for 5 hours. The sample is then characterized by XRD, homogeneous morphological structure and the most optimum composition seen through SEM / EDS. However, the optimum variation obtained at x = 1 obtained the absorption ability of electromagnetic wave equal to -36 dB. This means the material can absorb up to 95% with the absorption s field thickness of 2 mm. Where the average distance between the crystal atoms is 0.217 nm.

1. Introduction
BaO oxide magnetic material (6Fe\textsubscript{2}O\textsubscript{3}) is a type of ceramic that are often found in addition to other magnetic material, such as SrO.6 (Fe\textsubscript{2}O\textsubscript{3}) and PbO.6 (Fe\textsubscript{2}O\textsubscript{3}). Development of BaFe\textsubscript{12}O\textsubscript{19} (M-type feritte hexagonal) material as magnetic material is needed in various application fields, because it has characteristics: relatively high Curie temperature, coercive value, magnetic saturation and high magnetic anisotropy and excellent chemical stability and cheap as well as easily obtainable. One of the applications of the permanent magnetic barium hexaferrite material of concern today is the microwave absorber (RAM). This is because the electrical and magnetic properties of this ferrimagnetic material are very supportive in the application, which has high permeability and resistivity [1].

Radar (Radar Detection and Ranging) is an object detection system that uses electromagnetic waves to identify the distance (range), the direction (direction) or speed (speed), both moving and stationary objects such as aircraft, ships vehicles and weather conditions. The radar system consists of transmitters and receivers located in the same location or can be separated. Transmitter will emitte radio wave at a certain frequency and power. When the energy of the radio wave emission of the object will be reflected in all directions (scattered). Partially reflected back (reflected back) to the receiver and having little change in wavelength (wavelength) frequency even if the target moves. The energy of the return signal is usually very weak so it needs to be amplified using the electronics technique in the receiver and configured antenna [2].
In the field of defense, microwave absorption is used for coatings or coatings on fighter (stealth aircraft), warships or submarines (warship), and for the armor, especially the front vanguard. Radar aircraft so far have been suspected to have an impact on humans located around radar installations. The impact is that radar agitates the water molecules present in the human body. If the intensity of electromagnetic radiation is strong enough, then the water molecules are ionized, the impact is similar to the impact of nuclear radiation [3]. To overcome the impact of the development of information technology is required a material capable of absorbing electromagnetic wave radiation to minimize or reduce the hazards posed by radiation wave electromagnetic.

Sand is a very abundant natural material in Indonesia. Sand commonly used for building materials as a mixture of cement in the manufacture of walls as brick linings. Iron sand in general has the main composition of iron oxide (Fe₂O₃ and Fe₃O₄) [4]. In a study conducted by Wishnu Ari Adi in 2010 that made barium hexaferrite with substitution of Mn and Ti showed that dribbling was successful by minimizing Hc (coercivity field) but the resulting saturation magnetization (Ms) was also low [5]. So in this study with the same system with substitution of Al₂O₃ and NiO. Iron sand used in this study comes from iron sand area Sukabumi. On early stage of this study using coprecipitation method (precipitation) to get Fe₃O₄, coprecipitation method is one of the methods of synthesis of inorganic compounds based on the process of precipitation. The next process uses solid state reaction (solid state reaction). In this research, a wave absorbing material of iron oxide (Fe₂O₃) mixture of local iron sand material was added with BaCO₃, Al₂O₃, NiO.

The oxidation results have a smaller magnetic susceptibility when compared to the original magnetite mineral. Due to the higher oxidation temperature [6]. Ferries can be applied primarily to technologies such as high frequency electromagnetic waves ranging as Radar. But Wave absorption requires Fe cation substitution with fixed ratio. At a higher substitution level the uniaxial anisotropy changes to planar magnetocrystalline [7].

Magnetite and maghemite have cube phases while hematite has a hexagonal phase. The maghemite and hematite phases were obtained by oxidation at different sintering temperatures. Maghemite become hematite phase transition has occurred at 550°C. When the heating temperature of 250°C and increasing to a temperature of 350°C whereas in these circumstances, maghemite dominate a sample while phase at a temperature of 550 °C, it has emerged that hematite Fe₂O₃ phases [8].

Absorption of electromagnetic waves is a form of energy that can be emitted or absorbed by charged particles, which exhibit wave-like direction as it travels through space. The electromagnetic waves can be absorbed by the magnetic absorber. Electromagnetic waves are charged from couples (pairs) of electric fields and magnetic fields perpendicular to each other. The type of electromagnetic wave absorption is divided into 2 (two) namely engineering materials and engineering geometry (Form). Material engineering is when making a material by adding some elements of the structure remains. Barium hexaferrite magnetic material has advantages such as high coercivity (Hc) and magnetic saturation (Ms), high transition temperature (Tc = curie temperature) and stable and corrosion-resistant chemical properties [9].

While engineering geometry of its manufacture should pay attention to particle shape, thickness of surface morphology, electric field and magnetic field. Electromagnetic wave absorption technology has given rise to a new material called Radar Absorber Material (RAM). This material is dampening the reflection or microwave absorber, so that objects coated with RAM was not detected by radio detection and ranging (RADAR). The absorber material is affected by the impedance matching of the material with the electromagnetic wave through the resonance frequency mechanism. To obtain a single phase of ferrite-based magnetic material is not easy to do. The synthesis of barium hexaferrite can produce impurity phases, namely: hematite (Fe₂O₃) and monoferrite (BaFe₂O₄) [10].

Synthesized iron rock is used as a filler material in a microwave absorbent composite material. The iron strands are synthesized into magnetic nanoparticles, such as Fe₂O₃. The oxidized iron has a very high permeability [11]. According to Alvin Lie, an aviator observer, the impact of aircraft disturbances is actually very small. Note that only one phone is active. Due to the electromagnetic waves emitted from one cell phone entering the micro scale. Alvin concludes that enough impact for aviation safety
has the potential to interfere with communication and navigation [12]. Microwave absorption occurs due to wave interaction with materials that produce a Reflection loss energy effect which is generally dissipated in the form of heat. Microwaves are divided into several internationally defined coverage areas. According to the table below.

Table 1. Distribution of Micro current Reach Areas [13]

| Band | Frequency Range (GHz) |
|------|-----------------------|
| L    | 1.22-1.70             |
| R    | 1.70 - 2.60           |
| S    | 2.60-3.95             |
| H    | 3.95-5.85             |
| C    | 5.85-8.20             |
| X    | 8.20-12.4             |
| My   | 12.4-18.0             |
| K    | 18.0-26.5             |
| Ka   | 26.5-40.0             |
| U    | 40.0-60.0             |
| E    | 60.0-90.0             |
| F    | 90.0-140.0            |
| G    | 140.0-220.0           |

Characteristics of a good absorber material is to have both magnetic and electrical well. The material must have a certain impedance value that the relative permeability value ($\mu_r$) and its relative permittivity ($\varepsilon_r$) correspond to the value of $\mu$ and $\varepsilon$ air or vacuum in order to occur impedance resonance, the value of the reflection loss generated by the material is quite large [14]. The mechanism of electromagnetic wave absorption of the material in general was influenced by two factors namely the thickness factor occurs in all the material and the thicker the material absorber also greater and the absorption of electromagnetic radiation on magnetic material beside because thickness factor also happened other interaction that is electromagnetic wave from outside will rotate magnetic dipole so happened material impedance [15]. *Barium hexaferrite* which has lossy material properties, has dielectric loss factor and high magnetic loss so that the material has good properties for absorption of electromagnetic waves [1].

BaFe$_{12}$O$_{19}$ is a type M-type hexaferrite type M. This is also called magnetoplumbite. Ion Fe is tetrahedral (FeO$_4$) in a trigonally bipiramide (FeO$_5$) octahedral with spin parallel orientation to Fe in 4f plane. The value of space group $p$ 63 / mmc with lattice parameter is $a = 0.58836$ nm where $a = b$ and $c = 2.306$ nm at room temperature. While Crystal density through measurement with X-Ray obtained $5.33$ g / cm$^3$ [5].

2. Method

2.1 $Fe_3O_4$ precipitation process

5M NaOH solution per 1000 ml using hot plate and placed magnetic stirrer. After NaOH dissolves then cooled. The iron sand is heated over the hot plate while continuously stirring with a magnetic stirrer until the temperature is 70°C measured with the thermometer and slowly adding the NaOH solution using the peristaltic pump. After all the NaOH solution and air mixed black, measured and obtained his PH PH 12. Solutions is cooled and added right with distilled water until glass beaker filled. L the landing is allowed to settle and be held close to a permanent magnet to separate the iron sand with the aquades. Aquades is removed. Repeat this leaching process until PH 7 using PH indicator. After PH reaches 7, Fe$_3$O$_4$ is still wet inserted into the mammert oven at 110°C for 10 hours until Fe$_3$O$_4$ is completely dry and dark brown. Then the scour using mortal and the result is weighed.
D load into the furnace with a temperature of 750°C for 5 hours. And formed Fe$_2$O$_3$ from iron sand marked with brown color. Once in the furnace is carried out again to smooth grinding powder.

Fe$_3$O$_4$ annealed in the furnace with a temperature of 750°C for approximately 5 hours. It is intended to convert Fe$_3$O$_4$ contained in iron sand to α-Fe$_3$O$_4$. Then the grinding process for Fe$_3$O$_4$ is a fine powder. The mixing process is done by mixing the necessary ingredients weighed in accordance with the stoichiometric phase BaNi$_x$Al$_6$-$x$Fe$_6$O$_{19}$ at ($x = 0.5; 1; 2; 3$) mixing of material is done by using Vial HEM. The mixing is supplemented with ethanol until all mixtures are submerged.

The addition of ethanol is meant that the sample to be dimilled is not too attached to the HEM vial when it is removed. Each powder of the base material with a certain composition was mixed through solid mixing using mechanical milling for 5 hours at a rate of 1000 rpm. With running tool for 60 minutes continued rest tool for 30 minutes.

Due to the addition of ethanol from the mixture of wet material mixture, to dry, the mixture is heated with temperature 110°C for 5 hours. After that is done scouring to form a fine powder. Then put into reversible for sintering process. This is useful for bonding the mass of particles on the powder by interactions between molecules or atoms through heat treatment with sintering temperatures approaching the melting point resulting in solidification. Where sintering temperature 1000°C for 4 hours. After cooling the sample is then mashed to a characteristic in powder form.

3. Results and Discussion

3.1 Results of XRD Characterization on Samples BaNi$_x$Al$_6$-$x$Fe$_6$O$_{19}$

The hexagonal structure of ferrite (type M) or often referred to as the hexaferrite material structure. The amount of comparison between BaO and Fe$_2$O$_3$ of this conventional type M has a ratio of 1: 6 with an empirical compound BaO.6Fe$_2$O$_3$ or BaFe$_{12}$O$_{19}$. This hexaferrite (type M) also has an anisotropy field (Ha) and very high crystalline anisotropic constants. So it has the potential to be used as a permanent magnet by generating maximum product energy. Besides, this type of material can be modified by engineering its structure in such a way that it can be used as an absorption of electromagnetic waves. 1 is shown in Figure 4. The measurement results of X-ray diffraction pattern of the sample BaNi$_x$Al$_6$-$x$Fe$_6$O$_{19}$.

![Figure 1. Results of XRD Characterization Materials BaNi$_x$Al$_6$-$x$Fe$_6$O$_{19}$](image-url)
The result of phase identification shows that the sample $\text{BaNi}_x\text{Al}_{6-x}\text{Fe}_6\text{O}_{19}$ ($x = 1$) or $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$ has a crystal structure with one phase barium hexaferrite. Then the sample ($x = 2; 3$) consists of multiphase (more than one phase). This means that structural engineering of barium hexaferrite material after modification by substitution of nickel (Ni) into aluminum structure (Al) has been successfully performed with nickel content of no more than one nickel atom (Ni).

### 3.2 Results of VNA Characterization on Samples $\text{BaNi}_x\text{Al}_{6-x}\text{Fe}_6\text{O}_{19}$

The coming electromagnetic waves comprise the components of the magnetic field and the electric field interacting with the magnetic material. The interaction between materials with electromagnetic waves is schematically shown in Figure 2. In the case of EM energy absorption, the overall interaction can be represented by the impedance of dielectric and inductive material ($Z_m$).

![Figure 2. Schematic of Absorption Process of Electromagnetic Waves](image)

_Bandwidth_ absorption of 10 dB means the _bandwidth_ frequency can reach 60% of the _reflection loss_, if the absorption _bandwidth_ is 20 dB means the frequency _bandwidth_ can reach 90% _reflection loss_. Figure 3 shows the _reflection loss_ measurement as a frequency function of 8-12 GHz. _This reflection loss_ indicates the existence of magnetic spin resonance mechanism between electromagnetic wave with material so that ultimately can happen absorption of electromagnetic wave (EM).

![Figure 3. RL curve in the sample $\text{BaNi}_x\text{Al}_{6-x}\text{Fe}_6\text{O}_{19}$](image)

Figure 3 shows that the absorption of EM waves in the sample increases after the substitution of nickel $x = 1$, but after $x > 1$ the absorption of EM waves appears to decrease. Thus the optimum composition of $\text{BaNi}_x\text{Al}_{6-x}\text{Fe}_6\text{O}_{19}$ system which wave absorption can be used as absorption of EM wave at frequency of 11.24 GHz is $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$ ($x = 1$).
3.3 Results of Optimum Composition of $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$

The XRD analysis shows that the sample has a single phase with Hexagonal sketch structure with P63/mmc space group with lattice parameter is $a = 0.58836$ nm and $c = 2.30376$ nm at room temperature. The XRD analysis results are supported by the presence of Al and Ni atoms in the sample by morphologic and elementary characterization using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). In Figure 5 shows the result of microstructure observation on sample $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$.

![XRD spectrum](image)

**Figure 4.** Identifying the Sample Phase $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$

![SEM images](image)

**Figure 5.** Photographs of Morphological Sample Particle Samples $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$

Based on the observation of particle morphology, the sample of $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$ looks homogeneous and evenly distributed throughout the sample surface which means that the sample $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$ has a single phase with the composition as presented in Table 2

| No. | Element | Chemistry | % Weight | % Atom |
|-----|---------|-----------|----------|--------|
| 1.  | Carbon  | C         | 9.47     | 18.73  |
| 2.  | Oxygen  | O         | 41.76    | 62.01  |
| 3.  | Sodium  | Na        | 0.34     | 0.54   |
| 4.  | Magnesium | Mg      | 0.55     | 0.54   |
| 5.  | Aluminum | Al       | 5.72     | 5.04   |
| 6.  | Silicon  | Si        | 0.26     | 0.22   |
| 7.  | Titanium | Ti        | 2.95     | 1.46   |
The elements contained in the sample $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$ have a stoichiometric suitability with the results of the sample preparation. So means that the sample phase has been well formed single phase with a composition that has been in accordance with the preparation. So that the distribution of the particles is evenly distributed. The impurity comes from of raw materials used are iron sand. results of electromagnetic wave absorption test on sample $\text{BaNiAl}_5\text{Fe}_6\text{O}_{19}$ indicated

![Absorption wave electromagnetic Samples BaNiAl$_5$Fe$_6$O$_{19}$](image)

**Figure 6.** Absorption wave electromagnetic Samples BaNiAl$_5$Fe$_6$O$_{19}$

The highest peak absorption frequency is at 11.24 GHz, by ~ 36 dB. On the highest peak frequency calculated magnitude absorption of EM wave reach 95% with thickness of absorption field 2 mm and distance between atom -atom 0.217 Å This can be seen in figure 6.

![Intensity vs Distance d (hkl)](image)

**Figure 7.** XRD result of sample BaNiAl$_5$Fe$_6$O$_{19}$

For samples BaNiAl$_5$Fe$_6$O$_{19}$ (Barium Hexaferrite) $x = 1$. At $x > 1$ have average distance between the atoms of 0.228 Å. The magnitude of Intensity can be seen in each figure below.
Figure 8. Results of XRD samples $\text{BaNi}_2\text{Al}_4\text{Fe}_6\text{O}_{19}$

Figure 9. XRD result of sample $\text{BaNi}_3\text{Al}_3\text{Fe}_6\text{O}_{19}$

Figure 10. Results XRD sample $\text{BaNi}_{0.5}\text{Al}_{5.5}\text{Fe}_6\text{O}_{19}$
Therefore $\text{BaNiAl}_6\text{Fe}_6\text{O}_{19}$. The sample may be a candidate sample of electromagnetic wave absorption.

4. Conclusion
1. Based on results testing using XRD (X-Ray Diffraction) shows has succeeded in substitution with Ni and Al into the Hexaferrite Barium having phase single with hexagonal structure up with variation ($x = 1$). When variations improved at ($x = 2; 3$) the phase already no again single phase caused already existence compound Ba O and mixed NiFe$_2$O$_4$. So too with SEM results of its structure already uniform on $x = 1$, with the atomic average distance in the sample = 0.228 Å.

2. Absorption EM waves on sample increased after to substitution nickel $x = 1$, however after $x > 1$ absorption EM wave appears increasingly decreased. So optimum composition of the system $\text{BaNi}_{x}\text{Al}_{6-x}\text{Fe}_6\text{O}_{19}$ absorption wave could used as ingredients absorption EM wave is $\text{BaNiAl}_6\text{Fe}_6\text{O}_{19}$ ($x = 1$).

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