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*by* Aris Doyan

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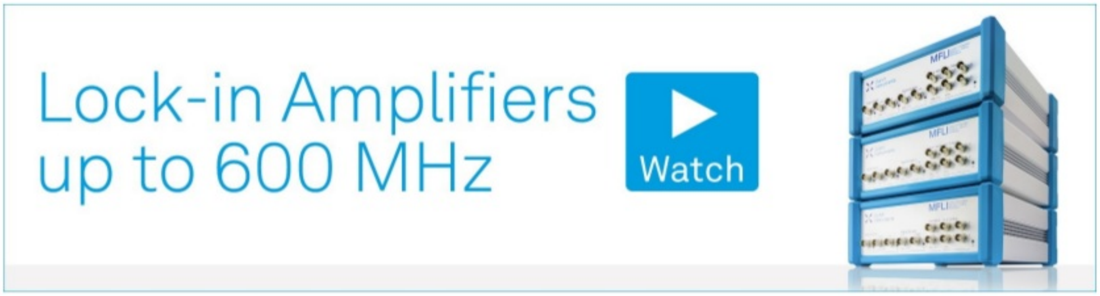

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# Synthesis and Characterization of Barium Hexaferrite with Manganese (Mn) Doping Material as Anti-Radar

Susilawati[a], Aris Doyan[b], Khalilurrahman[c]

*Department of Physics Education , University of Mataram, Lombok, West Nusa Tenggara, 83125 Indonesias.*

[a]Corresponding author: susilawatihambali@yahoo.co.id
[b]arisdoyan @yahoo.co.id
[c]khalil.ong06@gmail.com

**Abstract**. Have been successfully synthesized barium powder doping Manganese hexaferrite with the expected potential as anti-radar material. Synthesis was done by using the co- precipitation method, the variation of the variable x concentrations used were 0; 0.2; 0.4; and 0.6 and calcined at temperatures of 400, 600 and 800$^0$C. Characterization powders of hexaferrite have used XRD (X-Ray Diffraction), SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy), LCR (inductance, capacitance, and resistance) meter, and VSM (Vibrating Sample Magnetometer). The higher the concentration and temperature of calcinations given affect the color of the powder. The test results using XRD indicates that it has formed barium hexaferrite phase with a hexagonal crystal structure. Tests using SEM showed that all the constituent elements barium powder hexaferrite by doping Manganese powders have been spread evenly. XRD test results were confirmed by a test using a TEM showing the crystal structure and the powder was sized nano particles. The results from the LCR meter showed that the barium powder hexaferrite by doping Manganese that has been synthesized classified in semiconductor materials. The result from VSM showed that the value of coercivity magnetic powder doped barium hexaferrite Manganese is smaller when compared with barium hexaferrite without doping and belong to the soft magnetic. Based on the results of the synthesis and characterization, we can conclude that the barium powder heksaferrite by doping Manganese potential as a material anti-radar.

## INTRODUCTION

Radar absorbing materials (RAM) are classified into two i.e the material of a dielectric and the material of the magnetic. The microwave-absorbing materials can be used to minimize the electromagnetic reflection from the metal plate such as aircrafts, ships, tanks, and electronic equipment. Barium hexaferrite is large magneto crystalline anisotropy, high Curie temperature, relatively large magnetization, excellent chemical stability, and corrosion resistivity [1]. M-type barium hexaferrite with hexagonal molecular structure $BaFe_{12}O_{19}$ (Ba ferrite) is a promising material for permanent magnet, advanced recording, and microwave absorbing. The hexagonal barium hexaferrite ($BaFe_{12}O_{19}$) is known as a hard magnetic material with high coercivity and large saturation magnetization. It is used in high frequency microwave technology, magnetic recording media and as a permanent magnetic material [2]. Nano-sized ferrite particles can be obtained by various ways, such as, co-precipitation method, micro emulsion techniques and decomposition of organo-metallic compounds. Co-precipitation has a simple synthesis route and can be prepared on a large scale of products. It is one of the techniques used frequently for preparation of nano sized particles [3]. The co-precipitation allows one to vary the average size of nano particles by adjusting the pH and the temperature of the aqueous media, but one has only limited control over the particles size distribution [4]. The doped hexaferrites show that the saturation magnetization decreases slightly and coercivity decreases dramatically with increasing doping content. It is suggested that a combination of dopants can be used to control or to reduce the coercivities with only a small change of their saturation magnetizations. Thus, the substitutions of Fe ions by isovalent cations can be generally investigated BaM constituent element iron can be replaced by other metal cations from the group of transition which has almost the same size as Co, Zn, Ni and Mn [5]. This study concentrates

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synthesis of barium hexaferrite by doping Mn with the variable value x = 0; 0.2; 0.4; and 0.6 and calcinations at 400, 600, and 800$^0$C. Furthermore characterization of barium hexaferrite powder with doping to determine the changes that occur after being given additional element Mn dopants and calcinations temperature.

## METHOD

The basic ingredients of synthesis of barium hexaferrite with Mn doping among others such as $BaCO_3$, $FeCl_3.6H_2O$, and $MnCl_2.4H_2O$ as dopants. Doped barium hexaferrite powder Mn synthesized using co-precipitation method. Dopant concentration values varied from x = 0; 0.2; 0.4; 0.6; and 0.8. Stages of barium hexaferrite powder synthesis by doping Mn among others.

$FeCl_3.6H_2O$ Dissolves in $H_2O$ using a magnetic stirrer for 30 minutes (solution 1). $BaCO_3$ dissolving in HCl using a magnetic stirrer hot plate at a temperature of 70 °C (solution2). Dissolve the powder $MnCl_2.4H_2O$ in $H_2O$ (solution 3). Mixing a solution of 1, 2, 3 and distirrer for 30 minutes (solution 4). Titrating the fourth with $NH_4OH$ solution until precipitation occurs, then washed with distilled water until pH neutral. The Samples were dried at a temperature of 80 °C and then calcined at a temperature of 400, 600 and 800 °C for four hours [6]. Samples were assayed using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Inductance, Capacitance and Resistance meter (LCR Meter) and Vibrating Sample Magnetometer (VSM).

## RESULTS AND DISCUSSION

The result of the synthesis of barium hexaferrite powder doped Mn can be seen in Figure 1. below:

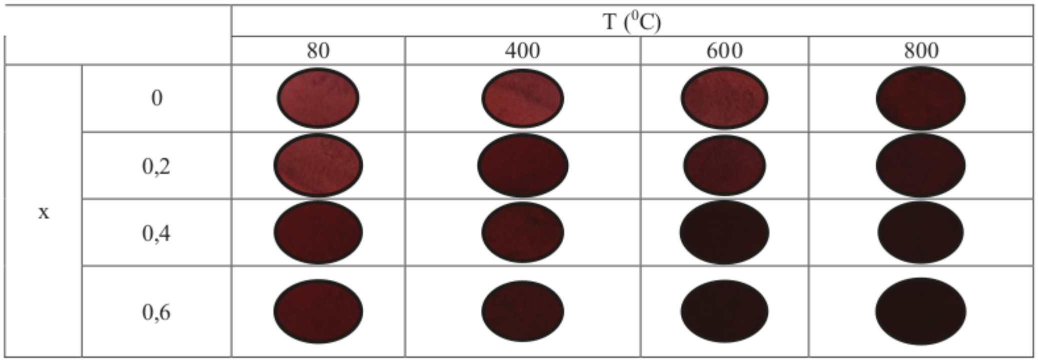


**FIGURE 1**. The powder barium hexaferrite with various concentrations of doping x and the calcination temperature T.

Without doping barium hexaferrite powder had a brown color which is brighter when compared with powder doped Mn. The addition of doping affects the color of the powder which more variations doping variable x is given, the color of the sample becomes more dark chocolate. Calcination temperature variations also affect the color of powdered barium hexaferrite. The higher the calcination temperature is given, the more brown color powder with increasing calcination temperature.

### X-Ray Diffraction

To characterize the crystalline of samples, XRD measurements were carried out on the samples treated under different reaction times and temperatures. Tests using XRD phase aims to determine the crystal structure formed of barium hexaferrite powder synthesized. Testing was conducted at a voltage of 40 kV and current of 30 mA using CuKa targets at a wavelength λ = 1541 A.

The test results using XRD can be seen in Figure 2. Based on the chart below, the peak phase of barium hexaferrite at an angle of diffraction $2\Theta$ on 28.4954°. Barium phase forming iron oxide and manganese 35.704° form of iron oxide phase. Furthermore, to test the structure of the crystal, the test results show that the lattice parameters a = b = 5.0371 Å and c = 13 749 Å, with an angle $\alpha = \beta = \gamma$ = 90o and 120°. These results indicate that it has formed a hexagonal crystal structure [7].

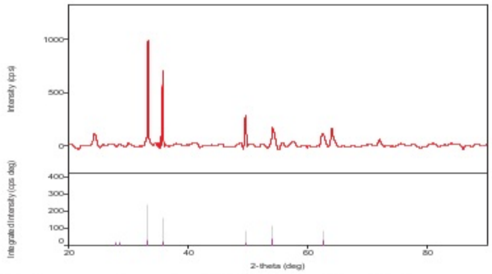
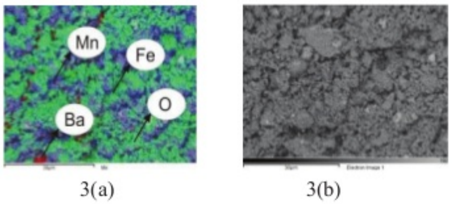


**FIGURE 2**. Graph of the relationship between the angle of diffraction (2θ) with the x-ray spectrum (intensity) is diffracted as barium hexaferrite powder test results with Mn doping using XRD

## SEM with EDX

Tests using a SEM aims to examine the morphology and the elements contained in the powder doped barium heksaferit Mn. The test results using the SEM can be seen in Figure 3 below:



3(a) 3(b)

**FIGURE 3** (a). barium hexaferrite powder with a magnification of 2,000 times, 3(b) barium hexaferrite powder color spectrum.

Constituent elements barium hexaferrite with doping Mn based on the color spectrum each can be seen in Figure 4. below:

4(A). Manganese (Mn)
0,61%

4(B). Iron (Fe)
55,57%

4(C). Barium (Ba)
5,77%

4(D) Oksigen (O)
38,05%

**FIGURE 4**. The elements making up the powder doped barium hexaferrite Mn based on the color spectrum.

Graph the percentage of constituent elements barium hexaferrite with Mn doping can be seen in Figure 5 below:

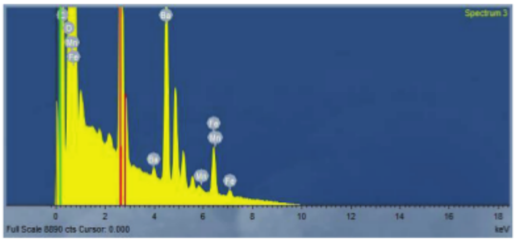


**FIGURE 5**. Graph of the elements contained in the powder doped barium hexaferrite with Mn

SEM imaging results can be seen in Figure 5. at 2000 times magnification. Based on the results of SEM, it is known that the elements of Ba in the powder amounted to 5.77%, Fe = 55.57%, Mn = 0.61%, O = 38.05%. Generally the presence of barium hexaferrite constituent elements by doping Mn powder was mixed uniformly. This can be seen from Figure 3 (B) showing the distribution of constituent elements barium hexaferrite based spectrum of different colors [8].

## Transmission Electron Microscopy (TEM)

The TEM images of the nanoparticles were taken to observe the morphology and the size of nanoparticles prepared under different reaction times. Tests using TEM is performed to determine the size and structure of the particles of the powder doped barium hexaferrite with Mn. The test results barium hexaferrite powders with doping Mn can be seen in Figure 6. below:

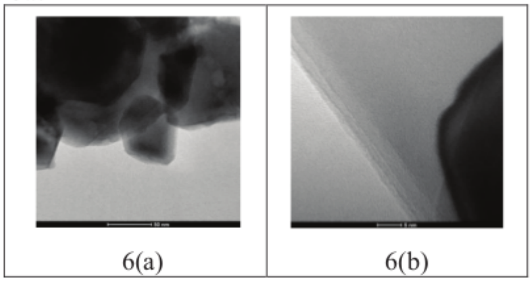

6(a)
6(b)

**FIGURE 6** (a). Shows the average particle size of 50 nm (scale in the figure represents 50 nm). 6(b) the orderly stripes on a particle that shows the crystal structure

The test results using the TEM in Figure 6(A) shows that the powder has been formed nano-sized particles with an average powder size of 50 nm (scale in the figure represents 50 nm) [10]. Next look for the diffraction pattern formed from particles showed that the particle shaped crystals. Based on Figure 6(B), particles with a magnification of 5 nm indicates the orderly stripes subtle which is the special characteristic that distinguishes between the crystalline amorphous. Constituent atoms or single crystal having the structure remains as atoms or molecules constituent regularly arranged in three-dimensional patterns and these patterns repeated periodically in a range of infinitely long [9].

## Electrical Properties

Tests using LCR meter is intended to obtain the resistance value of barium hexaferrite powder doped with Mn, which in turn can be known conductivity value of the powder. The test results using the LCR meter powders can be seen in Table 1 below:

**TABLE 1.** Values of resistance and conductivity of barium hexaferrite powders with doping Mn

| d | 1.18 cm | 1.17 cm | 1.17 cm |
|---|---|---|---|
| Thick (l) | 0.33 cm | 0.31 cm | 0.394 cm |
| f (Hz) | Mn (0.6) 400 | Mn (0.6) 800 | without doping |
| | R (Ω) value | | |
| 100000 | 6.70E+01 | 4.15E+01 | 5.89E+03 |
| 0.5 | 1.26E+03 | 1.33E+03 | 1.28E+06 |
| average | 4.86E+02 | 4.35E+02 | 3.19E+05 |
| A | 1.093588403 | 1.075132 | 1.075132 |
| σ | 6.20E-04 | 6.62E-04 | 1.15E-06 |

Measurement of resistance value is intended to get the value of the electrical conductivity of powder using equation 1, [10]:

$$\boldsymbol{\sigma = \frac{l}{R.A}} \tag{1}$$

Where :

σ = electrical conductivity of materials (S/cm)
l = material thickness (cm)
R = resistance (Ohm)
A = cross-sectional area of material (cm$^2$)

Electric resistance of powder barium hexaferrite with Mn doping is done using the LCR - meter. Based on Table 1, note that the average value of electric resistance material with a variable x = 0.6 with calcinations temperature of 800 °C for 486 Ω and for the variable x = 0.6 with calcinations temperature of 400 °C for 435 Ω. This result is lower when compared with barium powder hexaferrite without doping Mn (3.19x $10^5$ Ω ). This indicates that more and more doping Mn, then the value of electric resistance of barium hexaferrite will be smaller and have an impact on increasing the value of the electrical conductivity of the powder. Heksaferrite barium powder conductivity values to the variables x = 0 of 1.15 x $10^{-6}$ S/cm, while for barium hexaferrite powder with a variable x = 0.6 with calcinations temperature of 400 °C and 0.6 with calcinations temperature of 800 °C, the magnitude sequentially among others of 6.20 x $10^{-4}$ S/cm and 6.62 x $10^{-4}$ S/cm. Increasing the value of the electrical conductivity of these powders is influenced by the addition of Mn that replaced the Fe in Barium hexaferrite. The more Mn is added, the greater the value of the electrical conductivity of the material. The addition of metallic materials in the manufacture of Barium hexaferrite magnets that can be soft magnetic, this material has a permeability properties, barrier types and high conductivity and has a low coercivity, where nature can make such materials as an absorber. Based on the value of this electrical conductivity, the powder doped barium hexaferrite with Mn classified into a semiconductor material with a range between 1 x $10^{-7}$ to 1 x $10^3$ S/cm. Semiconductor material is best used as an absorber of microwaves because these ingredients can transform the microwave energy into heat energy. When microwaves regarding material coated with a microwave absorbing material, it will form an electric field on the surface of the absorbent, after which the current flows as surface currents. When a current flows in the absorber surface, the microwave energy is converted into heat energy form.

## Magnetic Properties

Magnetic measurements of nanoparticles were carried out using the vibrating sample magnetometer and the results are shown in final testing in this study that is testing the magnetic properties of the material. The test results barium hexaferrite powder magnetic properties by doping Mn using a VSM can be seen in Figure 7. One major goal is to engineer the process of giving doping is manipulating magnetic properties of barium hexaaferrite. Based on Figure 7, it was found that a large magnetic remanence of barium hexaferrite with doping Mn of 0.103 emu/g, while the value of coercivity field H amounted to 0,197 T whose value is less than the coercivity field of conventional (Barium hexaferrite without dopants ) of 0.28 T [11].

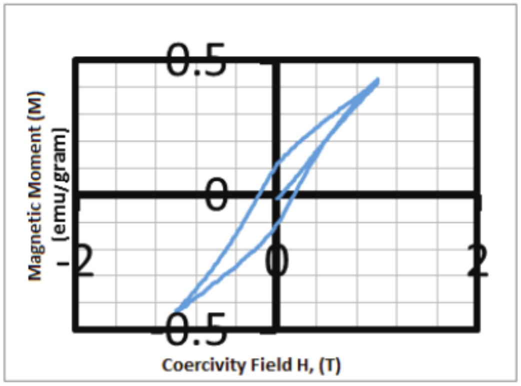


**FIGURE 7**. Hysteresis curve relationship between the coercivity field (H) with a magnetic moment (M) powder doped barium hexaferrite with Mn using VSM

Based on the value of the coercivity field of less than 0.2 T, it can be concluded that it has successfully carried out the reduction of hard magnetic properties of barium hexaferrite through Mn doping mechanism so as to form a soft magnet. Reduction of the hard magnetic properties of soft magnetic is expected to reduce the frequency of the magnetic resonance loss Barium hexaferrite as radar absorbing material [11].

## CONCLUSION

Based on the research that has been done, it can be concluded that; has been successfully performed the synthesis of barium hexaferrite powder with doping Mn as an anti- radar material. The results from XRD indicate that it has formed a hexagonal crystal structure. Based on the results of SEM, it is known that the elements of Ba in the powder amounted to 5.77%, Fe = 55.57%, Mn = 0.61%, O = 38.05%. Generally the presence of barium hexaferrite constituent elements by doping Mn powder was mixed uniformly. The result from TEM, powder has been formed nano-sized particles with an average powder size of 50 nm. Thus the electric conductivity value of barium hexaferrite with Mn classified into a semiconductor material with value 6.62 x $10^{-7}$ S/cm for calcinations until $800^0$C. Semiconductor material is best used as an absorber of microwaves because these ingredients can transform the microwave energy into heat energy. A large magnetic remanence of barium hexaferrite with doping Mn of 0.103 emu/g, while the value of coercivity field H amounted to 0,197 T whose value is less than the coercivity field of conventional (Barium hexaferrite without dopants ) of 0.28 T. The results showed that the powder doped barium hexaferrite with Mn has a hexagonal crystal structure, measuring nano particles, are semiconductors, and classified as soft magnetic. Furthermore based on the results of barium hexaferrite powder characterization by doping Mn is expected to be the anti-radar material.

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