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by Susilawati Susilawati

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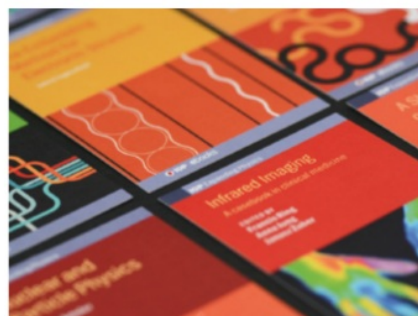
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Synthesis and Characterization of Barium M-Hexaferrite with Metal Doping Mn and Ni for Microwaves Absorbent

Susilawati^{1,a)}, Aris Doyan^{2,b)}, Muhammad Taufik³⁾, Wahyudi⁴⁾

Physics Education, FKIP, University of Mataram, Lombok,
West Nusa Tenggara Indonesia^{1,2,3,4)}

susilawatihambali@unram.ac.id a), aris_doyan@unram.ac.id b)

Abstract. Hexaferrite doped barium powder metal Mn and Ni was synthesized using coprecipitation method. Dopant concentration values varied from $x = 0; 0.2; 0.4; 0.6; 0.8$ and 1.0 , and calcined at temperatures of $400, 600$ and 800 °C. Hexaferrite barium powder synthesized by doping Mn and Ni metal to produce a brown color. The higher the concentration and temperature of calcination given color from dark brown powder further. It has formed barium hexaferrite phase with hexagonal crystal structure is shown from testing using XRD. The crystal structure of the powder measuring nanoparticles using TEM test results. Barium powder hexaferrite by doping Mn and Ni metal belonging to the semiconductor material produced from the test LCR meter. The Barium hexaferrite this falls in soft magnetic seen from the value of coercivity magnetic metal doped Mn and Ni is smaller when compared with barium hexaferrite without doping obtained from measurements using the VSM. All of the measurements indicate powder doped barium hexaferrite Mn and Ni metal potential as an absorber of microwaves.

Keywords: Barium Hexaferrite, Doping, Manganese (Mn), Nickel (Ni), Micro Wave Absorbent

1. Introduction

Microwave absorbing material, including waves emitted by the radar is being developed at this time is the material of the ferrite material. Ferrite material that is emerging today is derived from the type M (Ba, Sr, and Pb) were substituted. BaM as ferrite magnet, besides having the permeability and permittivity relatively high, also has a high electrical resistivity or good semiconductors. The combination of the intrinsic properties of the magnetic properties and electrical properties of BaM like this puts this ferrite magnet material as a buffer of microwaves includes waves with frequencies used in a Radio Detection and Ranging (RADAR) [1]. BaM structure of hexagons ($\text{BaFe}_{12}\text{O}_{19}$) is known as a magnetic material permanently with high performance, it is theoretically composed of crystalline uniaxial anisotropy strong having polarization magnet high saturation (78 emu/g), field coercivity large (6700 Oe), the temperature of Curie High (450 °C) and excellent in chemical stability and resistance to corrosion [2].

Previous study on the BaM that is M-hexaferrite doped with Zn show floured dark colored with a particle size of 100 nm [3]. The results from the SEM-EDX of $\text{BaFe}_{12-x}\text{Co}_x\text{O}_{19}$ obtained that the sample already contained the desired elements evenly. The states that the M-phase composition Barium hexaferrite through coprecipitation method many calcination formed at 800 °C for four hours and states that the optimum condition of the electrical conductivity by doping NiBaM achieved at the calcination temperature of 800 °C with a value of $x = 0.7$. For coercivity value at $x = 0.7$ and the calcination temperature of 800 °C at 0.05 T and the value of magnetization of 2.25 emu/g and can be said to have become the material soft-magnetic material [4].

Based on the study above, the researchers wanted to combine the two doping Mn and Ni in the material barium M-hexaferrite. This is what has been behind the study, and have been synthesized powders BaM ($\text{BaFe}_{12}\text{O}_{19}$) with coprecipitation method, varying the temperature and doping concentration of Ni metal at BaM ($\text{BaFe}_{12}\text{O}_{19}$) to form BaM as a material that has properties of electricity and magnetism. BaM powders were



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characterized by FTIR (Fourier Transform Infra Red Spectroscopy) to determine the crystal structure bonding and XRD (X-ray diffractometer) to determine the crystal structure. To know the elements of the distribution and morphology of the sample used tool of SEM-EDX (Scanning Electron Microscopy) and TEM (Transmission Electron Microscopy) as well as for electrical and magnetic characteristics are used LCR meter and VSM (Vibrating Sample Magnetometer).

2. Method

The basic ingredients of synthetic barium hexaferrite with metal doping Mn and Ni among others, such $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, BaCO_3 , $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ and pure nickel as dopant. Hexaferrite doped barium powder metal Mn and Ni were synthesized using coprecipitation method. Dopant concentration values varied from $x = 0; 0.2; 0.4; 0.6; 0.8$ and 1.0 . Hexaferrite barium powder synthesis stages with metal doping Mn and Ni as follows. The first, dissolve $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in H_2O using a magnetic stirrer for 30 minutes (solution 1), then dissolve BaCO_3 in HCl using a magnetic stirrer hot plate at a temperature of 70°C (solution 2), thus dissolve the powder $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ in H_2O (solution 3). Next step mixing the solution of 1, 2, 3 and distirrer for 30 minutes and add a solution of Ni (solution 4) and titrating (solution 4) with NH_4OH solution until precipitation occurs, then washed with distilled water until pH neutral furthermore samples were dried at a temperature of 80°C [5]. The samples calcined at a temperature of $400, 600, \text{ and } 800^\circ\text{C}$ for four hours and tested using FTIR, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Inductance, Capacitance, and Resistance meter (LCR meter) and Vibrating Sample Magnetometer (VSM).

3. Result and Discussion

3.1 Tests using FTIR instrument

To determine the type of bonding of functional groups contained in the powder samples, then tested using FTIR (Fourier Transform Infra Red). FTIR measurement range at wave number 4000 cm^{-1} to 400 cm^{-1} . Testing samples of barium M-hexaferrite needs to be done to find out the impurities remaining in the samples through functional groups that appear on the test results FTIR form of peaks on the graph. In addition, the wave number less than 750 cm^{-1} is an overview of the functional group Fe-O bond. FTIR testing results can be seen in the figure 1.

There is a peak at wave number 1600 cm^{-1} , these results are similar to previous studies get a peak of 1633 cm^{-1} indicating the presence of water in the sample [3]. Samples without calcination are very large peaks and continue to experience a reduction in the value of the transmission is inversely proportional to the value of the smallest peak calcination temperature up to 800°C calcination contained in the signaling on calcination to 800°C water is present at least. Wave numbers less than 1000 cm^{-1} is said to be as a fingerprint region. In this area there are bonding metal oxide [6]. There is a peak in the area of the fingerprint that is precisely at 530 cm^{-1} , 475 cm^{-1} , 460 cm^{-1} , these results are similar to results of previous studies 585 cm^{-1} , 546 cm^{-1} , 440 cm^{-1} is an area of the presence of functional groups Fe-O and Ba-O [7],[8],[9]. In this area has a transmission value is directly proportional to the value of the calcination temperature. The greater the temperature calcination also increases the value of the transmission [10].

3.2 Tests using X-Ray Diffraction (XRD) instrument

Phase formation in Barium M-hexaferrite are substituted by materials and nickel manganese (Mn and Ni) for $x = 0.6$ at a temperature of 800°C shown in Fig. 2. When analyzed seen some of the peaks and formation phase with a chemical formula that is formed as Table I.

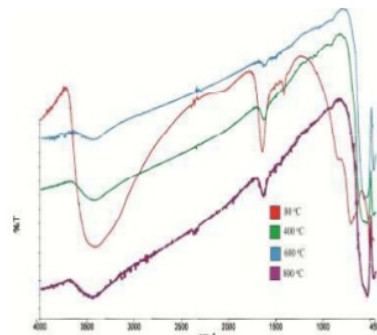


Figure 1a. Relations with the transmission wave number on doping $x = 0.0$

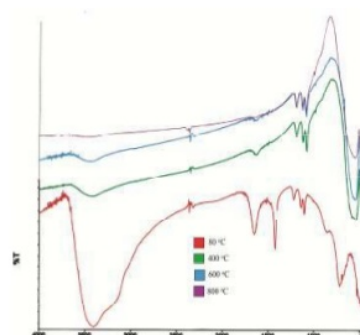


Figure 1b. Relations with the transmission wave number on doping $x = 0.2$

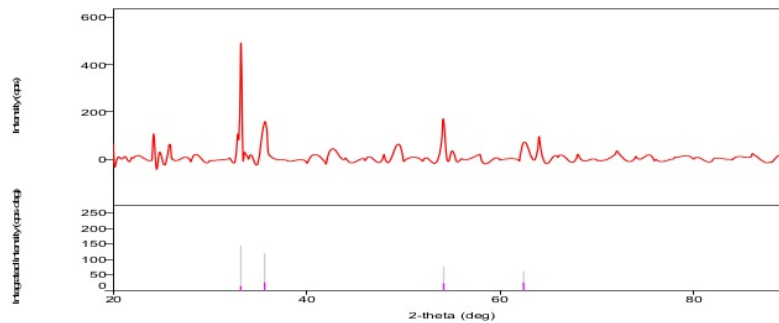


Figure 2. Establishment phase at Barium M-hexaferrites are substituted by materials manganese and nickel (MnNi) $x = 0.6$ at a temperature of 800°C

Table 1. Peak Formed

No	2-theta (deg)	Phase name	Chemical formula
1	33.191	Manganese Iron Oxide	$\text{Mn}_{0.176}\text{Fe}_{1.824}\text{O}_3$
2	35.628	Manganese Iron Oxide	$\text{Mn}_{0.176}\text{Fe}_{1.824}\text{O}_3$
3	54.156	Manganese Iron Oxide	$\text{Mn}_{0.176}\text{Fe}_{1.824}\text{O}_3$
4	62.422	Manganese Iron Oxide	$\text{Mn}_{0.176}\text{Fe}_{1.824}\text{O}_3$

Based shown in Table I. X-ray diffraction pattern with the addition of manganese and nickel dopant concentration of 0.6 with a peak temperature of 800°C are at an angle of 33.191, 35.628, 54.156 and 62.422 deg phase containing Manganese Iron Oxide. The addition of the dopant concentration of the presence of new peaks which are compounds of the dopant [11].

The diffraction pattern is more stable phase transformation occurs after decomposition at temperatures of 840 °C with a concentration $x = 0.4$ to form a single phase is quite stable. In accordance with the results of previous studies in which barium M-hexaferrites phase formed at high temperatures ($T \geq 500$ °C) will form a single phase ie hematite phase [12]. In Table 1. shows that the M-hexaferrite barium-doped manganese and nickel have a hexagonal structure in accordance with the basic material used. This is indicated by the formation of the angle that the axis $a = b \neq c$, $\alpha = \beta = \gamma = 90$ and 120 are $a = 5.0373$ $b = 5.0373 \neq c = 13.752$, $\alpha = \beta = \gamma = 90$ and 120.

3.3 Test Using Scanning Electron Microscopy (SEM) Instrument

The test results of morphological Barium M-hexaferrites ($\text{BaFe}_{11}\text{MnNi}_1\text{O}_{19}$) with Scanning Electron Microscopy shown in Fig 3. Based on observations using Scanning Electron Microscopy (SEM) for each element is given a different color depending goal is to be able to distinguish each element of which (Ba) are given purple, (Fe) yellow, (Mn) of dark blue and (Ni) is light blue, the material is uniformly dispersed in the formation of Barium M-hexaferrites. According to research results in the presence of doping appears it indicates that doping has been substituted for Fe and has been formed microstructure of powder barium M-hexaferrite them [4], [13], [14].

3.4 Test Using Transmission Electron Microscopy (TEM) Instrument

For the analysis of microstructures, crystal structure, and the elemental analysis of nanometer scale testing using a TEM. The test results ($\text{BaFe}_{11}\text{MnNi}_1\text{O}_{19}$) As can be seen in Fig. 4. In Fig. 4. the morphological structure of the nanoparticles formed, $\text{BaFe}_{12}\text{MnNi}_1\text{O}_{19}$ TEM image shows the distribution of particle size reaches the order of nm. So we can say that this material included in the nanomaterial because these particles are particles with a smaller size to reach 10 nm. TEM images provide information that the crystalline structure is formed and includes nanomaterial, according to the study the size of 25 nm to 70 including nano meter, the size of the material is very important because when the dimensions of the material to the value of a few nanometers, many physical properties or chemically depending on the size [4], [15].

3.5 Test Using LCR meter Instrument

Conductivity Test Results Barium M-hexaferrites ($\text{BaFe}_{11}\text{MnNi}_1\text{O}_{19}$) Using LCR Meter seen in Table II. Based on calculations, the conductivity values obtained in the samples without doping of 1.15×10^{-06} S/cm. The conductivity value indicates that the sample is still a conductor. While the sample with $x = 0.9$ doped with calcination temperature of 400 °C has a conductivity value of 4.52×10^{-05} S/cm, which means the sample has semiconductor properties. In samples with calcination temperature of 800 °C has a conductivity value 3.74×10^{-04} S/cm, which means the sample has semiconducting properties together with the calcination temperature of 400 °C. Although both include semiconductor materials, but the samples at the calcination temperature of 800 °C has a conductivity of greater value when compared with a sample of 400 °C. In accordance with the study of Barium M-hexaferrite doped Zn using coprecipity properties of semiconductor materials [3].

The value of conductivity of 1×10^{-5} - 6×10^{-5} S/m by synthesis using sol-gel method without doping [16]. It also obtained the conductivity value of 10.03×10^{-4} S/m using sol-gel method and doped with Mg [17]. The different results showed performs synthesis by doping Mn obtain conductivity value of 0.05 S/m [18].

3.6 Test Using Vibrating sample Magnetometer Instrument

The result of magnetic properties of Barium M-hexaferrites ($\text{BaFe}_{11}\text{Co}_1\text{O}_{19}$) with Vibrating Sample Magnetometer show in Fig 5. To determine the magnetic properties of Mn and Ni substitutes dopants on barium M-hexaferrite then be tested using the VSM. This tool can be obtained information about the quantities of magnetic properties as a

result of changes in the external magnetic field that depicted in hysteresis curve showing the magnetic properties, the valuecoercivity, remanence magnetization and magnetic properties of materials as a result of temperature changes [19].

Fig. 5. shows that the hysteresis curve on a sample of barium M-hexaferrites with the dopant concentration $x = 1.0$ and the calcination temperature of $800\text{ }^{\circ}\text{C}$ has a value of coercivity of 0.04 T and the value the magnetization of 5.0 emu/g . Hysteresis curve of the sample has a fairly narrow curves. Refinements in the sample width of the curve due to the presence of structures other than $\text{BaFe}_{12}\text{O}_{19}$ but the presence of Fe_2O_3 which tend to be soft magnetic. The best radar absorbing material contained in the value the low coercivity and high magnetization ranged at 0.05 T and 5.0 emu/g at $x = 0.7$ [4]. Meanwhile, another showed that the value of a coercivity is 0.0506 T and a the value of magnetization 14.782 emu/g at $x = 0.3$ [20]. This was disclosed that at $T < 800\text{ }^{\circ}\text{C}$ has the value of a coercivity under $0,025\text{ T}$ and magnetization under 0.53 emu/g [21].In this research, the the value coercivity of 0.04 T and the magnetization of 5.0 emu/g with calcination temperature $T \geq 800\text{ }^{\circ}\text{C}$ showed that the barium M-hexaferrites that metal doping Mn and Ni can be said to have included soft magnetic.

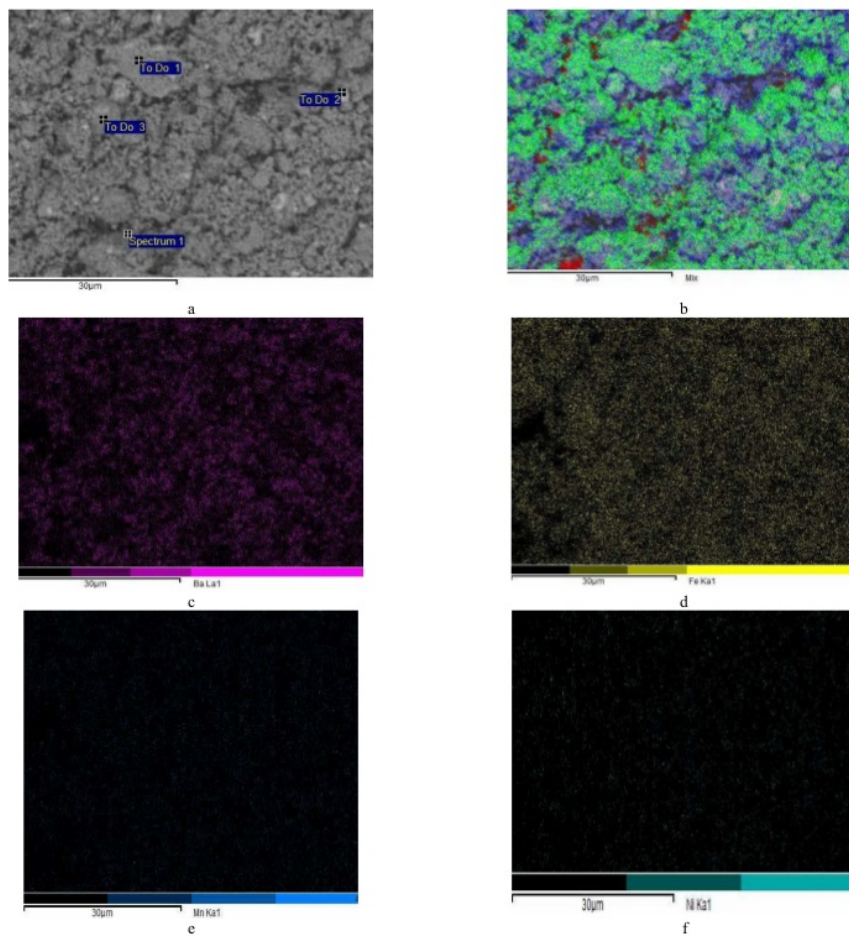


Figure 3. Results of morphological Barium M-hexaferrites shaped like a sponge (a) and (b) the distribution of Barium M-hexaferrite (c). Ba (Barium) (d). Fe (Iron) (e). Mn (Manganese) (f). Ni (Nickel)

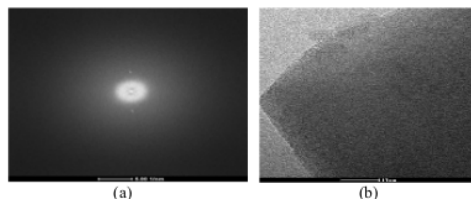


Figure 4. The morphological Barium M-hexaferrites structure of nanoparticle formed using *Transmission Electron Microscopy* (TEM) Image, (a) 5 nm and (b) 10 nm

Table 2. Result of Testing the Electrical Properties of Barium M-Hexaferrite Samples using LCR Meter

Diameter	1.17 cm	1.17 cm	1.17 cm
Thick	0.314 cm	0.296 cm	0.394 cm
Frequency (Hz)	MnNi 0.9 800 ⁰ C	MnNi 0.9 400 ⁰ C	Nodoping
Z aveage (ohm)	7.37E+02	6.46E+03	3.19E+05
Area A	1.075131546 cm ²	1.075131546cm ²	1.075131546 cm ²
Conductivity σ	3.74E-04 S/cm	4.52E-05 S/cm	1.15E-06 S/cm

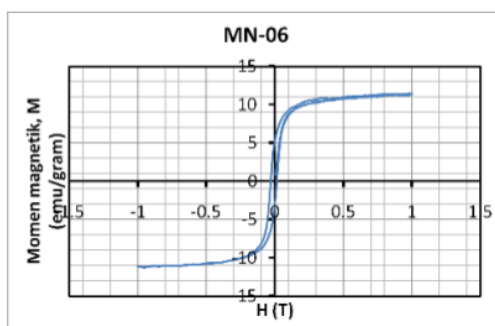


Figure 5. The hysteresis curve Barium M-hexaferrites doping MnNi ($BaFe_{11}MnNi_1O_{19}$) with a temperature of 800⁰C

4. Conclusion

Has managed to synthesize barium powder M-hexaferrite by combining two doping Mn and Ni with coprecipitation method, varying the temperature and doping concentration of Ni metal at BaM ($BaFe_{12}O_{19}$) to form BaM. All the results of characterization data showed BaM doped with Mn and Ni as a material that has properties of electricity and magnetism can be said to have included soft magnetic material and is suitable as an absorber of microwaves.

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