

# Crystal Structure and Magnetic Properties of Zn doped Barium M-Hexaferrite

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## Abstract

Synthesis of Zn doped Barium M-Hexaferrite ( $\text{BaFe}_{12-x}\text{Zn}_x\text{O}_{19}$ ) has been performed by coprecipitation method. The purified iron sand from Tulungagung is used as a precursor of  $\text{Fe}_3\text{O}_4$ . Synthesis of Zn doped Barium M-Hexaferrite with variations of  $x = 0.3, 0.5,$  and  $0.7$  has been calcined at temperatures of  $1000^\circ\text{C}$  for 5 hours. Ion  $\text{Zn}^{2+}$  (with  $0 \leq x \leq 0.7$  wt %) does not change the crystal structure of Barium M-Hexaferrite (BaM), but give a slight displacement of the peak position of the diffraction pattern. SEM figures showed that Zn doped Barium M-Hexaferrite have a hexagonal structure, similar to BaM structure. Doping of Zn has changed the magnetic properties of Barium M-Hexaferrite (BaM), from hard magnetic become soft magnetic. Barium M-Hexaferrite (BaM) has a value of Coercivity Field (Hc) and Remanence Magnetization (Mr) is  $0.03734$  T and  $8.334$  emu/g. At variation  $x = 0.3$ , the Remanence Magnetization (Mr) reaches the highest value. At this point, a value of Coercivity Field (Hc) and Remanence Magnetization (Mr) is  $0.0506$  T and  $14.782$  emu/gram respectively.

KEYWORDS: Coprecipitation method, Barium M-Hexaferrite, Crystal Structure, Magnetic Properties.

## I. INTRODUCTION

Tulungagung is one of village having abundant natural resources, one of which is a natural iron sand. The low knowledge of the content and benefits of iron sand led to its use is still less than the maximum. Therefore, iron sand should be changed to material science that has a higher economic value and environmental friendly.

Radar Absorbing Materials (RAM) has been studied and applied using different materials. Barium M-Hexaferrite is a permanent magnet having high magnetic anisotropy [1], good stability, high magnetic saturation and coercive field (hard magnetic material)[2]. Barium M-Hexaferrite's magnetic properties can be reduced by the substitution of  $\text{Fe}^{3+}$  with divalent ions (Zn, Co, Ni, etc). Through this substitution process it is expected that Barium M-Hexaferrite will be able to be applied as electromagnetic wave absorber within X-band. The goal of this research will be the production of Barium M-Hexaferrite particles from natural iron sand.

## II. METHODS

This research started with the purification of iron sand. First, wet milling with a speed of 150 rpm for 15 minutes performed of iron sand to get a smaller size. Synthesis process was done by co-precipitation methods. Fundamental materials ( $\text{BaCO}_3$ , Zn powder, and iron sand of  $\text{Fe}_3\text{O}_4$ ) following their corresponding mass and molar ratio were then dissolved sep-

arately in HCl, then It's being mixed up by hot stirrer media. The next step was neutralization using 7 M NaOH solution. That solution was cleaned up by aquades from impurity contents. The obtained precursor was then calcinated at the temperature that was set by the DTA/TGA test result, this temperature was  $1000^\circ\text{C}$  hence resulting Barium M-Hexaferrite powder.

The phase compositions analysis was done with the help of X-ray diffraction data that were collected from X-ray diffractometer using  $\text{Cu}_{k\alpha}$  1,5418 Å radiation source, operated at 40 kV voltage and 30 mA current. Analysis of magnetic properties of Barium M-Hexaferrite was undertaken using VSM with induction magnetic field up to 1 Tesla. While for microstructure and atomic distribution analysis, SEM and EDX were utilized respectively.

## III. RESULTS AND DISCUSSION

### Phase Analysis of The X-Ray Diffraction

Figure 1 shows that the two phases were formed after calcinations at temperature of  $1000^\circ\text{C}$  for 5h, those are Barium M-Hexaferrite ( $\text{BaFe}_{12}\text{O}_{19}$ ) and Hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ). Hematite phases ( $\alpha\text{-Fe}_2\text{O}_3$ ) is the stable phases of  $\text{Fe}_3\text{O}_4$  contained in the iron sand. Figure 1 also indicates a diffraction pattern under variation of  $\text{Zn}^{2+}$  ion doping condition.  $\text{Zn}^{2+}$  ion having  $0.074$  nm ionic radius was dissolved into the crystal structure of Barium M-Hexaferrite replacing  $\text{Fe}^{3+}$  ion having  $0.064$  nm in tetrahedral position. This statement can be proved from the fact that there is a slight displacement of the peak position around  $2\theta$  that indicate the replacement of  $\text{Fe}^{3+}$  ion with  $\text{Zn}^{2+}$  ion that has greater ionic radius than  $\text{Fe}^{3+}$  does.

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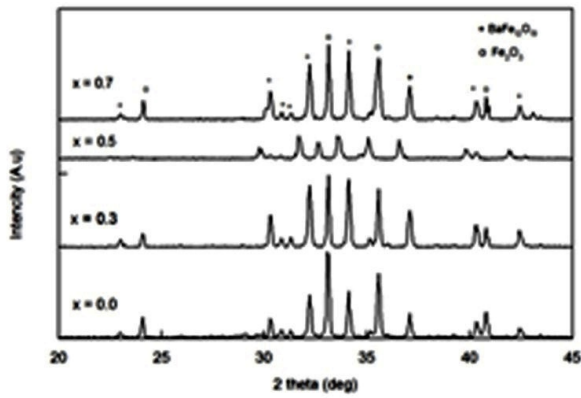


FIG. 1: X-ray diffraction pattern of Barium M-Hexaferrite under Zn doping variation.

So the distance between crystal planes becomes farther after  $Zn^{2+}$  ion doping. Aside of those, there is also lattice parameter change a and c that caused from the ionic radius difference between  $Zn^{2+}$  and  $Fe^{3+}$ . This increase of lattice parameter confirms the pre-statement that  $Zn^{2+}$  would substitute the position of  $Fe^{3+}$  throughout the structure of Barium M-Hexaferrite. Rietveld refinement using Rietica software was executed in order to get relative phase composition of Barium M-Hexaferrite.

**Microstructure Analysis Using SEM**

Sample in this observation was  $BaFe_{11.7}Zn_{0.3}O_{19}$ . Figure 2 shows that Barium M-Hexaferrite crystal has hexagonal shape with  $5\mu m$  diameter. This agrees with the theory that says that Barium M-hexaferrite has hexagonal crystal structure charac-

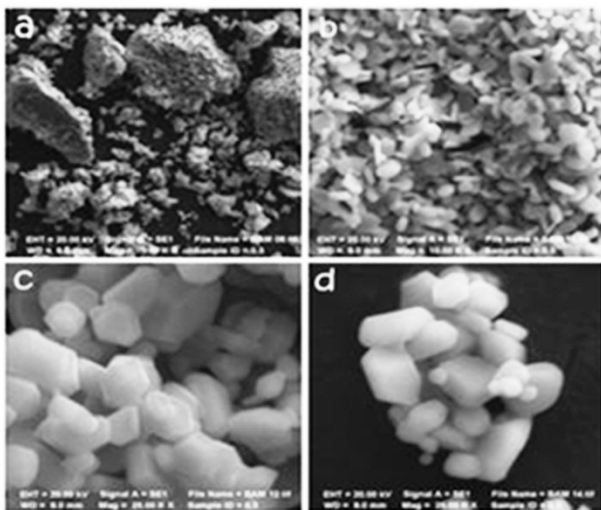


FIG. 2: Morphological shape of  $BaFe_{11.7}Zn_{0.3}O_{19}$  as seen through SEM.

TABLE I: Relative phase composition of Barium M-Hexaferrite for different doping concentrations.

Doping Variation (X)	% weight Barium M-Heksaferrit	% weight Hematit	GoF
0	43.45	56.55	1.8
0.3	62.64	37.36	1.6
0.5	62.04	37.96	1.5
0.7	64.66	35.34	1.5

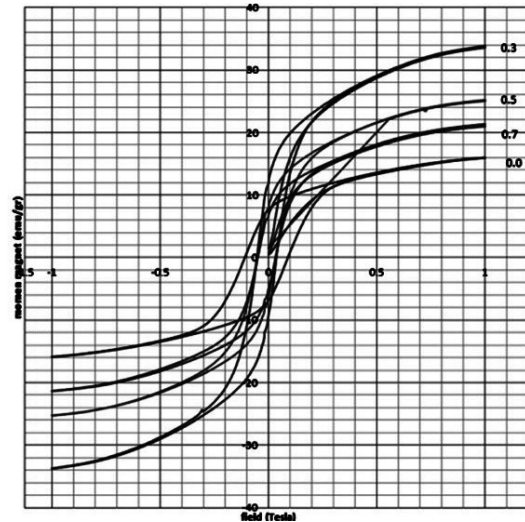


FIG. 3: VSM's result of Barium M-Hexaferrite for each concentration of  $Zn^{2+}$  ion doping.

terized by two lattice parameters: hexagonal plane width (a), and crystal height (c), with  $a = 0.588\text{ nm}$  and  $c = 2.32\text{ nm}$  [3].

**Analysis of Magnetic Properties using VSM**

Figure 3 are the results of magnetic properties measurement for each doping variation. From the hysteresis curve resulted from VSM for each doping variation displayed above, one can make a table relating the values of remanence magnetization ( $M_r$ ) and coercivity ( $H_c$ ) for each doping variation. This is provided in Table II.

For doping addition  $x = 0.3; 0.5; 0.7$ ; the value of coercivity is smaller compared to BaM without doping. This result suggests that the addition of doping would reduce the significant amount of energy loss so that it exhibits soft magnetism characteristic. In line with the purpose of adding  $Zn^{2+}$  ion doping in this research, it's expected that the magnetic domains within BaM become again disoriented in all direction.

The magnetic properties of Barium M-Hexaferrite are originated from  $Fe^{3+}$  ion having  $5\mu_b$  magnetic moment. The distribution of magnetic moments within Barium M-Hexaferrite structure are  $1\uparrow$  trigonal bipiramidal  $+7\uparrow 2\downarrow$  oktahedral  $+2\downarrow$  tetrahedral, this means the total magnetic moment for Barium M-Hexaferrite is  $4\uparrow = 20\mu_b$  [3]. Magnetic saturation of

TABLE II: Measurement results of the magnetic properties of Barium M-Hexaferrite.

$X$	Coercive field, Hc (Tesla)	Remanence, Mr (emu/gram)
0	0.1734	8.334
0.3	0.0506	14.782
0.5	0.0591	10.243
0.7	0.0528	8.667

Barium M-Hexaferrite can be made higher by the substitution of non magnetic ions such as  $Zn^{2+}$ . It's because  $Zn^{2+}$  ions likely tend to occupy tetrahedral position, meanwhile inside Barium M-Hexaferrite structure the tetrahedral position yields structure that opposes octahedral position wherein it's the octahedral position that produces total magnetic moment. That explains why the substitution of  $Zn^{2+}$  ions can reduce

the negative magnetic moment thus increasing total magnetic moment as well as decreasing coercive field.

#### IV. CONCLUSIONS

The synthesis of Barium M-Hexaferrite (BaM) has been synthesis from natural iron sand. Ion  $Zn^{2+}$  (with  $0 \leq x \leq 0.7$  wt%) does not change the crystal structure of Barium M-Hexaferrite (BaM), but give a slight displacement of the peak position of the diffraction pattern. Barium M-Hexaferrite crystal has hexagonal shape with  $5\mu m$  diameter. The coercive field and magnetic remanence of BaM before doping yielded the value 0.1734 T and 8.334 emu/gram respectively. Magnetic properties of BaM has been successfully reduced with optimum doping concentration at  $x = 0.3$ , coercive field at 0.0506 T, and magnetic remanence at 14.782 emu/gram.

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