

# Synthesis and Characterization $BaFe_{12-2x}Ni_xCo_xO_{19}$ Using Chemical Coprecipitation and Variations of Calcination Temperature

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## ABSTRACT

$BaFe_{12-2x}Ni_xCo_xO_{19}$  ( $x = 0, 0.1, 0.2$  % mol) was synthesized using chemical coprecipitation method.  $BaCl_2$ ,  $FeCl_3$ ,  $NiCl_2$  and  $CoCl_2$  powder were mixed and precipitated with 1M NaOH solution using as precipitant. Nickel and Cobalt dopan influence on the structure, morphology and magnetic properties were characterized using XRD, SEM and VSM. The results of XRD analysis showed that all samples have a dominant phase  $BaFe_{12}O_{19}$  hexagonal while the calcined samples showed no peak. Shifting the corner in terms of the field (114) indicates  $2\theta$  angle shifted towards larger. VSM data indicates that the sample without calcination showed paramagnetic properties and low coercivity figures showed. Calcined sample has a coercivity value tends to increase with increasing calcination temperature used.

**Keywords:** Heksaferrit Barium, Cobalt, Calcination, Coprecipitation, Nickel

## I. INTRODUCTION

Nanomagnetic has been studied and conducted research for a very wide applications such as electronic components for *mobile* and wireless at microwave frequencies / GHz, electromagnetic wave absorbers and radar absorber material (RAM). Nanomagnetic material developed in this research is Barium Heksaferrit. Barium-Heksaferrit has been widely studied because it has many advantages such as a relatively cheap price, coercivity and a high curie temperature, corrosion resistant. Barium Heksaferrit can be synthesized by several methods such as sol-gel, crystallization gases, aerosols, scouting mechanical and coprecipitation [5].

The magnetic properties such as coercivity permanent magnet is highly dependent on the grain size [6].

Processing precursor by coprecipitation method requires a precipitant which has  $[OH^-]$  group such as NaOH and  $NH_4OH$ . Engineering heksaferrit magnetic properties of barium can be done by substitution with divalent ions. There are several types of cations that allow for substituted for barium heksaferrit, such as  $Ni^{2+}$ ,  $Co^{2+}$ ,  $Zn^{2+}$ ,  $Cu^{2+}$  [1]. Transition  $Ni^{2+ / 3+}$  and  $Co^{2+ / 3+}$  in the  $Fe^{2+ / 3+}$  occurs due to similarity of the ionic radius and the electron configuration [3]. Coprecipitation method is the best method for heksaferrit mempreparasi purely because it produces better homogeneity, ease in controlling weight in it and can be synthesized at low temperatures [2]. In this study used additive materials Nickel (Ni) and Cobalt in the form of nickel chloride ( $NiCl_2$ ) and Cobalt Chloride ( $CoCl_2$ ).

Research has been done related to the manufacture of permanent magnets barium heksaferit done by coprecipitation and material variation additive NiCl<sub>2</sub>·COCl<sub>2</sub> with x = 0, 0.1, 0.2 (mol%) and temperature treatment before calcination and calcination at 750 °C and 950 °C.

## II. METHODS AND MATERIAL

Sample preparation barium heksaferit substituted with Nickel and Cobalt from powder BaCl<sub>2</sub>·FeCl<sub>3</sub> was mixed and stirred with 100mL demineralised water. The solution was stirred precipitant. Precipitant used is 1M NaOH and then dropped into a precursor solution on an ongoing basis. The solution was stirred and allowed to settle. The precipitate was then calcined at a temperature of 750 °C and 950 °C for 4 hours. The precipitate was dried and then ground to a powder in order to facilitate the process of characterization. Characterization is done by using X-Ray Diffraction (Smartlab-Rigaku, with Cukα radiation, λ = 1.5406 Å) which aims to observe the phases formed in the test sample before and after calcination of 750 °C and 950 °C for 4 hours in the atmospheric environment and Vibrating Sample Magnetometry which aims to determine the magnetic properties.

## III. RESULTS AND DISCUSSION

Figure 1 is a graph of the TGA showed that the mass shrinkage is most clearly seen at a temperature of 24.3 °C - 350 °C with a mass reduction of 2.58 mg. Depreciation identified mass to a temperature of 962.5 °C and no more mass shrinks significantly. Contained a small peak at a temperature of 715.6 °C on the DTA graph above shows an exothermic reaction which indicates the occurrence of precursor Barium pengintian Heksaferit. In addition, the higher peaks are also formed at a temperature of 849 °C, which indicates the optimum temperature formation of crystallization. DTA chart above does make reference to the calcination process. Calcination performed at

two temperatures: at 750 °C and 950 °C. This is done in order to be the optimum temperature comparison to the formation of large crystals of barium heksaferit before and after the crystallization temperature (849 °C).

Temperature calcination affects the structure of barium heksaferit formed. In this study, given three variations in temperature that is not calcined, and calcining at 750°C and 950°C. The diffraction pattern of the three temperature treatment can be seen in Figure 2.

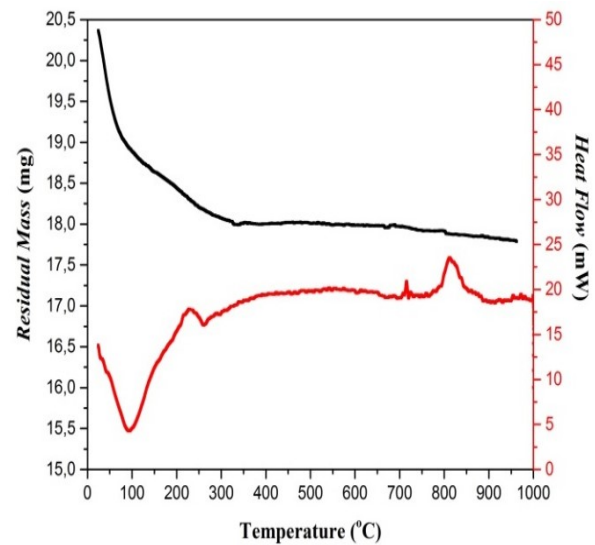


Figure 1. TGA and DTA

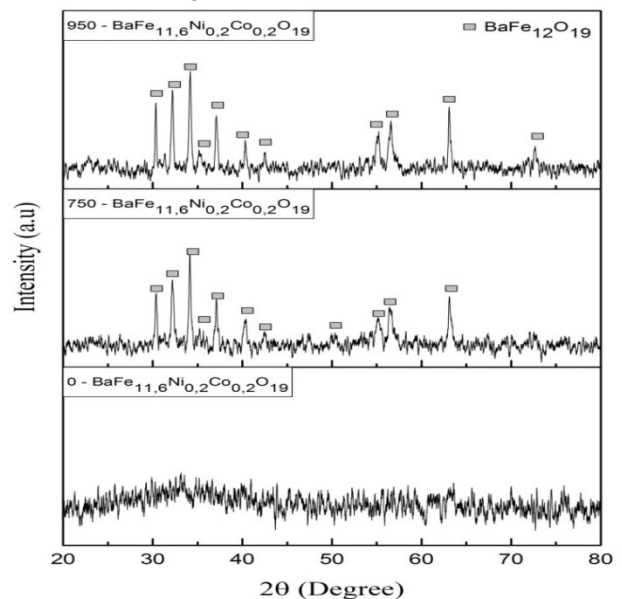


Figure 2. X-ray Diffraction Patterns on BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> (x = 0.2 mol% Ni and Co) without calcination and calcination at 750 °C and 950°C.

Figure 2 shows that the samples before calcined not indicate a dominant peak of barium heksaferit. The diffraction pattern shown by the sample before calcination is a diffraction pattern for the amorphous structure. The sample has been calcined at a temperature of 750°C and 850°C showed peaks heksaferit barium. The tops have been identified using *software Match!*, The data indicates that the sample BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> are calcined at a temperature of 750°C and 950°C has a dominant peak heksaferit barium and there are no impurities such as hematite and monoferit in it. Data from this study indicate that the calcination is very influential on the growth of crystals of barium heksaferit.

Based on the XRD patterns so we can determine the value of the lattice parameters such as the lattice constant parameter, the ratio of the lattice parameter, *d-spacing*, volume. Table 1 shows the values of the lattice parameters. Lattice parameters a and c BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> showed a nearly significant with data from the ICSD Card No.201654 ray wavelength CuKα = 1.54060 Å with lattice this parameter a = 5.892 Å and c = 23.18 Å. A value in this study was 5.891 Å to a temperature of 750 °C and 5.896 Å for the temperature of 950 °C. The values of 23.24 Å c to 750 °C and 23.245 Å to 950 °C, whereas for the samples calcined barium dominant peak is not found heksaferit. The value of the magnetization of the sample BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> (x = 0 and 0.2 mol% of Ni and Co) before calcination can be seen in Table 2.

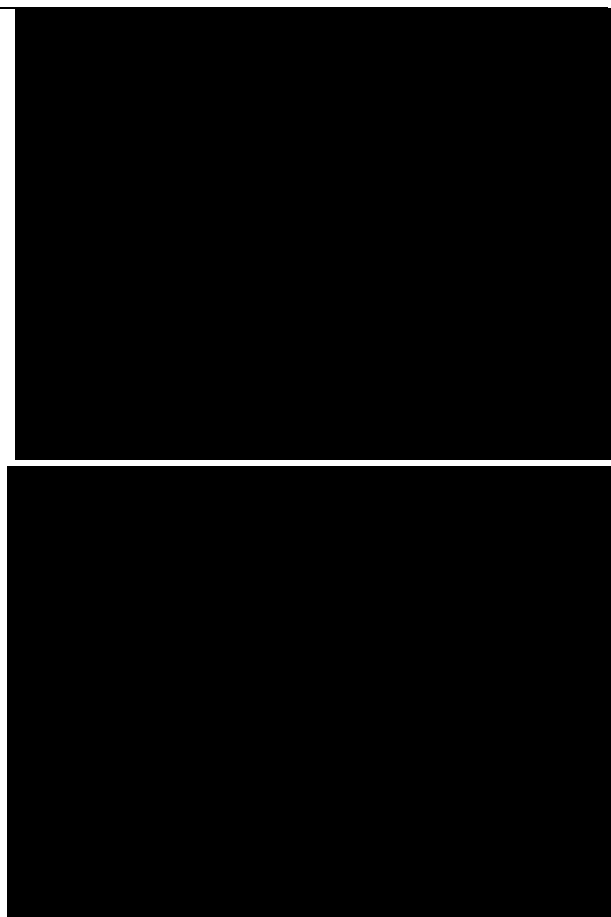
Table 2 shows that the sample before calcination showed a low value of magnetization. It is characterized also by the image represented by Figure 3.

Figure 3 shows that the barium heksaferit without doping with calcination temperature of 750 °C to form a new phase which causes the small value of the magnetization and coercivity. Another phase is formed which is BaFe<sub>2</sub>O<sub>4</sub>. Barium Heksaferit calcined at low temperature indicates the value of the

magnetization and coercivity are small because they are amorphous product and there is still a phase of intermediaries such as Fe<sub>2</sub>O<sub>3</sub>, BaFe<sub>2</sub>O<sub>4</sub> and BaCO<sub>3</sub> [4]. Hysteresis loop of BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> (x = 0.1 and 0.2 mol%) with a calcination temperature of 750 °C and 950 °C can be seen in Figure 4 and Figure 5.

**Table 1.** Lattice parameters of BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> (x = 0; 0.1; 0.2; 0.3 mol%) with a calcination temperature of 750 °C

Sample	Tem	a (Å)	c (Å)	d (Å)
	p (°C)			
BaFe <sub>11,6</sub> Ni <sub>0,2</sub> Co <sub>0,2</sub> O <sub>19</sub>	750	5,891	23,24	2,52
BaFe <sub>11,6</sub> Ni <sub>0,2</sub> Co <sub>0,2</sub> O <sub>19</sub>	950	5,896	23,25	2,90

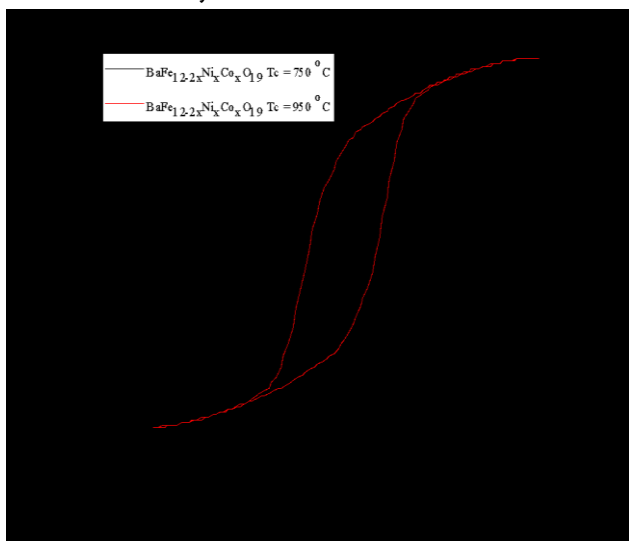


**Figure 3.** Hysteresis loop (a) precursor Heksaferit pure Barium (b) BaFe<sub>11.6</sub>Ni<sub>0.2</sub>Co<sub>0.2</sub>O<sub>19</sub> before calcination.

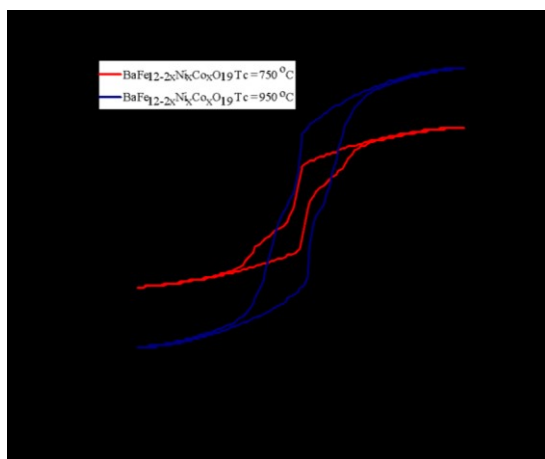
**Table 2.** Value magnetization for samples, BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> (x = 0 and 0.2 mol% of Ni and Co) before calcination.

No	magnetization	x (mol%)	
		0	0.2
1	saturation (emu / g)	1.32	2.5
2	remanence (emu / g)	0.02	0.01
3	Coersivity (kOe)	225.67	110, 96

Values of saturation magnetization of pure Barium Heksaferit at a temperature of 950 °C is 57.18, remanence magnetization value is 28 , 45 emu / g with a coercivity value of 6.515 kOe



**Figure 4.** Hysteresis loop BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> (x = 0.1 mol%) with a calcination temperature of 750 °C and 950 °C.



**Figure 5.** Hysteresis loop BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> (x = 0.2 mol%) with a calcination temperature of 750 °C and 950 °C.

#### IV. CONCLUSION

It has successfully prepared material BaFe<sub>12-2x</sub>Ni<sub>x</sub>Co<sub>x</sub>O<sub>19</sub> ( x = 0, 0.1, 0.2, 0.3 mol%) with Ni and Co doping using coprecipitation method shown by the analysis results of XRD and VSM. XRD characterization results indicate that all samples have been synthesized have a dominant structure of barium heksaferit. The shifting of the diffraction peak at the (114) direction of greater prove a change in lattice parameter. VSM testing results show that the samples calcined has a coercivity that increase compared to the figure without calcination and increasingly with increasing calcination temperature used.

#### V. ACKNOWLEDGEMENT

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