

Synthesis of BaFe₁₂O₁₉ Using Templates from Starch

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Abstract

BaFe₁₂O₁₉ synthesis studies have been conducted based sol-gel method using chitosan as a dispersant and starch as an organic template. Starch is derived from tapioca. The test results using x-ray diffractometer (XRD) indicates the phase formed is barium hexaferrite. Based on the image of the Scanning Electron Microscopy (SEM) obtained morphology varies namely: hexagonal, granular and nanorod with size varies according to the amount of starch used. Based on the curve hysteresis test results of the samples using a Vibrating Sample Magnetometer (VSM) shows that the magnetic properties BaFe₁₂O₁₉ namely: the magnetic moment saturation (Ms), the magnetic moment remanence (Mr) and coercivity (Hc) tends to increase after the addition of starch. A very significant increase in coercivity value amounted from 0.07 tesla to 0.28 tesla occurred in the use 10% starch volume per volume and best BaFe₁₂O₁₉ magnetic properties obtained in the use of starch 0.5% wight per volome (w/v) as much as 10% volume per volume (v/v). Thus the starch can produce nanorod morphology lead to improving the magnetic properties of barium hexaferrite.

Keywords: BaFe₁₂O₁₉, Sol-gel, Chitosan, Starch.

INTRODUCTION

Barium Hexaferrite is one of the permanent magnets of the most widely used and is still promising because it has properties: superior, low production cost, high Curie temperature, high coersivity, chemical stability and corrosion resistance (Kaynar et al, 2015). Barium hexaferrite can also be written as Ba₆.(Fe₂O₃) or BaFe₁₂O₁₉ by (Kaynar et al, 2015). The permanent magnet material is one of a very wide application, such as: the electric generator systems, electric drive systems / electric motors, industrial automation, data storage devices, microwave devices, transformers, speakers, and so on. The use of a permanent magnet is determined by the nature and quality of the magnetic material (Widyan and Hanitsch, 2012).

Barium hexaferite also a magnet as it includes oxygen, it tends to be a hexagonal shaped morphology. Magnet based oxide has all the traits commonly associated with metal-based magnet (or atom based), such as remanence to saturation and remanent magnetization and koersivity (Miller, 2014). According Kanagesana et al (2011) coercivity barium ferrite depends on

many factors such as chemical composition, particle size, degree of crystallinity, micro, magnetic anisotropy, and others.

Some of the results of relevant research previously morphology barium hexaferrite obtained, generally hexagonal (Mahgoob and Hudeish, 2012; Li et al, 2012; Tan and Chen, 2013; Zhao et al, 2013; Ahmed et al, 2013) and a round shape (Chen et. al, 2012; Khorrani et al, 2013; An et al, 2014b; Mosleh et al, 2014; Xu et al, 2015). Based on the morphology of the barium ferrite, a magnetic field in the rod-shaped morphology (nanorod) will be greater than the spherical morphology (Mohapatra et al, 2015).

To get the morphology of the nanorod one of them can be done through a wet-chemical process the sol-gel method using a dispersant and templates. Dispersant acts to minimize the size of the nanoscale morphology to obtain while the template serves as a shaper of the desired morphology. Sol-gel method is a wet chemical process engineering (chemical solution deposition), which has been widely used in the fields of materials science (Tseng et al, 2010). Sol-gel process has advantages not only in reducing the temperature but also can control the homogeneity of the solution (Gurbuz et al, 2012), Sol-gel is seen as a useful method for preparing nano crystal BaFe₁₂O₁₉ (kaynar et al, 2015) using the soles we gel easily add dispersant and templates, one type of dispersant is Chitosan.

Chitosan is a polysaccharide derived from the deacetylation of chitin animal (Elsabee and Abdou, 2013). Chitosan acts as a structural dispersant because it contains amino and hydroxyl functional groups capable of binding metal ions. Chitosan can be used as a dispersant (Zhang et al, 2010). Chitosan is good for the iron oxide nanoparticles (Tsai et al, 2010). Templates are used in the relevant research beforehand is a template synthesis, among others: (Ethylene Glycol (EG), Propylene Glycol (PG), Electrospun polyethylene terephthalate) / citric acid (PET / CA) microfibers and α -FeOOH, in this study template used is a natural organic template, namely starch.

Starch is a glucan soluble composed of two glucose polymers, amylopectin and amylose (Corre et al, 2010). According Elgadir et al (2012) starch derived from botanical sources vary in composition, molecular structure and sequence of the constituent polysaccharides (amylose and amylopectin). Amylopectin has a branched structure while amylose has a linear structure. Amylose content in starch is expected to act as a template nanorod. One of starch are easily available and relatively cheap is tapioca starch, tapioca starch use once using local wisdom.

Relevant research before the synthesis of barium hexaferrite that produce nanorod morphology has been done by: Cao et al (2010) used for the oxidation of α -FeOOH, γ -FeOOH to form a crystal nucleus generating particle morphologies such as barium hexaferrite rod diameter 72.24 nm (α) and 74.74 nm (γ), with magnetic properties $M_s = 51.30$ emu/g, $M_r = 26.83$ emu/g, $H_c = 0.552$ T (α) and $M_s = 49.99$ emu/g, $M_r = 24.33$ emu/g, $H_c = 0.330$ T (γ). Galvao et al (2014) uses a template ethylene glycol to produce particles of barium hexaferrite nanorod morphology has a length of 90 nm magnetic properties saturation magnetization (M_s) = 55 emu / g; coercivity (H_c) = 0.49 Tesla, Septiadi and Purwasasmita (2014) uses a template tapioca produce barium hexaferrite particle morphology of the nanorod diameter = 200 nm and (200-500) nm, having magnetic properties remanence magnetization (M_r) = (23.2 and 19.6) emu / g, coercivity = (0.14 and 0.12) T and Xu et al

(2014) uses a template to produce α -FeOOH particles of barium hexaferrite nanorod morphologies having a length of 250 nm M_s magnetic properties = 62.5 emu/g; $H_c = 0.38$ Tesla.

EXPERIMENT

Barium Nitrate ($Ba(NO_3)_2$), 99.9%, Merck), Iron (III) Nitrate nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$, 99.9, Merck) are used as precursors with molar ratio of 1:12 according to the ratio stoichiometry. Acetic acid (CH_3COOH , 99.8%, Merck) was used as the solvent of chitosan, chitosan types of low molecular, tapioca brands Rose, aqua Demineralized (DM) as the solvent. First created a 1% solution of chitosan (a ratio of 1 g of chitosan was dissolved in 100 ml of 2% acetic acid) and then made a solution of tapioca 0.5% (with a ratio of 0.5 grams of tapioca in 100 ml aqua DM) at 70°C of temperature.

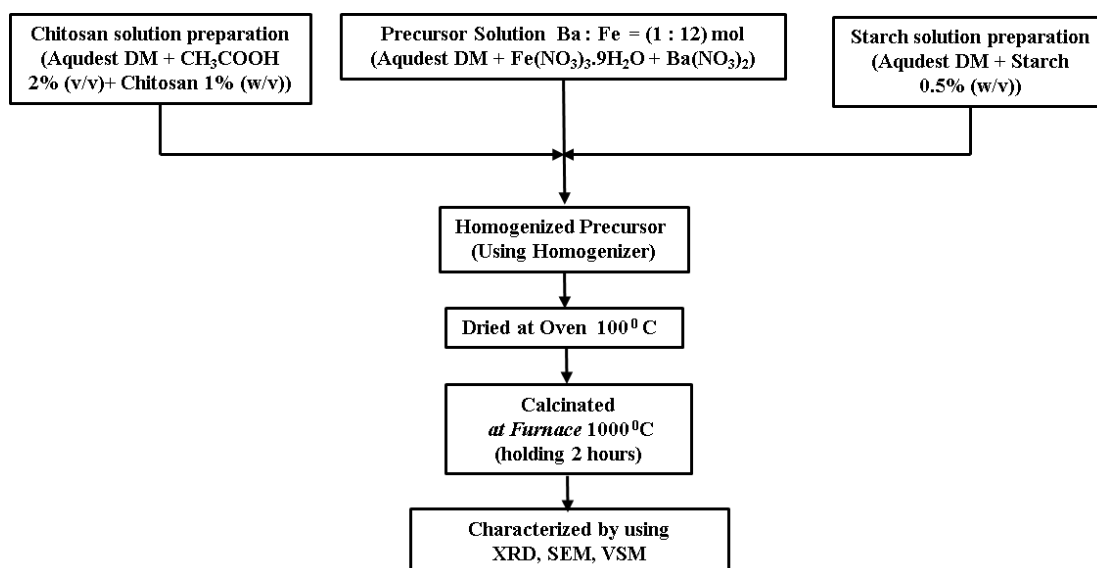


Figure 1. Flow chart of barium hexaferrite powders synthesis

A precursor solution prepared by introducing $Fe(NO_3)_3 \cdot 9H_2O$ into aqua DM while stirring using a magnetic stirrer for 10 minutes then added $Ba(NO_3)_2$ added to the precursor solution, stirring constantly for 10 minutes. To the precursor solution was added a solution of chitosan 1% w/v as much as 10% v/v solution was continuously stirred for 15 minutes and then added a solution of 0.5% as much as : 5% v/v, 10% v/v and 15% v/v of the solution keep stirring for 30 minutes, then the solution is stirred using homogeneizer with rotational speed of 10.000 rpm in order to form a homogenous solution. After that the solution is condensed by drying in an oven at a temperature of 100°C for 48 hours. Dry the precipitate formed was taken and crushed using a mortar to obtain a powder. Powder is then calcined in the furnace at a temperature of 1000°C with waiting time for 2 hours to obtain a compound $BaFe_{12}O_{19}$. The powder was tested contents the crystals using a X-Ray Diffraction (XRD) Brand: Philips Analytical type: PW1710 BASED then photographed their morphology using a scanning electron Microscope

(SEM) brand JEOL-JSM-6510LV and last examined the nature of the magnetic using a Vibration Sample Magnetometer (VSM) Brand: OXFORD TYPE: VSM1.2H while the flow diagram of the experiment can be seen in Figure 1

RESULTS AND DISCUSSION

XRD analysis.

The X-Ray Diffraction (XRD) results on Figure 2 show that barium hexaferrite is successfully formed. X-ray diffraction pattern of powder synthesized $BaFe_{12}O_{19}$ shows the dominant phase heksaferit barium contained in the powder calcination results. Heksaferit barium phase is characterized by intensity peaks that are at the corners (2θ) as follows: 30.24; 32.11; 34.06; 37.02; 40.19; 42.33; 55.13; 56.52; 63.08 this is in accordance with the card Powder Diffraction File (PDF) No. 43-0002. The sample prepared using 5% starch (v/v) generated

BaFe₁₂O₁₉ (84 %) and small amount of hematite (16%) by intensity peaks that are at the corners (2θ) as follows : 24.01;

33.11; 35.57; 49.51 and 54.16 this is in accordance with the card Powder Diffraction File (PDF) No. 33-0664, prepared using 10% starch (v/v) resulted in 100% BaFe₁₂O₁₉.

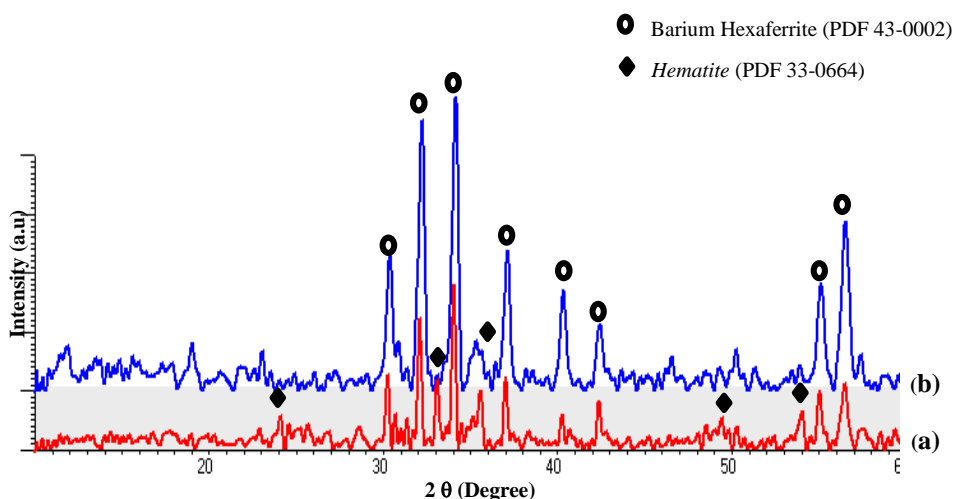


Figure 2. Diffraction pattern of barium hexaferrite, synthesized using starch
 (a) addition 5% starch (v/v); (b) addition starch 10 % (v/v)

Analysis-SEM

The images barium hexaferrite morphology can be seen in Figure 3. From this figure shows with the addition of chitosan morphology formed tends to decompose is caused by the dispersant, and with the addition of starch obtained rod-shaped morphology is due to the size of the template while the

morphology is likely to get increasingly thicker size thinning reaches the size of 62.5 nm, the number of rod-shaped morphology is still small compared with the number of hexagonal shape morphology this is most likely due to less amount of amylose than amylopectin. Thus Tapioca can be used as a template to obtain a nanorod morphology. Thus starch can be used as a template to obtain a nanorod morphology.

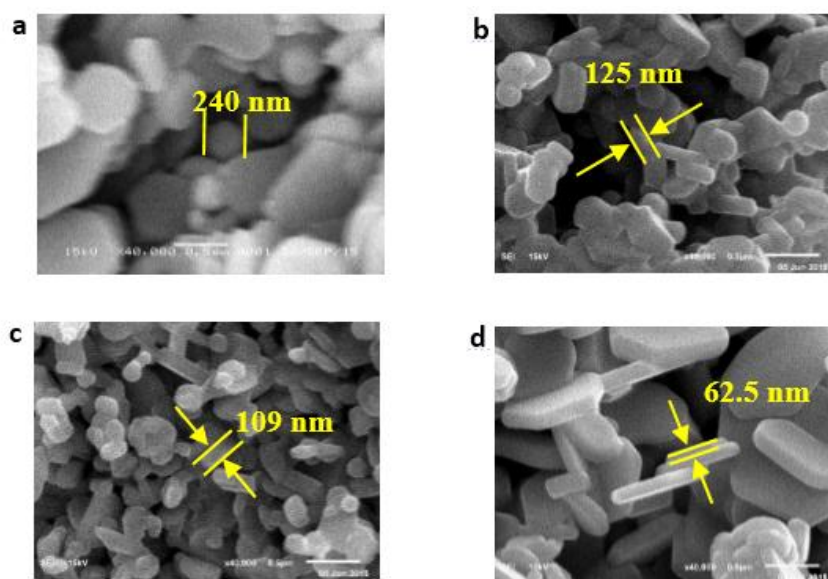


Figure 3. SEM images of barium hexaferrite, synthesized using various composition of starch.

(a) Without addition Chitosan & starch (0% (v/v)); (b) starch 5% (v/v); (c) starch 10% (v/v); (d) starch 15% (v/v).

The shape and size of the morphology can be seen in Table 1 below:

Table 1. Morphology Size of BaFe₁₂O₁₉

No	Starch (% (v/v))	Morphology	Thick (nm)	Length (nm)
a	0	Hexagonal & Granular	240 – 574	574 – 957
b	5	Rod & Hexagonal	125 – 775	500 – 625
c	10	Rod & Hexagonal	109 – 312,5	406 – 531,5
d	15	Rod & Hexagonal	62.5 – 140.6	406 – 1250

According to the table 1 morphology is a granular material formed, hexagonal and rods, using tapioca nanorod morphology is obtained, the higher the percentage of tapioca smaller particle size is obtained.

Analysis-VSM.

Results characterization using Vibration Sample Magnetometer (VSM) can be seen in Table 2, Figure 4 and Figure 5 below.

Table 2. Magnetic properties of BaFe₁₂O₁₉

No	Starch (% (v/v))	Saturation Ms (emu/g)	Remanence Mr (emu/g)	Coercivity Hc (tesla)
a	0	58.6	33	0.07
b	5	54	31.6	0.21
c	10	59.8	36.4	0.32
d	15	62.2	37.5	0.28

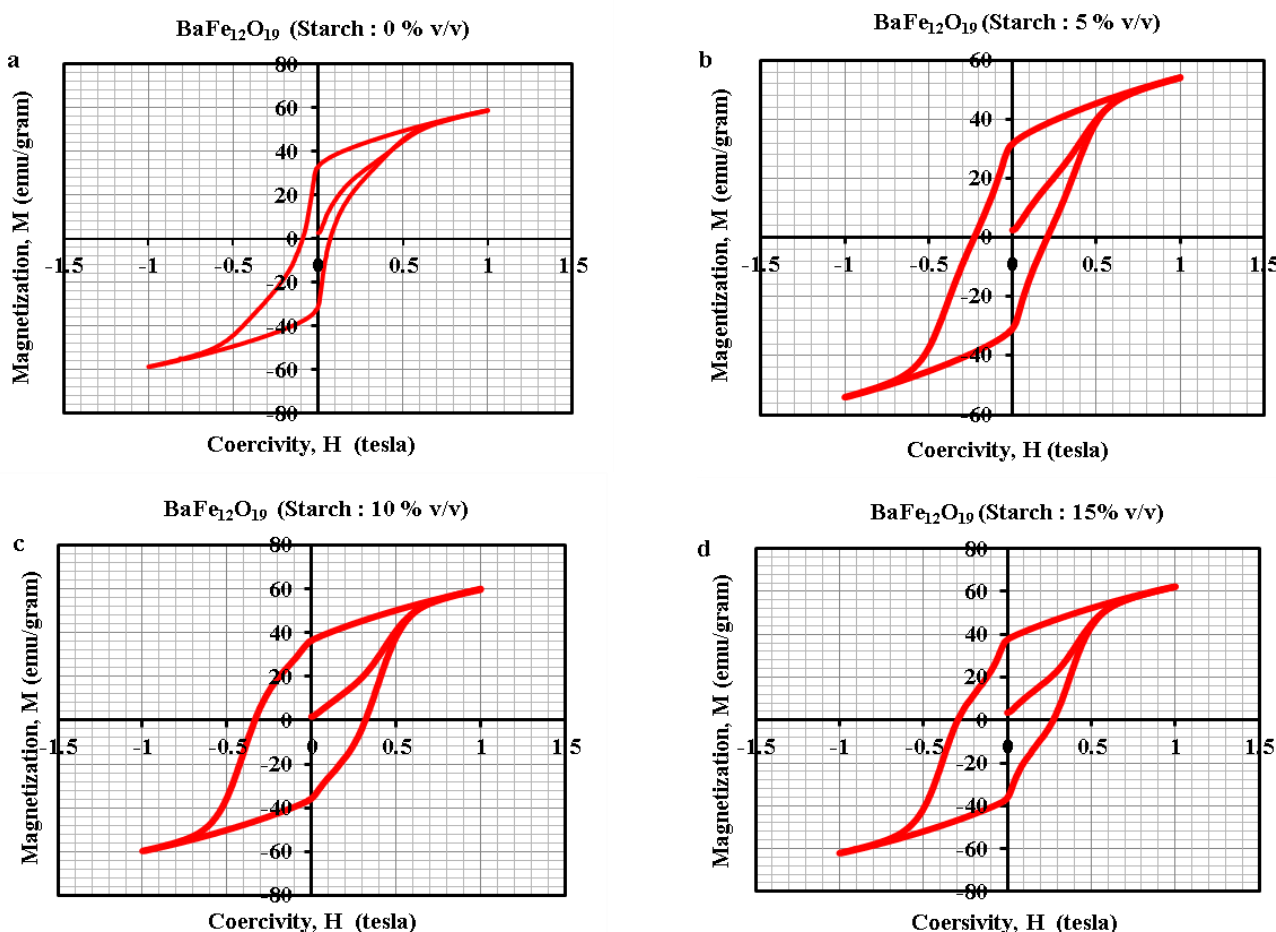


Figure 4. Hysteresis curves of Barium Hexaferrite, synthesized using various composition of starch (a). Without addition starch (0% (v/v)); (b) starch 5% (v/v); (c) starch 10% (v/v); (d) starch 15% (v/v).

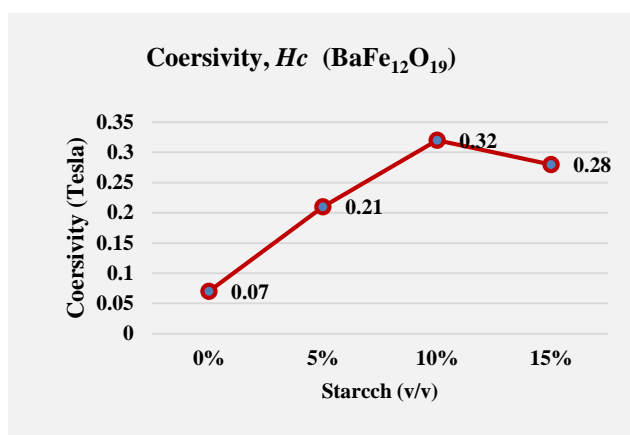
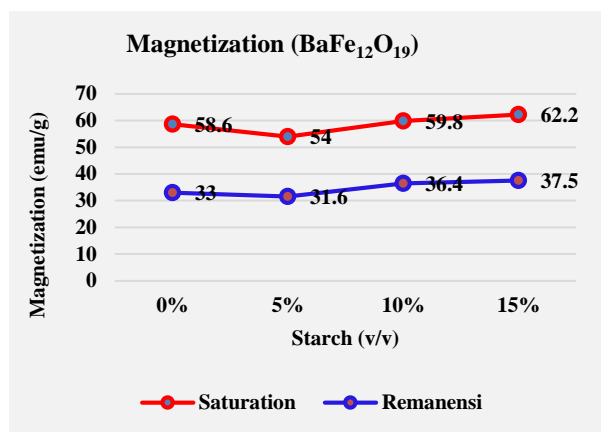


Figure 5. Magnetization of (BaFe₁₂O₁₉) at different uses Starch. (a) The magnetization BaFe₁₂O₁₉. (b) coersivity BaFe₁₂O₁₉.

Based on Table 2, Figure 4 and Figure 5 looks increasingly high percentage of starch that is used tends to increase the value of saturation and remanence and coersivity barium hexaferrite but koersifitasnya optimal value obtained on the use of stach 10% v/ v. it demonstrates the use of tapioca can enhance the magnetic properties of barium hexaferrite. Based on morphological forms (SEM results) are nanorod morphology, mean amylose derived from tapioca starch content can serve as a template rod thus nanorod morphology in BaFe₁₂O₁₉ powder can improve magnetic properties. It mendakan BaFe₁₂O₁₉ synthesis using a template from tapioca starch can improve magnetic properties, while the use of chemical process (sol-gel) and bio-process (use of natural template) will have low energy and low cost.

Magnetic properties (Ms and Mr) the results of this research is still small compared with the results of Xu et al (2014) but larger than the results Cao et al (2010); Galvao et al (2014) and Septiadi and Purwasmita (2014) while the magnetic properties (Hc) the results of this research is still small compared with the results of Cao et al (2010); Galvao et al (2014) and Xu et al (2014) but larger than the research Septiadi and Purwasmita (2014).

Synthesis of previous research that produces barium hexaferrite nanorod morphology generally use a synthetic template

(ethylene glycol, ethylene glycol, propylene glycol and α -FeOOH), this research could produce nanorod morphology using a template from natural materials in order to raise the local content of the tapioca starch.

CONCLUSION

Material formed results of this study are based on test results hexaferrite Barium X-Ray Difrraction. Synthesis of barium hexaferrite can produce hekasadecimal morphology, grain and nanorod through the process of using Sol-gel method by adding 1% chitosan solution as a dispersant and 0.5% Starch solution as a template. Barium hexaferrite magnetization value obtained increased after use tapioca as a template that is characterized by the formation nanorod morphology. Pnelitian results were also obtained significant increase in coersivity value in use of starch 10% v/v. For further research is expected to increase the amount of amylose are used in a manner that reduces the number of existing amolopektin the tapioca so as to multiply the number of nanorod morphology is formed it is expected to further increase the value of the magnetization of barium hexaferrite.

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