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Synthesis of Nanorod $Ba(Nd)_{0.015}Fe_{12}O_{19}$ Using Starch as Template

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Abstract

A synthesis of barium hexaferrite doped Nd atom based sol-gel method using chitosan solution as a dispersant and a starch solution as a natural template was performed. As Precursors used $Ba(NO_3)_2$, $Fe(NO_3)_3.9H_2O$ and $NdCl_3.6H_2O$ with mole ratio (1: 12:0.015). The results of the test using x-ray diffractometer showed that the phase formed is barium hexaferrite. Scanning electron microscopy photographs showed the morphology obtained in the form of: hexagonal, granules and nanorod with size also vary according to the amount of starch solution used ie: without hexagonal-shaped morphology and grain size (39.5-289.5) nm, length (118.4-447.4) nm, the use of starch contained nanorod morphology of size also varied in thickness (90.9-393.9) nm, length (393.5-1121.2) nm. Hysteresis curve of sample test result using Vibrating Sample Magnetometer showed that magnetism of Ba $(Nd)_{0.015}Fe_{12}O_{19}$ namely: suturation, remanence and coercivity of tends to increased in addition of starch solution. Significant coercive enhancement occurred with 0.5% (wight per volome) starch ingestion of 5% (volume per volume). Thus the use of starch can produce the morphology of $Ba(Nd)_{0.015}Fe_{12}O_{19}$ nanorod while increasing the magnetic properties of Ba(Nd) $_{0.015}Fe_{12}O_{19}$ that is saturation and remanensi. The increase in saturation and remanence is not as significant as the increase in coercivity is probably due to the small number of nanorods formed.

Keywords: Barium Hexaferrite, Sol-gel, Chitosan, Starch.

INTRODUCTION

Barium hexaferrite is one of the most widely used magnetic materials and still promising because it has superior properties such as: high coercivity, low production costs, high curie temperature, chemical stability and corrosion resistance. [1-5]. Barium hexaferrite is a type M hexagonal ferrite M. The M-Ferrite molecule consists of block S and block R, overlapping hexagonal shape and cubic shape [6], thus hexagonal ferrite is also called hexaferrite. Magnetic material (barium hexaferrite) is widely applied in industries such as: magnetic recording, data storage devices, microwave devices, and permanent magnets. Barium hexaferrite is also said to be ceramic magnet because it consists of metal and nonmetallic elements. Molecular-based ceramic magnets have all the characteristics commonly associated with metal-based magnets (based or atoms), for example, hysteresis and remanence, saturation magnetization and coercivity. [7]. Some of the results showed barium hexaferrite morphology shaped: hexagonal [2], [8-11], and there are shaped granules [12-16]. The magnetic field present in barium ferrite-shaped morphology of a rod will be larger than the magnetic field present in granular-shaped morphology [17].

In this study using Neodymium (Nd) as doping. The use of Nd has increased significantly in the last three decades after the discovery of a neodymium-boron-iron alloy that makes a very strong permanent magnet [18]. Although the numbers are limited, neodymium plays a major role in the high-strength permanent magnet on the base of the $Nd_2Fe_{14}B$ alloy. Permanent magnets exhibit outstanding performance in the electronics industry in applications such as electric motors, computer hard drives and powerful electric generators, capturing the attention of wind turbine manufacturing aspects and increasing efficiency [19].

One method that is widely used in the manufacture of barium hexaferrite is the sol-gel method. The sol-gel method applies a wet-chemical process. The sol-gel method can reduce the temperature on the process and can control the homogeneity of the solution [20]. In the sol-gel method can be added dispersant and template materials. Use of dispersants to minimize morphological size while templates serve to obtain the desired morphological form. One natural template obtained from starch. Starch contains amylose and amylopectin is a polymer that can function as a template.

A good dispersant for iron oxide nanoparticles is Chitosan [21]. Chitosan can act as a structural dispersant because it contains hydroxyl and amino functional groups capable of binding metal ions. Chitosan is obtained from deacetylation of chitin which is a biosourced polysaccharide [22]. The main source of chitin comes from shrimp or crab shells [23].

Amylose and amylopectin are the two glucose polymers present in starch [24], amylopectin polymer branched while linear amylose polymer. Amylose in starch may act as a nanorod template. The use of chitosan as dispersant and starch as a template on the synthesis of barium hexaferrite-based sol-gel can produce barium hexaferrite morphology of nanorod [25]. The synthesis of this research used sol-gel method to obtain morphology of barium hexaferrite nanorod doped Nd atom in order to improve magnetic properties of Ba $(Nd)_{0.015}Fe_{12}O_{19}$. This research is a continuation of previous research [25].

EXPERIMENT

Precursors include: Barium nitrate ($Ba(NO_3)_2$, 99.9%, Merck), Iron (III) Nitrate nonahydrate ($Fe(NO_3)_3.9H_2O$, 99.9, Merck) and $NdCl_3.6H_2O$ with molar ratio 1: 12 : 0.015 based on stoichiometry. Acetic acid (CH_3COOH, 99.8%, Merck) was used to dissolve chitosan, demineralized water (aqua DM) was used as a precursor solvent. Preferably prepare a 1% weight per volume (w/v) low molecular chitosan solution by dissolving 1g of chitosan in 100 ml 2% acetic acid solution) then prepare 0.5% weight per volume (w/v) starch solution by dissolving 0.5 g starch is in 100 ml of aqua DM at 70^0C. The solution of the precursor was prepared by dissolving Iron (III) Nitrate nonahydrate into aqua DM stirred for 10 minutes, then added $Ba(NO_3)_2$ into solution Iron (III) Nitrate nonahydrate stirred continuously for 10 minutes, then the solution was added $NdCl_3.6H_2O$ stirred during 10 minutes. The solution of the precursor was added 10% volume per volume (v/v) solution stirred for 15 minutes and then added 0.5% (w/v) starch solution as much as: (5, 10 and 15)% v/v) was stirred for 30 minutes then stirred using a homogenizer with a rotational speed of 10.000 rpm for 5 x 5 minutes for the solution to be homogeneous. After that the solution was dried in an oven at 100°C for 48 hours to obtain xerogel, then the xerogel was crushed using mortar to obtain a fine powder. The fine powder is calcined in the furnace at a temperature of 1000^0C with a holding for 2 hours to obtain a barium hexaferrite phase. The powder was tested using Philips Analitycal PW1835 diffractometer to investigate mineral phases and SEM+EDS composition of the JOEL-JSM-6510LV Brand to observe the morphological structure and atomic composition. The magnetic properties of Ba(Nd) $_{0.015}Fe_{12}O_{19}$ powder were tested using Oxford VSM1.2H magnetometer. The experimental flow diagram can be seen in Fig.1.

RESULT AND DISCUSSION

XRD Results. Analysis

The result of X-Ray Diffraction (XRD) test can be seen in Fig. 2, the formed phase is barium hexaferrite as shown by the main peaks which appear at diffraction at 2θ angle at level 23.13; 30.84; 32,60; 34.46; 35,37; 37.29; 40,62; 42.73; 47.12; 50.47; 55.33; 56.68; 63.22 the XRD test apparatus is unable to show Nd crystals because of very small amounts.

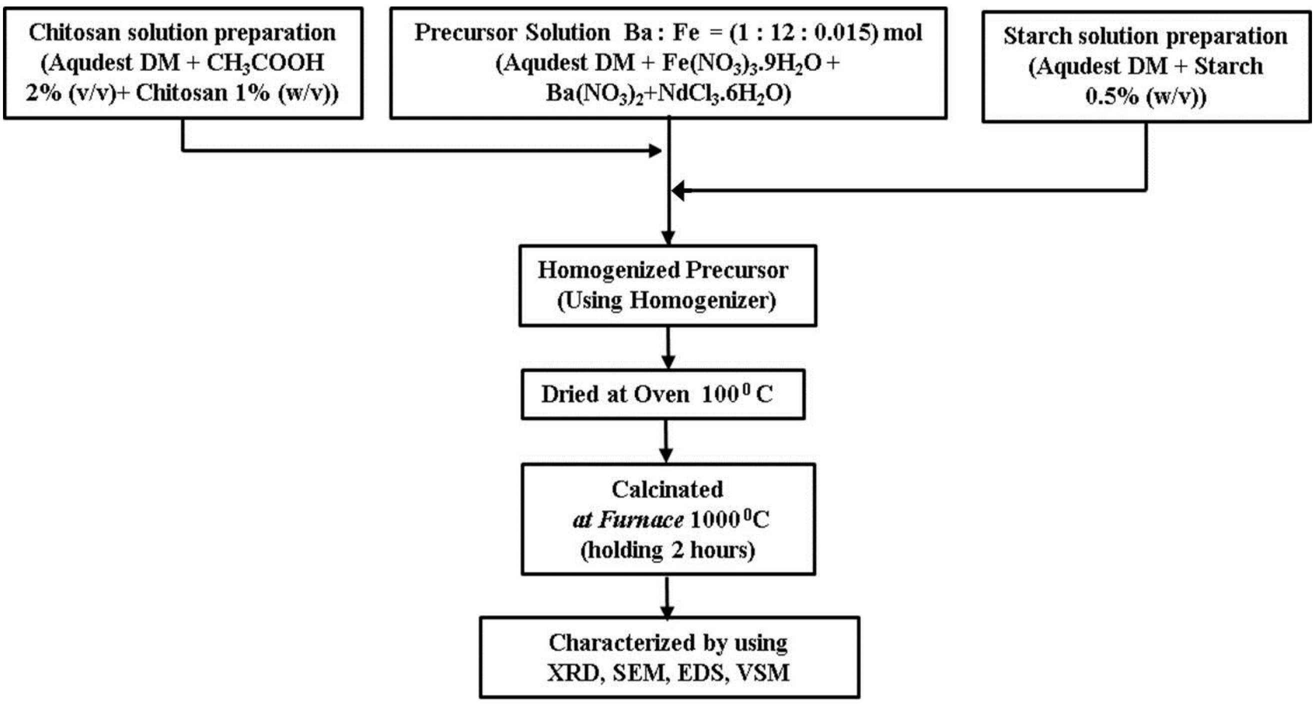

Figure 1: Flow chart Synthesis of $Ba(Nd)_{0.015}Fe_{12}O_{19}$

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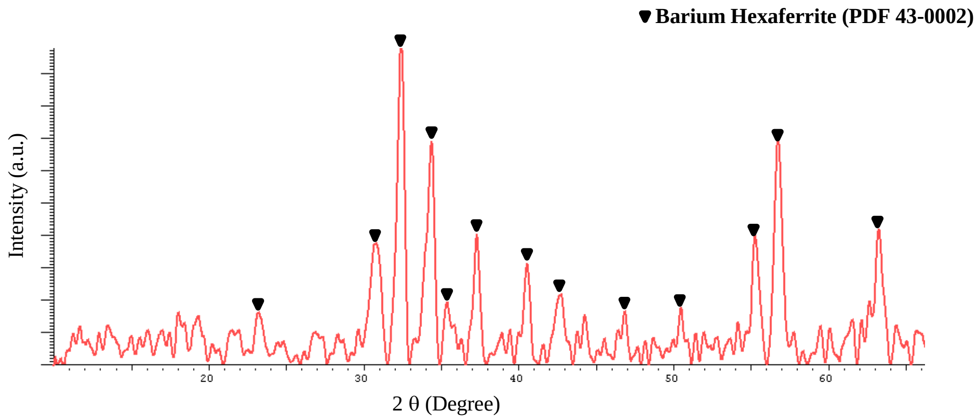


Figure 2: Diffraction pattern of barium hexaferrite, synthesized using starch

SEM Results Analysis

The morphological image of barium hexaferrite based on the results of Scanning Microscope Electron (SEM) can be seen in Fig. 3 and the size in Table 1. The addition of starch has nanorod morphology, and the morphology is obtained from rod-shaped caused by templates while the morphological size is likely to obtain a size diminished in size 62,5 nm, the number of stem-shaped morphologies is still less than the morphology of the hexagonal shape likely due to the lower amylose number of amylopectin. Metal ions in precursors (Ba2 + and Fe3 +) capable of binding the hydroxyl functional groups of amylose, form a linear structure. The amylose (starch).

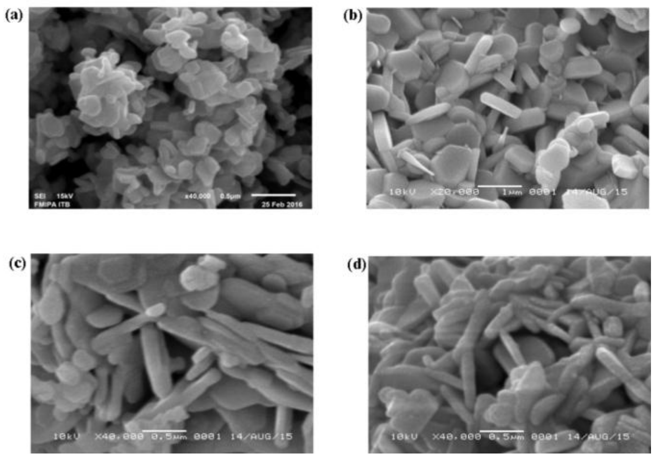


Figure 3: SEM photo sample Ba (Nd) 0.015Fe12O19, with different variations of starch composition

(a) starch (0% (v/v)); (b) starch 5% (v/v); (c) starch 10% (v/v); (d) starch 15% (v/v).

Table 1: Morphological Size $Ba(Nd)_{0.015}Fe_{12}O_{19}$

No	Pati (% (v/v))	Morfologi	Thick (nm)	Length (nm)
a	0	Hexagonal & Granules	39.5 – 289.5	118.4 – 447.4
b	5	Hexagonal & Granules & nanorod	90.9 – 393.9	393.5 – 1121.2
c	10	Hexagonal & Granules & nanorod	136.3 – 303.0	303.0 – 909.1
d	15	Hexagonal & Granules & nanorod	136.5 – 242.4	545.4 – 909.1

From Table 1. shows the rod-shaped morphology obtained by addition of starch.

EDS Results Analysis

Characterization using Energy Dispersive X-ray Spectroscopy (EDS) aims to determine the composition of the elements contained in the sample. The composition of sample elements based on sample characterization using EDS can be seen in Fig. 4 whereas the number of atoms in% can be seen in Table 2.

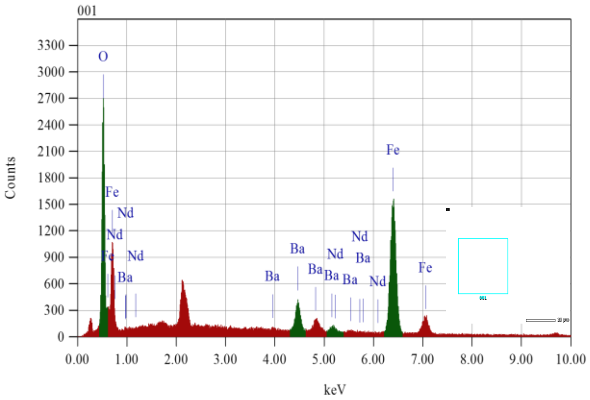


Figure 4: Composition of $Ba(Nd)_{0.015}Fe_{12}O_{19}$ sample elements based on EDS characterization results

Table 2: The Elements Contained in $Ba(Nd)_{0.015}Fe_{12}O_{19}$ Based on EDS Test Results.

No	Contained atoms	Amount (%)
1	Ba	3.24
2	Nd	0.30
3	Fe	34.00
4	O	62.46
	Total	100.00

VSM Results. Analysis

The result of characterization using Vibrating Sample Magnetometer (VSM) can be seen in Fig. 5. and Table 3

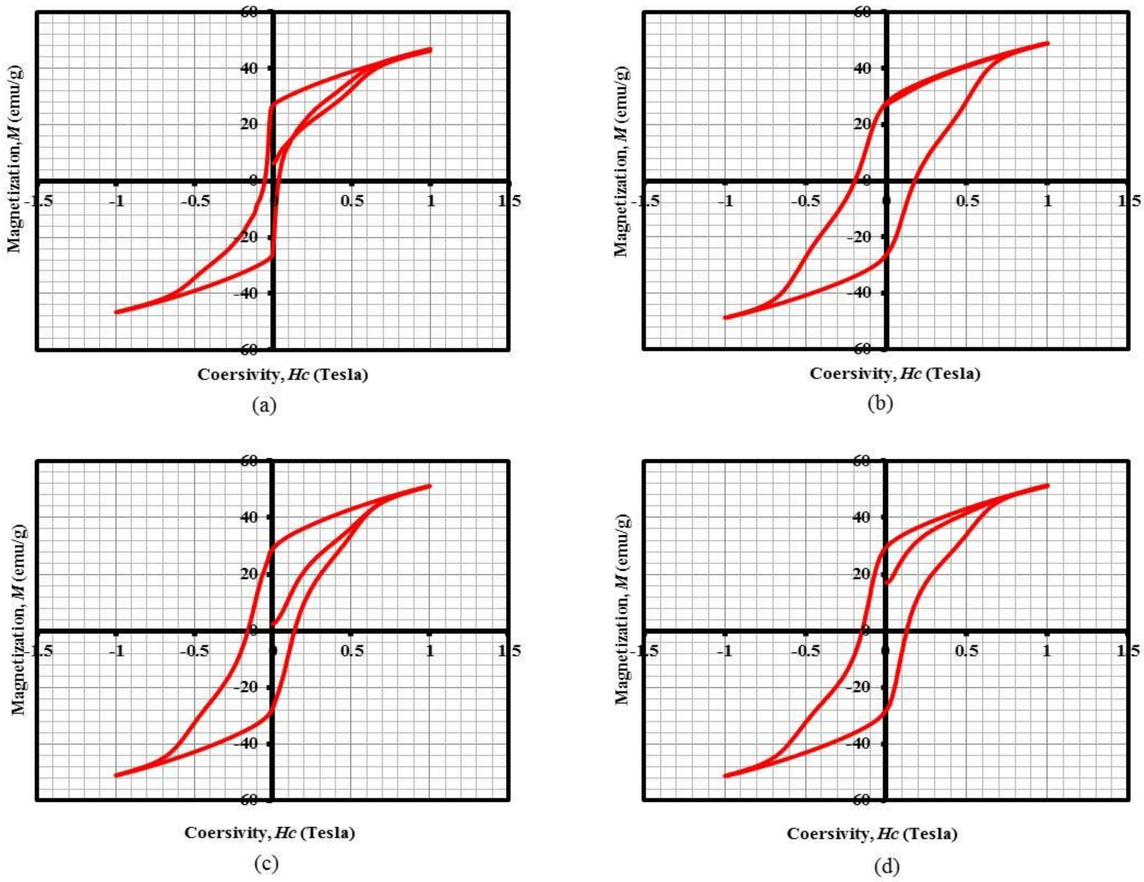
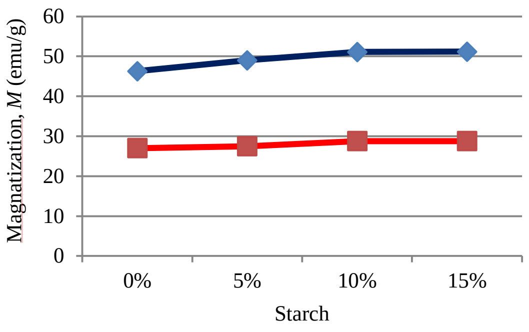


Figure 5: Hysteresis curve $Ba(Nd)_{0.015}Fe_{12}O_{19}$, the synthesis results using variations of starch composition.
(a) starch (0% (v/v)); (b) starch 5% (v/v); (c) starch 10% (v/v); (d) starch 15% (v/v).

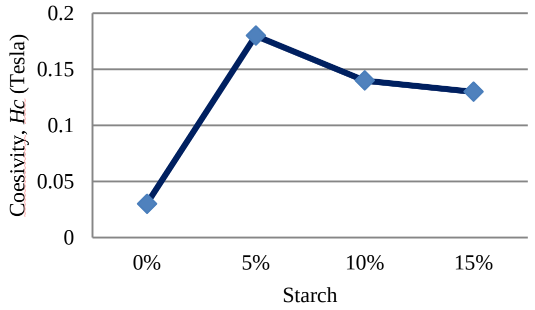
Table 3: Properties of Magnets $Ba(Nd)_{0.015}Fe_{12}O_{19}$

No	Starch (% (v/v))	Saturation *Ms* (emu/g)	Remanence *Mr* (emu/g)	Koercivity *Hc* (tesla)
a	0	46,3	27,0	0,03
b	5	49,0	27,5	0,18
c	10	51,1	28,8	0,14
d	15	51,2	28,8	0,13

Changes in magnetic properties of $Ba(Nd)_{0.015}Fe_{12}O_{19}$ based on variations in the use of starch graphically can be seen in Fig. 6.



(a)



(b)

Figure 6: Magnetic properties of Ba $(Nd)_{0.015}Fe_{12}O_{19}$ based on variations of starch usage

(a). Saturation (1) and remanence (2). (b) Coersivity

Based on Table 3, the magnetic properties of $Ba(Nd)_{0.015}Fe_{12}O_{19}$ (saturation, remanence) increased as the addition of the starch solution while the coercivity value increased significantly on the use of 5% starch solution (v/v) it showed that the use of starch can improve the magnetic properties of $Ba(Nd)_{0.015}Fe_{12}O_{19}$. The highest saturation value is 51.2 emu/g The highest remanence value is 28.8 emu/g and the highest coercivity value is 0.18 tesla. Results of magnetic properties obtained by the results of this study is still smaller when compared with the results of research S.B. Galvao et al [26] used ethylene glycol templates to produce 90 nm diameter nanorods and magnetic properties of 55 emu/g, coersivity 0.49 Tesla and Cheng-Yan Xu et al [27] using α-FeOOH producing nanorod 250 nm in diameter and saturation magnetic properties 62,5 emu/g, 0.38 Tesla coercivity but better than Samira Mandizadeh Q et al [5] using ethylene glycol template Propylene glycol producing nanorod 80-120 nm in diameter and magnet saturation 30 nm, coercivity 0.18 Tesla.

Previous research of barium hexaferrite synthesis that produces nanorod morphology using synthetic template (ethylene glycol, ethylene glycol Propylene glycol and α-FeOOH), this study actually use the template from natural materials to lift the local content of starch. The magnitude of $Ba(Nd)_{0.015}Fe_{12}O_{19}$ obtained is probably due to the small number of nanorods formed in addition to the magnetic properties also influenced by many factors, such as chemical composition, particle size, crystallinity degree, magnetic anisotropy, and so on. Nevertheless, the novelty of organic templates used derived from natural polymer starch has improved the magnetic properties of $Ba(Nd)_{0.015}Fe_{12}O_{19}$.

CONCLUSION

The material formed by this study is barium hexaferrite based on x-ray diffraction test results. Nanorod morphology was obtained after addition of starch solution as a template. The magnetic properties of $Ba(Nd)_{0.015}Fe_{12}O_{19}$ increased after using starch as a template characterized by the formation of nanorod morphology. The test results also obtained a significant increase in the coercivity value of 0.03 Tesla to 0.18 Tesla on the use of 5% starch (v/v) in the precursor solution. For further research is expected to increase the number of morphologies of nanorod such as by the addition of amylose in a way that reduces the amount of amylopectin present in the starch aims to further improve the magnetic properties of $Ba(Nd)_{0.015}Fe_{12}O_{19}$.

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