

## FTIR Spectrum of BiFeO<sub>3</sub> Ceramic Produced By Sol-Gel Method Based On Variation of Sinter and Calcination Treatment

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### ABSTRACT

High purity BiFeO<sub>3</sub> (BFO) powder was synthesized by sol-gel method using bismuth nitrate, iron nitrate as sources and citric acid as fuel. The calcination treatment of the citric acid gel was at 160°C and 200°C for 4 hours respectively. Sintering treatment was varied at 600°C for 4, 6 and 8 hours respectively. The powder has been characterized by Thermogravimetric Analysis/Differential Thermal Analysis (TGA/DTA) to know reference temperatures of calcination and sintering, X Ray Diffraction (XRD) test to confirm BFO phase and Fourier Transform Infra Red (FTIR) to identify chemical bonds in a molecule by producing an infrared absorption spectrum. TGA/DTA results show that loss of mass and heat flow is at 160°C approximately, it is used as reference of calcination temperature. XRD results show that the BFO powder sintered at 600°C for 8 hours exhibits no secondary phase, Bi<sub>2</sub>O<sub>3</sub>. Meanwhile, FTIR Spectrum shows that powder had single phase BFO is having wave number 1385 cm<sup>-1</sup> (C-H), 1560 cm<sup>-1</sup> (C-C) and 2930 cm<sup>-1</sup> (C-H).

**Keywords:** FTIR spectrum, Sol-Gel, Sintering, Calcination

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### I. INTRODUCTION

The production of single phase BiFeO<sub>3</sub> (BFO) ceramics presents many difficulties. The main ones are the presence of impurities in the final product, mainly Bi<sub>2</sub>O<sub>3</sub> and the difficulty to obtain dense ceramics with high resistivity. Powder obtained by conventional solid state reaction does not give ceramic materials with high densities. Bismuth ferrite (BFO) with a rhombohedral distorted perovskite structure exhibits antiferromagnetic ordering with high Neel temperature (643 K) and ferroelectric behavior with ferroelectric Curie temperature (1103 K) [1]. Due to this magnetoelectric coupling, BiFeO<sub>3</sub>-based systems could be applied in information storage, sensors, spintronic and microwave devices [2–4]. However, its practical applications are greatly hampered by the leakage current arising from impurities, defects or nonstoichiometry, which is mainly due to the difficulty in preparing pure BFO phase based on the kinetic and thermodynamic properties of the Bi<sub>2</sub>O<sub>3</sub>–Fe<sub>2</sub>O<sub>3</sub> system [5].

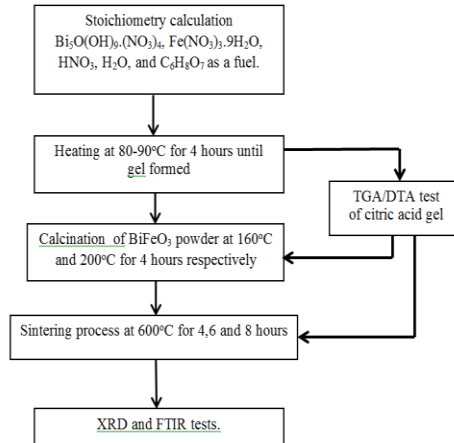
Wet chemical methods are a promising route to prepare fine and homogeneous powder. Various wet chemical methods such as hydrothermal [6], co-precipitation, combustion synthesis [7], molten-salt method [8], thermal decomposition, and sol–gel process [9,10] have been developed and designed to prepare pure BFO nanopowder. Recently, acid-assisted gel strategy has been proved to be an effective way to synthesize metastable BFO nanopowder. Pure BFO powder can be directly synthesized through the acetic acid-assisted or the tartaric acid-assisted sol–gel method.

However, BFO powder synthesized by the organic acid-assisted sol–gel method maybe has relatively low purity resulting from the easy formation of bismuth phase during calcining. Therefore, mineral acid should be considered as adjuvant to prepare BFO nanopowder. In the present paper, we report the synthesis of a pure BFO by sol-gel method using citric acid as a fuel. The method is more simple, energy saving, cost effective and requiring lower process temperature than other methods. Another advantages of using sol-gel method include: reagents required are simple compound, produces nanoparticles, no special equipment is needed, the elements of dopants could be easily incorporated into the final product, there is little possibility of agglomeration of particles, and uniform grain shape [11].

### II. METHOD

It is used basic compound pro analysis Merck product with a purity of 99.99% Bi<sub>5</sub>O(OH)<sub>9</sub>(NO<sub>3</sub>)<sub>4</sub>, Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O, HNO<sub>3</sub>, H<sub>2</sub>O and citric acid C<sub>6</sub>H<sub>8</sub>O<sub>7</sub> as a fuel to synthesize single phase of BiFeO<sub>3</sub> powder. The

all of basic compounds dissolved in aquabidestilate which was then heated on a hot plate at 80-90°C to form a gel (approximately for 4 to 5 hours). The gel formed was then heated in a furnace at calcination temperature of 160°C and 200°C for 4 hours respectively. The goal was to evaporate the water and the elements C, N and H. The powder obtained was then carried out by sintering process in the furnace at temperature of 600° C for 4,6 and 8 hours respectively. The flowchart of the synthesis was shown in Fig.1.

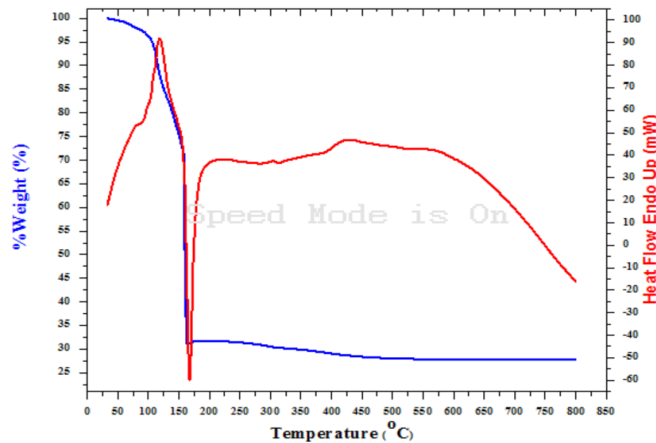


**Fig.1.** Synthesis of BiFeO<sub>3</sub> Using Citric Acid Gel

Testing by XRD were performed using an XRD PW 1835 Phillips type with diffraction angle of 20°-100° and using CuKα radiation. XRD test is used to confirm BFO phase. Characterization using Thermogravimetric Analysis (TGA) / Differential Thermal Analysis (DTA) aimed to observe changes in mass and heat of samples (still in gel form) to the increase in temperature, using a TGA / DTA Thermal Balance Research type LINSEIS L81-Series I / L81- STA (TGA-DTA). The data of TGA/DTA test result is used as temperature reference of sintering and calcination process. Fourier Trasform Infra Red spectroscopy is used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas.

**III. RESULTS AND DISCUSSION**

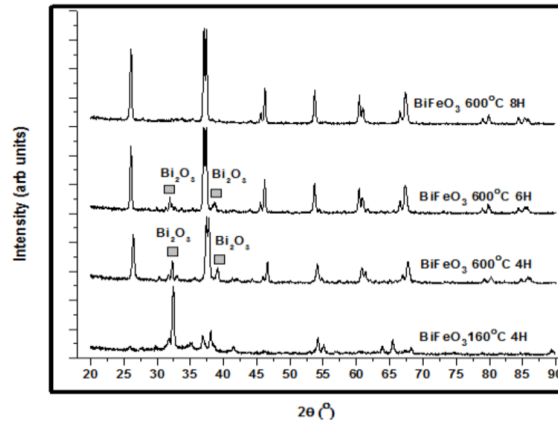
The process starts with heating the solution of bismuth nitrate, Fe nitrate, nitrate acid, H<sub>2</sub>O, and citric acid as a fuel until gel formed. After sintering it is produced BFO powder. To determine the temperature of sintering, it has been already done TGA / DTA test showed in Fig. 2.



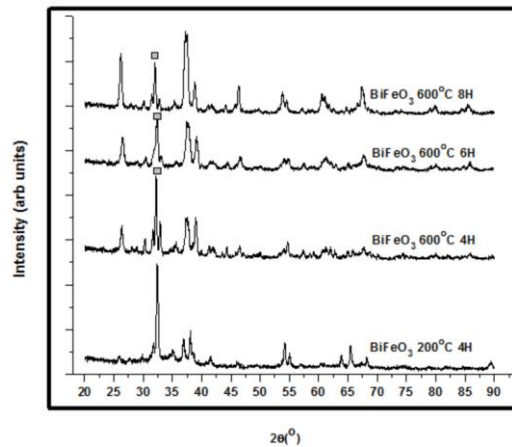
**Fig. 2** TGA/DTA Graphic of BiFeO<sub>3</sub> Gel

The result shows that phase transition occurs at 160-200°C approximately due to mass reduction (blue line) and increasing of energy (red line) at the temperature. The temperature is used as reference to calcination temperature. The next phase transition occurs at temperature of 550-600°C approximately. The temperature is used as reference to sintering temperature.

To confirm the formation of BFO phase, it is performed XRD test for all samples and the results are shown in Figures 3 and 4.

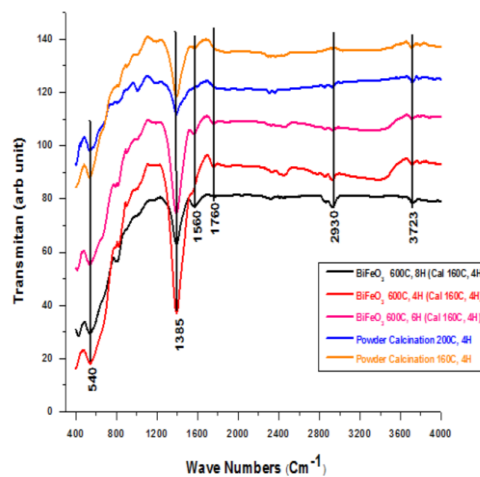


**Fig. 3** XRD Pattern of Powder Calcined at 160°C



**Fig. 4** XRD Pattern of Powder Calcined at 200°C

Figure 3 and 4 show that after calcination process at 160 and 200°C for 4 hours respectively the pattern has not had yet peaks of BFO. So then sintering process is carried out. Time of sintering that is applied varied at 600°C for 4,6 and 8 hours respectively. Figure 3 shows that powder having no impurity phase, Bi<sub>2</sub>O<sub>3</sub>, is of sintered at temperature of 160°C for 8 hours. Sintering for 4 dan 6 hours produce powder having impurity phase, Bi<sub>2</sub>O<sub>3</sub>. Meanwhile calcination at temperature of 200°C for all sintering conditions (600°C for 4, 6 and 8 hours) produce powder with impurity phase, Bi<sub>2</sub>O<sub>3</sub> with higher intensity. Producing phase-pure of BFO ceramic due to longer time of sintering and lower calcination temperature that causes crystallization perfectly and all Bi<sub>2</sub>O<sub>3</sub> has already oxidized to BiFeO<sub>3</sub>. The production of BiFeO<sub>3</sub> could also be confirmed by identifying chemical bonds in molecule by producing an infrared absorption spectrum with FTIR test as shown in Figure 5.



**Fig. 5** FTIR Spectrum of BFO Powder

Figure 5 shows that powder having no impurity phase has wave numbers 1385 cm<sup>-1</sup> (C-H), 1560 cm<sup>-1</sup> (C-C) and 2930 cm<sup>-1</sup> (C-H). Meanwhile powder with impurity phase, sintered at 600°C for 4 hours has no wave number 1560 cm<sup>-1</sup> (C-C), and sintered at 600°C for 6 hours has no wave number 2930 cm<sup>-1</sup> (C-H). Figure 5 also shows that powder calcined at 160°C for 4 hours has spectrum pattern identically with sintered at 600°C for 8 hours.

#### IV. CONCLUSION

Calcination process that produces BiFeO<sub>3</sub> powder with no impurity phase (after sintering at 600°C for 8 hours) is at temperature of 160°C for 4 hours. The powder has chemical bonds C-C and C-H. Meanwhile powder with impurity phase, Bi<sub>2</sub>O<sub>3</sub> only has chemical bonds C-H (sintered for 4 hours) and C-C (sintered at 6 hours) calcined at 160°C for 4 hours. Gel of BiFeO<sub>3</sub> calcined at 200°C for 4 hours has pattern of FTIR spectrum different from calcined at 160°C for 4 hours that could produce single phase of BiFeO<sub>3</sub> powder after sintering at 600°C for 8 hours.

#### ACKNOWLEDGMENT

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#### REFERENCES

- [1]. J.Way, J.B.Neaton, H. Zheng, V. Nagarajan, S.B. Ogale, B. Liu et al, *Science*, 299, 2003, pp 1719-1722.
- [2]. N.A.Spaldin, M. Fiebig, *Science*, 309, 2005, pp 391-392.
- [3]. D.S. Rana, I. Kawayana, K. Mavani, K. Takahashi, H. Murakami. M. Tonouchi, , *Adv Mater*, 21, 2009, pp 2881-2885.
- [4]. H. Bea, M. Gajek, M. Bibes, A. Barthelemy, *J.Physics : Condens Matter*, 20, 2008.
- [5]. M.Y. Shami, M.S. Awan, M. Anis-Ur-Rahman, *J.Alloy Compd*, 509, 2011, pp 10139-10144.
- [6]. L.J. Di, H. Yang, T. Xian, R.S. Li, Y.C. Feng, W.J. Feng, *Ceram Int*, 40, 2014, pp 4575-4578.
- [7]. J. Yang, X. Li, J. Zhou, Y. Tang, Y. Zhang, Y. Li *J.Alloy Compd*, 509, 2011, pp 9271-9277.
- [8]. Y. Liu, Q. Qian, Z. Yi, I. Zhang, F. Min, M. Zhang, *Ceram Int*, 39, 2013, pp 8513-8516.
- [9]. S. Dwita, W. Marlin, *ARPN Journal Engineering and Applied Sciences*, vol 11, No. 2, 2016, pp 901-905
- [10]. S. Dwita, W. Marlin, *ARPN Journal Engineering and Applied Sciences*, vol 11, No. 2, 2016, pp 891-895.
- [11]. S. Dwita, S. Bambang, H. Muhammad, *Applied Mechanics and Materials*, vol 493, 2014, pp 634-939.