

# Effect of Annealing on Structural and Morphological Properties of Bismuth Ferrite (BiFeO<sub>3</sub>) Nanoparticles

Thorat SM<sup>1</sup>, Kakade SB<sup>1</sup>, Mhaske SS<sup>1</sup> and Kalange AE<sup>1\*</sup>

<sup>1</sup>Department of Physics, Tuljaram Chaturchand College of Art's Science and Commerce, Baramati

\*Corresponding author: [kalangeashok@gmail.com](mailto:kalangeashok@gmail.com)

## Manuscript Details

Available online on <https://www.irjse.in>  
ISSN: 2322-0015

Editor: Dr. Arvind Chavhan

### Cite this article as:

Thorat SM, Kakade SB, Mhaske SS and Kalange AE. Effect of Annealing on Structural and Morphological Properties of Bismuth Ferrite (BiFeO<sub>3</sub>) Nanoparticles, *Int. Res. Journal of Science & Engineering*, 2023, Special Issue A12: 49-54.  
<https://doi.org/10.5281/zenodo.7802673>

Article published in Special issue of International Conference on "Recent Trends in Materials Science, Synthesis, Characterization and Applications (RTMS-2023)" organized by Department of Physics, Anekant Education Society's, Tuljaram Chaturchand College of Arts, Science and Commerce, Baramati, Dist Pune, Maharashtra, India (Autonomous) date, January 3-4, 2023.



Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons license, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons license and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this license, visit <http://creativecommons.org/licenses/by/4.0/>

## Abstract

Bismuth ferrite (BiFeO<sub>3</sub>/ BFO) nanoparticles with controlled particle size were synthesized via ethylene-glycol based auto combustion sol-gel method. The effect of the variation in the annealing temperature with time, on the structural and morphological properties of the sample was investigated. The as prepared samples were characterized by X-ray powder diffraction, to investigate the crystal structure and size of crystal. Different functional groups present in the sample were confirmed from Fourier transform infrared spectroscopy (FTIR). The surface morphology and elemental composition of the nanoparticles were investigated by scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS). The thermal decomposition of the sample was examined by Thermogravimetric analysis (TGA).

**Keywords:** Bismuth ferrite (BiFeO<sub>3</sub>/BFO), sol-gel method, nanoparticles

## Introduction

Bismuth iron oxide (BiFeO<sub>3</sub>/ BFO) is a single-phase multiferroic material which exhibit both the ferroelectric and antiferromagnetic ordering well above the room temperature [1]. Due to these unique properties, it has received a lot of interest in various fields such as magnetic storage, sensors, spinotronic devices etc [2]. However practical applications of BFO are restricted due to its low magnetization because of G-type antiferromagnetic nature below its Neel temperature. The ferroelectric value of BFO is also less as compared to many standard ferroelectric materials due to its high leakage behavior.

The high leakage behavior in BFO is due to the presence of defects like oxygen vacancies, variable states of Fe ( $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ ), Bi vacancy and formation of secondary phase during synthesis process [3]. Various syntheses methods like solid state [4], sol-gel [5], hydrothermal [6], soft-chemistry route [7] etc., are used for the formation of pure BFO. In BFO, the structural distortion can be induced by applying high pressure [8] or by doping [9] which in turn affects the multi ferroic properties. Instead of its multiferroic properties, BFO can be considered as visible light photocatalyst because of its

wide band gap energy ( $\sim 2.0 - 2.8$  eV) and good chemical stability [10]. Guo et al. had reported an improved photo catalytic activity in Gd-doped BFO nanoparticles by degrading rhodamine B (RhB) under visible light illumination [11]. Di et al. have synthesized  $\text{BiFeO}_3$  samples with different morphologies by hydrothermal method and functionalized BFO particles with Ag. They showed an improvement of RhB degradation by Ag-decorated BFO compared to non-decorated BFO under sunlight [12]. The potential use of BFO and doped BFO in the degradation of different types of organic dyes such as methylene blue [13], methyl orange [14] and congo red [15] are reported. In this article BFO nanoparticles are synthesized by auto combustion sol-gel method. The effects of annealing on the structural and morphological properties are investigated.

## Methodology

For the synthesis of  $\text{BiFeO}_3$  nanoparticles, the sol gel method is adopted. The starting materials used for the synthesis are bismuth nitrate ( $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ ), iron nitrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ) and ethylene glycol. All precursors are purchased from Loba Chemie (AR grade) and used without further purification. The typical procedure for the synthesis is as follows

Bismuth nitrate penta hydrate is dissolved in ethylene glycol is used as a solvent. The resultant mixture is stirred for 30 min. Further, the iron nitrate nonahydrate is added into the resulting homogeneous mixture and

stirred for 90 min at  $90^\circ\text{C}$  for the formation of gel. The ratio of Bi:Fe is 1:1. The formed gel is dried and annealed at  $600^\circ\text{C}$  for 6 hr and 8 hr respectively. The samples are further characterized by X-ray diffractometer in the range of  $20 - 80^\circ$  at the scan rate of  $5^\circ$  per min using Rigaku D/max with  $\text{Cu-K}\alpha$  anode ( $\lambda = 1.54056 \text{ \AA}$ ). Further, the confirmation of the sample is examined from Fourier transform infra-red spectroscopy (FT-IR). Scanning electron microscope (SEM) images are obtained using JEOL at an operating voltage of 120 kV.

## Result and Discussion

### 1. X-ray Diffraction (XRD)

The crystal structure and phase formation of  $\text{BiFeO}_3$  nanoparticles was identified by X-ray diffraction spectrometer (XRD) with  $\text{Cu-K}\alpha$  irradiation. Figure 3.1 shows the XRD patterns of as prepared BFO nanoparticles annealed at  $600^\circ\text{C}$  for 6 hr and 8 hr. From XRD patterns of BFO, it was observed that all the diffraction peaks from the sample prepared at  $600^\circ\text{C}$  corresponds to the reflections from different planes of rhombohedral BFO which are consistent with the reported values. (JCPDS No-01-071-2494) with space group R3c having lattice parameters as  $a = b = 5.5876 \text{ \AA}$  and  $c = 13.86 \text{ \AA}$ . This confirms the formation of perovskite BFO with rhombohedral crystal structure along with some impurities such as  $\text{Bi}_2\text{O}_4$  which are indicated by asterisks (\*) [16]. The presence of secondary phase is due to the kinetics of BFO formation in a narrow temperature range [17]. It was also observed that, the impurity phase gets reduced in the sample annealed at 8 hr.

### 2. Fourier Transform Infra-red spectroscopy (FT-IR)

In order to confirm the crystallinity and different functional groups present in the synthesized BFO sample using ethylene glycol as a solvent, the FTIR spectra was examined. Fig.3.2 shows, the FTIR spectra of BFO sample annealed at  $600^\circ\text{C}$  for 6 hr in the range of  $400-4000 \text{ cm}^{-1}$ . For the sample, the band range around  $400-600 \text{ cm}^{-1}$  corresponds to metal-oxygen bond and

confirms the existence of perovskite structure for the entire sample. The IR peaks below 1000  $\text{cm}^{-1}$  (such as 427.26, 558.75, 813,921)  $\text{cm}^{-1}$  were corresponding to the vibration's bonds of Bi-O or Fe-O respectively. The vibration of Fe-O at 427.26  $\text{cm}^{-1}$  and stretching vibration

O-Fe-O at  $\sim 558.75 \text{ cm}^{-1}$  is present in the octahedral  $\text{FeO}_6$  group [18]. The band present at 3457.51  $\text{cm}^{-1}$  is a result of antisymmetric and symmetric stretching of  $\text{H}_2\text{O}$  and O-H bond groups. A strong band at around 1412  $\text{cm}^{-1}$  is due to the trapped nitrates [19].

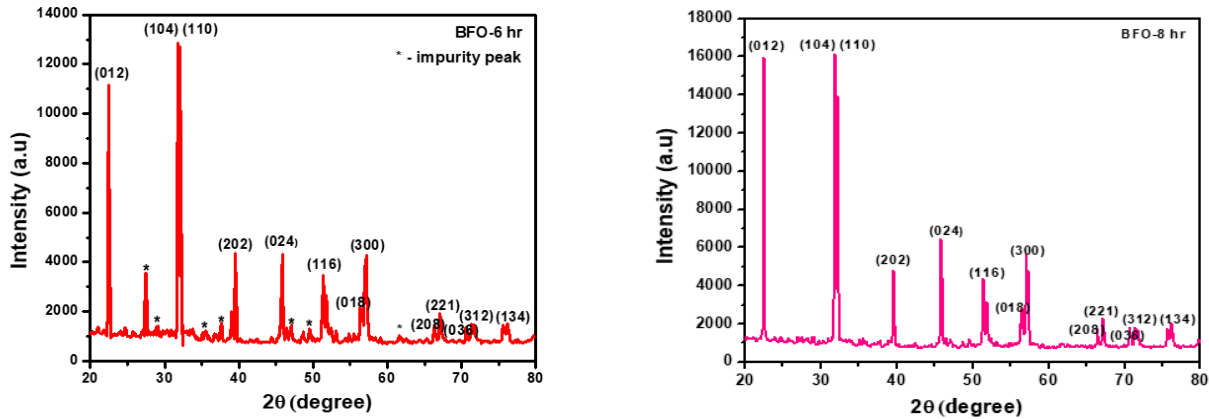


Fig 3.1 XRD pattern of BFO sample annealed at 600 °C for 6 hr and 8 hr

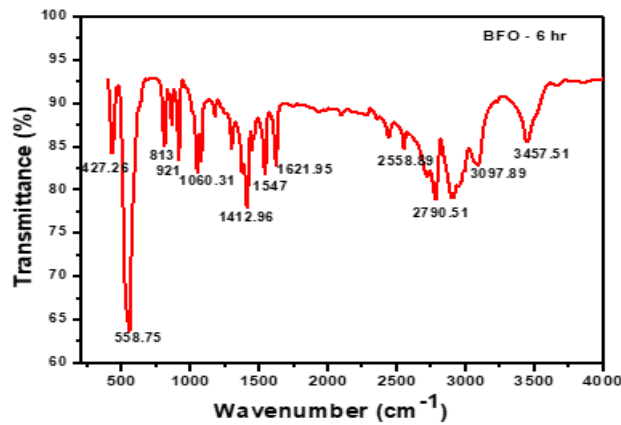


Fig 3.2: FTIR spectra of BFO annealed at 6hr

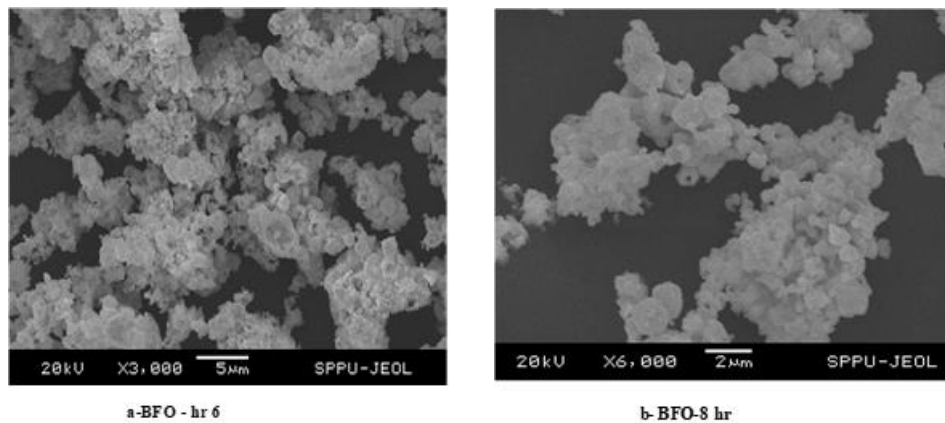


Fig 3.3 SEM images and EDS spectrum of BFO sample annealed at 600 °C a) 6 hr b) 8 hr

Spectrum processing :

Peak possibly omitted : 0.250 keV

Processing option : All elements analyzed (Normalised)

Number of iterations = 3

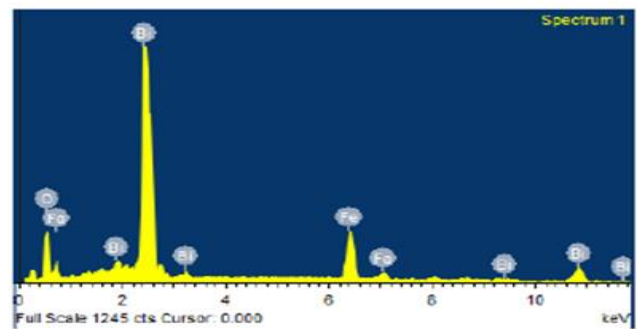
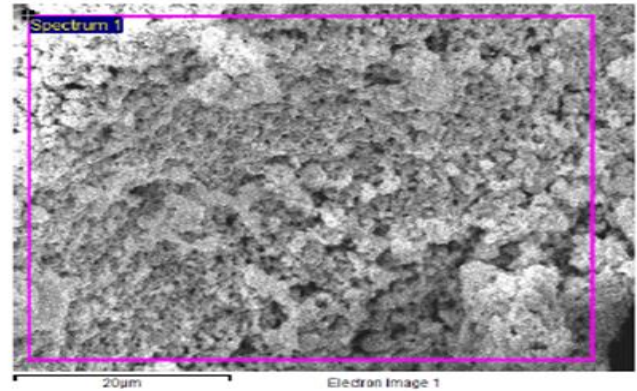
Standard :

O SiO2 1-Jun-1999 12:00 AM

Fe Fe 1-Jun-1999 12:00 AM

Bi Bi 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
O K	27.67	77.51
Fe K	11.87	9.53
Bi M	60.46	12.97
Totals	100.00	



a.EDS-BFO -6hr

Spectrum processing :

Peak possibly omitted : 0.252 keV

Processing option : All elements analyzed (Normalised)

Number of iterations = 3

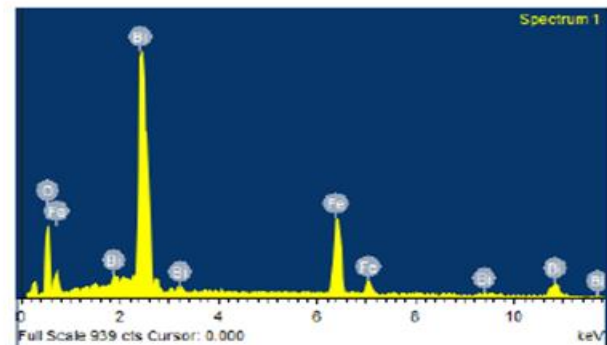
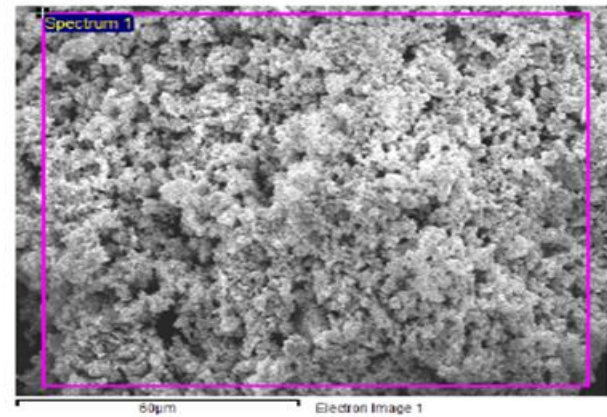
Standard :

O SiO2 1-Jun-1999 12:00 AM

Fe Fe 1-Jun-1999 12:00 AM

Bi Bi 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
O K	31.21	78.12
Fe K	16.55	11.87
Bi M	52.24	10.01
Totals	100.00	



b.EDS- BFO - 8 hr

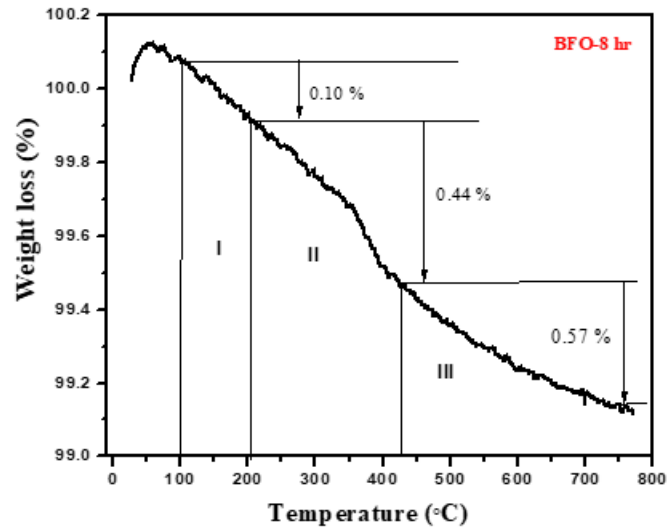


Fig 3.4 TGA curve of BFO sample annealed at 600 °C for 8 hr

### 3. Scanning electron microscopy (SEM) and EDS spectra

Figure 3.3 illustrates the SEM images and EDS spectra of BFO nanoparticles synthesized by sol gel method annealed at 600 °C for 6 hr and 8 hr respectively. From images, it is observed that the prepared particles have nearly spherical morphology as well they are in agglomerated form. The agglomeration of BFO is due to the magnetic nature of the BFO particles [20]. It was also observed that the particles are not uniformly distributed.

### 4. Thermogravimetric analysis

Thermo gravimetric analysis (TGA) was used to investigate the thermal degradation of the sample. From TGA curve, it is observed that 0.10% weight loss from 100 to 200 °C due to evaporation of adsorbed water from the sample. The weight loss from 200 to 750 °C is around 1% and it may be due to the loss of residual compounds after annealing, probably trapped nitrates (also confirmed from FTIR traces in 1300-1412.96  $\text{cm}^{-1}$  range, Figure 3.2) [21].

## Conclusions

The synthesis of  $\text{BiFeO}_3$  through the sol gel method resulted in the formation of highly crystalline BFO perovskite. An unexpected phase was identified in the sample annealed for 6 hr. and it may be attributed due to the pre-annealed precursor stoichiometry. SEM analysis indicates non uniform, agglomerated and nearly spherical distribution of the particles. The thermal degradation of sample shows  $\sim 1.5\%$  weight loss is observed around 100 -750 °C.

### Acknowledgments:

The authors acknowledge the facilities and technical support of the central facility center (CFC) and Department of physics, T. C. College, Baramati, Savitribai Phule Pune University. The authors also thankful to, 'Mahatma Jyotiba Phule Research and Training Institute,' (MAHAJYOTI), Nagpur for their financial assistance.

**Conflicts of interest:** The authors stated that no conflicts of interest.

## References

- Lee J. H, Oak M.A, Choi H, et al. Rhombohedral-orthorhombic morphotropic phase boundary in BiFeO<sub>3</sub>-based multiferroics: first-principles prediction. *J Mater Chem.* 2012;22:1667-1672
- Park T. J, Papaefthymiou G.C, Viescas A.J, et al. Size-dependent magnetic properties of single crystalline multiferroic BiFeO<sub>3</sub> nanoparticles. *Nano Lett.* 2007;7:766772.
- Dai H, Chen Z, Xue R, et al. Structural and electric properties of polycrystalline Bi<sub>1-x</sub>Er<sub>x</sub>FeO<sub>3</sub> ceramics. *Ceram Int.* 2013; 39:5373,5378.
- Valant M, Axelsson A-K, Alford N. Peculiarities of a solid-state synthesis of multiferroic polycrystalline BiFeO<sub>3</sub>. *Chem Mater.* 2007;19:5431, 5436
- Jiang Q.H, Nan C.W, Wang Y, et al. Synthesis and properties of multiferroic BiFeO<sub>3</sub> ceramics. *J Electro Ceram.* 2008;21:690693.
- Yang X, Xu G, Ren Z, et al. The hydrothermal synthesis and formation mechanism of single crystalline perovskite BiFeO<sub>3</sub> micro plates with dominant (012) facets. *Cryst Eng Comm.* 2014;16:41764182.
- Yang H, Xian T, Wei Z.Q, et al. Size-controlled synthesis of BiFeO<sub>3</sub> nanoparticles by a soft chemistry route. *J Sol-Gel Sci Technol.* 2011;58:238243.
- Gavriliuk A.Ge, Struzhkin V, Lyubutin ISe, et al. Phase transition with suppression of magnetism in BiFeO<sub>3</sub> at high pressure. *J Exp Theor Phys Lett.* 2005;82:224227.
- Yang C-H, Kan D, Takeuchi I, et al. Doping BiFeO<sub>3</sub>: approaches and enhanced functionality. *Phys Chem Chem Phys.* 2012;14:1595315962.
- Li S, Lin Y-H, Zhang B-P, et al. Photocatalytic and magnetic behaviors observed in nano structured BiFeO<sub>3</sub> particles. *J Appl Phys.* 2009;105:29032908.
- Guo R, Fang L, Dong W, et al. Enhanced photocatalytic activity and ferromagnetism in Gd doped BiFeO<sub>3</sub> nanoparticles. *J Phys Chem C.* 2010;114:2139021396.
- Di L.J, Yang H, Hu G, et al. Enhanced photo catalytic activity of BiFeO<sub>3</sub> particles by surface decoration with Ag nanoparticles. *J Mater Sci: Mater Electron.* 2014;25:24632469.
- Huo Y, Jin Y, Zhang Y. Citric acid assisted solvo thermal synthesis of BiFeO<sub>3</sub> microspheres with high visible-light photo catalytic activity. *J Mol Catal A: Chem.* 2010;331:1520.
- Liu Z, Qi Y, Lu C. High efficient ultraviolet photo catalytic activity of BiFeO<sub>3</sub> nanoparticles synthesized by a chemical co-precipitation process. *J Mater Sci: Mater Electron.* 2010;21:380384.
- Xu X, Lin Y-H, Li P, et al. Synthesis and photo catalytic behaviors of high surface area BiFeO<sub>3</sub> thin films. *J Am Ceram Soc.* 2011;94: 2296-2299.
- T. Liu, Y. Xu, and J. Zhao, "Low-Temperature Synthesis of BiFeO<sub>3</sub> via PVA Sol-Gel Route," *J. Am. Ceram. Soc.* 2010; 93 (11), 3637-41
- Qian F.Z, Jiang J.S, Guo S.Z, et al. Multiferroic properties of Bi<sub>1-x</sub>Dy<sub>x</sub>FeO<sub>3</sub> nanoparticles. *J Appl Phys.* 2009;106 (8):084312.
- T. Liu, Y. Xu, S. Feng, and J. Zhao, "A Facile Route to the Synthesis of BiFeO<sub>3</sub> at Low Temperature," *J. Am. Ceram. Soc.*, 2011; 94 (9), 3060-3.
- A. V. Zalesskii, A.A. Frolov, T.A. Khimich, A.A. Bush, *Physics of the Solid State*;2003, 45(1), 141.
- S. Chakraborty, M. Pal, *New J. Chem.* 2018, 42, 7188-7196.
- Xu, Y. B.Yuan, X.Huang, G. H.Long, H.; *Mater. Chem. Phys.* 2005, 90, 333.

© 2023 | Published by IRJSE

### Submit your manuscript to a IRJSE journal and benefit from:

- ✓ Convenient online submission
- ✓ Rigorous peer review
- ✓ Immediate publication on acceptance
- ✓ Open access: articles freely available online
- ✓ High visibility within the field

Submit your next manuscript to IRJSE through our manuscript management system uploading at the menu "Make a Submission" on journal website

<https://irjse.in/se/index.php/home/about/submissions>

For enquiry or any query email us: [editor@irjse.in](mailto:editor@irjse.in)