# Synthesis of Bismuth Ferrite nano particles by sol-gel method and their characterization

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**Abstract:** Sol-Gel method was used to synthesize crystalline Bismuth Ferrite ( $BiFeO_3$ ) nano particles BFO. For characterization and comparison, X-Ray diffractograms of  $BiFeO_3$  powder obtained by Sol-Gel method was taken before calcination and after calcination at 500° for 1 hour. Transmission Electron Microscopy (TEM) revealed a homogenous size distribution of nanometric Bismuth Ferrite perovskite powders with a grain size of 200 nm which is in well agreement with previous synthesis.

*Key Words:* Bismuth Ferrite, Nanoparticles, Sol- Gel method, X-ray diffraction(XRD), Transmission Electron Microscopy (TEM), Energy Dispersive X-Ray Profile (EDX).

# I. Introduction

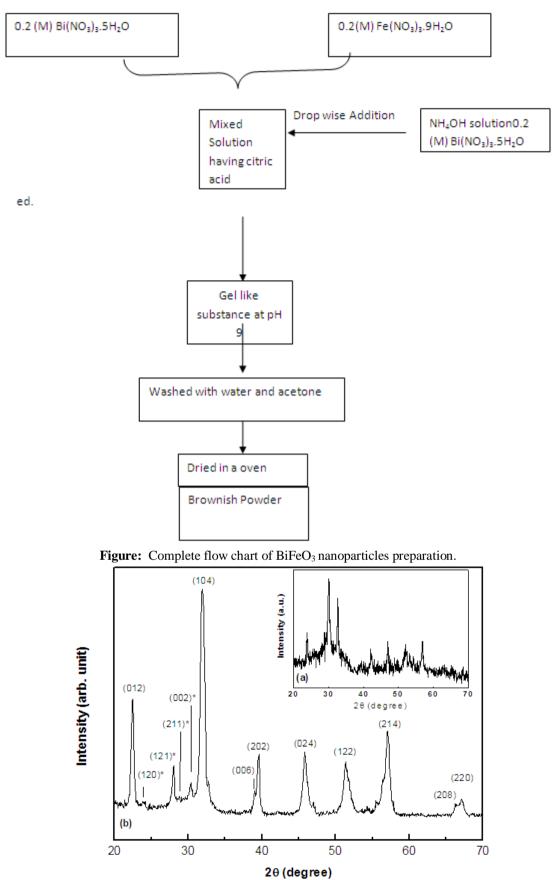
Bismuth Ferrite is one of the well known perovskite, widely investigated now-a days because of their simultaneous coexistence of ferroelectric and anti-ferromagnetic order parameters in perovskite structure, which are useful for applications is non linear optics, thin film capacitors, photo electrochemical cells, non volatile memories etc. Since its discovery in 1960, bismuth ferrite powders are being prepared by the solid state methods, mechano-chemical method, solution Chemistry methods, hydro thermal methods, sono chemical and sol-gel methods. Most of the mentioned methods require high temperature treatments greater than  $800^{\circ}$ . To get nano sized BFOs avoiding Bismuth volatilization, it is being developed gradually the low temperature synthesis methods. In the solid state method, nitric acid leaching was required to eliminate impurity phases such as  $Bi_2Fe_4O_9$  and  $Bi_{25}FeO_{40}$  after the calcinations of mixed Bismuth and iron oxides which produces coarser powders and has poor reproducibility.

In the present work BiFeO<sub>3</sub>(Bismuth Ferrite) nanoparticles were synthesized successfully by mixing 0.2 M Fe(NO<sub>3</sub>)<sub>3</sub> AND 0.2 M Bi(NO<sub>3</sub>)<sub>3</sub>.5H<sub>2</sub>O and adding Citric acid into them continuously. The nanoparticles synthesized were characterized using XRD and also the TEM (Transmission Electron Microscopy) images of BFO Nano Particles were taken to describe the grain size and surface morphology. Further Energy dispersive X-Ray(EDX) profile of BiFeO<sub>3</sub> nanoparticles after calcination at 500° C for 1 hour were taken.

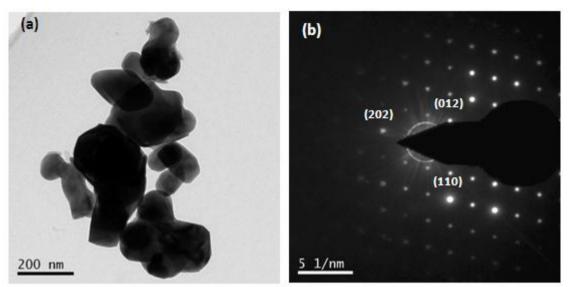
# II. Experimental Method

(i) **Chemicals Used:** Bismuth nitrate [Bi(NO<sub>3</sub>)<sub>3</sub>.5H<sub>2</sub>O], Ferric nitrate [Fe(NO3)<sub>3</sub>.9H<sub>2</sub>O], Citric Acid (ii) **Preparation of BiFeO3 materials by SOL-GEL method** 

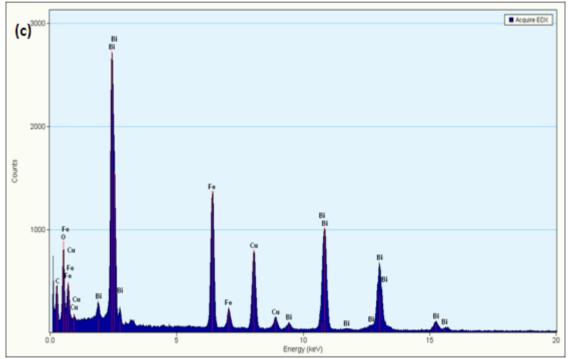
A stock solution of 0.2 M Bi(NO<sub>3</sub>)<sub>3</sub>.5H2O in aqueous medium was prepared. The 0.2 M Fe(NO<sub>3</sub>)<sub>3</sub> solution and 0.2 M Bi(NO<sub>3</sub>)<sub>3</sub> solutions were mixed in a 500 mL beaker under stirring condition. Citric acid was added cautiously. The ammonia solution was then added drop-wise into the mixed solution under continuous stirring condition and a light brownish gel was obtained.. The entire procedure is summarized in a flow diagram. The powders obtained from the above procedures were calcined at 500 °C so that the volatile matters like moisture and other unwanted components were removed.



X-ray diffractograms of BiFeO3 powder obtained by conventional precipitation method (a) before calcination and (b) after calcination at 500 °C for 1 h. The asterisks represent the Bi2Fe4O9 phase.



(a) TEM image and (b) corresponding selected area electron diffraction pattern BiFeO3 nanoparticles after calcination at 500 °C for 1 h.



Energy dispersive X-ray (EDX) profile of BiFeO3 nanoparticles after calcination at 500 °C for 1 h.

# III. Results and Discussions:

The synthesized BFO nanoparticles powder obtained by conventional precipitation method by XRD with Cu-K $\alpha$  radiation( $\lambda$ =0.154178nm) for their phase analysis studies before calcinations and after calcinations at 500<sup>o</sup>C for 1 hour. The asterriks in the X-ray diffractograms represent the Bi<sub>2</sub>Fe<sub>4</sub>O<sub>9</sub> phase. The prominent peaks in XRD plots are similar to various hkl planes of Bismuth Ferrite Nanoparticles thus confirming the synthesis. Besides, these prominent peaks some other peaks of low intensity were also observed, which definitely did not belong to BFO. The literature survey of various synthesis of BFOs indicated that at 500<sup>o</sup>C calcined BFOs the different peaks appeared were impurities related to Bi<sub>2</sub>.88Fe5O<sub>12</sub>.

The particle size estimated from TEM images for the BFO sample is about 200 nm. The corresponding selected area electron diffraction pattern of BFO particles are also shown after calcination at  $500^{\circ}$ C for 1 hour. The Energy dispersive X-Ray (EDX) profile of BiFeO<sub>3</sub> nanoparticles after calcination at  $500^{\circ}$ C for 1 hour has also been uniquely described.

#### **IV.** Conclusion:

In the performed experiment, Bismuth Ferrite nanoparticles (BiFeO<sub>3</sub>) are successfully synthesized by Sol-Gel method using Citric acid. The synthesized BFO particles were characterized by X-ray diffraction and TEM (Transmission Electron Microscopy). TEM analysis revealed the diameter of Bismuth Ferrite nanoparticles whereas XRD characterization indicates the rhombo-centred structure of Bismuth Ferrite nanoparticles. EDX (Energy dissipative X-ray) profile of BFO particles were also taken after calcinations at  $500^{\circ}$ C for 1 hour. This method doesn't use very high temperature for the synthesis such that Bismuth loss at high temperature is avoided.

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#### References

- [1]. Glenda Biasotto, A.Z.Simoes, C.R.Foschini, S.G.Antonio, Maria A.Zaghete, Jose A. Varela, "A novel synthesis of perovskite bismuth ferrite nanoparticles", Processing and Application of Ceramics 5(3)(2011)171-179.
- [2]. K.Y Yun, M.Noda. M. Okuyama, H.Saeki, H.Tabata, K. Saito, "Structural and multiferroic properties of BiFeO<sub>3</sub> thin films at room temperature", J.Appl. Phys., 96(2004)3399-3403.
- [3]. Y.P. Wang, L.Zhou, M.F. Zhang, X.Y.Chen, J.M.Liu, Z.G.Liu, "Room temperature saturated ferroelectric polarization in BiFeO<sub>3</sub> ceramics synthesized by rapid liquid phase sintering", Appl. Phys. Lett., 84(2004)1731-1734.
- [4]. S.Ghosh, S. Dasgupta, A. Sen, H. Shekhar, "Low temperature synthesis of nano sized bismuth ferrite by soft chemical route", J.Am.Ceram.Soc., 88(2005)1349-1352.
- [5]. C.Chen, J. Cheng, S.Yu, L.Che, Z. Meng, "Hydrothermal synthesis of perovskites bismuth ferrites crystallites", J.Crystal Growth, 291(2006)135-139.
- [6]. J.T. Han, Y.H Huang, X.J.Wu, C.L. Wu, W.Wei, B.Peng, W. Huang, J.B. Goodenough, "Tunable synthesis of bismuth ferrites with various morphologies". Adv. Mater. 18(2006)2145-2148.
- [7]. X.Qi, M. Wei, et.al., "High Resolution X-Ray diffraction and transmission electrons microscopy of multiferroic BiFeO<sub>3</sub> films, Appl.Phys.Lett.,87(2005)71913-71915.
- [8]. Yongming Hu, Lingfeng Fei, Yiling Zhang, Jikang Yuan, YU Wang, and Haoshuang Gu, "Synthesis of Bismuth Ferrite Nanoparticles via a Wet Chemical route at low temperature." Journal of Nanomaterials, vol.2011(2011), Article I.D.797639, 6 pages.
- [9]. S.M.Selbach, T.Tybell, M.AEinarsud and T. Grande, "Size dependent properties of multiferroic BiFeO<sub>3</sub> Nanoparticles", Chem. Mater., 19(26) 6478-84(2007).
- [10]. Anoopshi Johari, "Synthesis and Characterization of Bismuth Ferrite nanoparticles", AKGEC International Journal of Technology, Vol 2, No 2, Page No. 17-20.
- [11]. P. Fischer, M. Polomska, et. Al. "Temperature dependence of the crystal and magnetic structures of BiFeO<sub>3</sub>", Journal of Physics C, vol 13, No. 10, pp1931-40, 1980.
- [12]. W. Erenstein, N.D Mathur and J.F Scott, "Multiferroic and magnetoelectric materials", Nature, vol 442, no. 7104, pp 759-765.
- [13]. D.K Agarwal, "Microwave processing of ceramics", curr. Opin. Solid State Mater. Sci.3(1998)480-486.
- [14]. Y. Mao, T.J. Park, S.S. Wong, "Synthesis of classes of ternary metal oxide nanostructures", Chem. Commun., 46(2005)5721-5735.
- [15]. T. Kimura, T. Goto, H. Shintani, K. Ishizaka, T. Arima, Y. Tokura, "Magnetic Control of ferroelectric polarization", Nature, 426(2003)55-58.