Research Article **IJNR (2018) 2:5**

International Journal of Nanoparticle Research
(IJNR)

Synthesis & Structural Characteristics of ZnBi2O4 Nanoparticles Prepared by Citrate-Gel Auto Combustion Method

K. Vijay Kumar[1*] CH. Shilpa Chakra[2] K. Rama Krishna[3] A. Rajesham[4]

[1]Department of Physics, JNTUH, College of Engineering Sultanpur, Sultanpur(V), Pulkal(M), Sangareddy-Dist., 502 273, T.S., India; [2]Center for Nano Science and Technology, IST, JNTUH, Kukatpally, Hyderabad-500085, T. S., India; [3]Department of Physics, Malla Reddy College of Engineering &Technology, Secunderabad– 500 014, T. S., India; [4]Department of Physics, Sridevi Women's Engineering College, Vattinagulapalli, Hyderabad– 500 075Telangana, India

ABSTRACT

Zinc Bismuth nanoparticles with chemical composition of ZnBi2O4 is prepared by chemical citrate gel auto combustion method by using citric acid as fuel. The synthesized nano particles are sintered at different temperatures like 300^{O}C, 400^{O}C, 500^{O}C, & 600^{O}C for 4 hours in air medium. The single phase cubic structure is confirmed by X-ray diffraction, the average crystallite sizes of these samples are found to be in the order of 34 nm to 60 nm. The surface morphology of the samples is studied by using Scanning Electron Microscopy. The EDS spectrum confirms the presence of Bismuth, Zinc and Oxygen without any precipitating cations. FT-IR spectroscopy confirms the formation of spinel structure, the average particle sizes of the samples are measured by using DLS technique.

Keywords: Bismuth zinc nano particles, X-rd, SEM, EDS

***Correspondence to Author:**
K. Vijay Kumar
Department of Physics, JNTUH, College of Engineering Sultanpur, Sultanpur(V), Pulkal(M), Sangareddy-Dist., 502 273, T.S., India

How to cite this article:
Vijay Kumar, CH. Shilpa Chakra, K. Rama Krishna, A. Rajesham, Synthesis & Structural Characteristics of ZnBi2O4 Nanoparticles Prepared by Citrate-Gel Auto Combustion Method. International Journal of Nanoparticle Research, 2017; 2:5.

eSciencePublisher
eSciPub LLC, Houston, TX USA.
Website: http://escipub.com/

Introduction:

Nanoparticles are viewed as fundamental building blocks of nanotechnology. They are starting point for many 'bottom-up' approaches for preparing nano structured materials and devices.

Bismuth oxide as component finds use in wide applications in varsistors, catalyst and gas sensors. Bi_2O_3 based compounds are much better solid electrolytes than well-known stabilized Zirconia, because the face centered cubic (FCC) Bi_2O_3 exhibits the highest ion conductivity of all oxides conductors [1-3]

Mixed spinel oxides are complex metal oxides and have been an area of extensive study in recent years due to their significance in magnetic materials.

Mixed spinel oxides are represented by the chemical formula AB_2O_4 where *A*-denotes divalent cations occupying tetrahedral sites and B-denotes trivalent cations in octahedral sites [4]. Now a days, spinel oxides with the chemical formula MBi_2O_4 (where M stands for metal such as Zn, Cu, Co, Mg, Ni Fe etc.) has been the interest of many researchers because of their extraordinary properties [5].

It is a well-known fact that the intrinsic properties of ferrite materials are intensively influenced by the composition and the microstructure of ferrite particles, which are highly sensitive to the experimental conditions and preparation technique which is used for their synthesis. The wet chemical methods appear to offer an alternative way to control the composition and microstructures of the materials. There are generally various wet chemical methods for fabrication of ferrite nanoparticles such as co-precipitation method [6, 7], hydrothermal method [8, 9], sol-gel [10-11], spray-spin-heating-coating method [12]

In this communication, we report the synthesis of Zinc Bismuth nanoparticles with chemical composition of $ZnBi_2O_4$ is prepared by chemical citrate gel auto combustion method by using citric acid as fuel.

Materials and Methods:

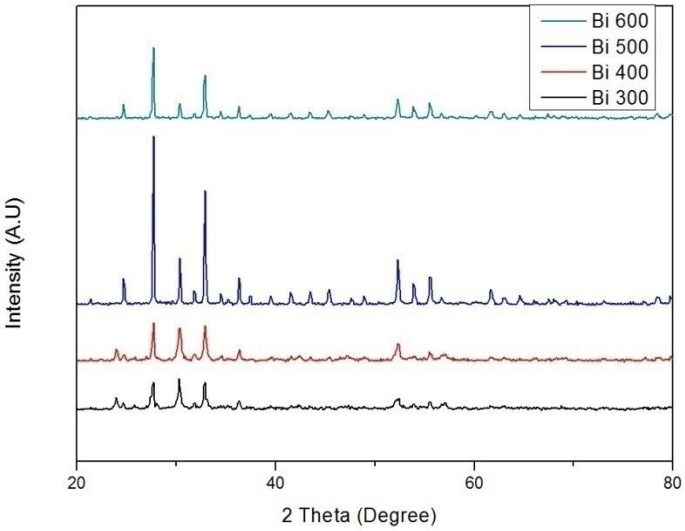
The starting materials used for the synthesis of Zinc Bismuth nanoparticles are Zinc nitrate, Bismuth nitrate, ammonia solution as buffer and citric acid as fuel for the synthesis of $ZnBi_2O_4$ by citrate gel auto combustion method.

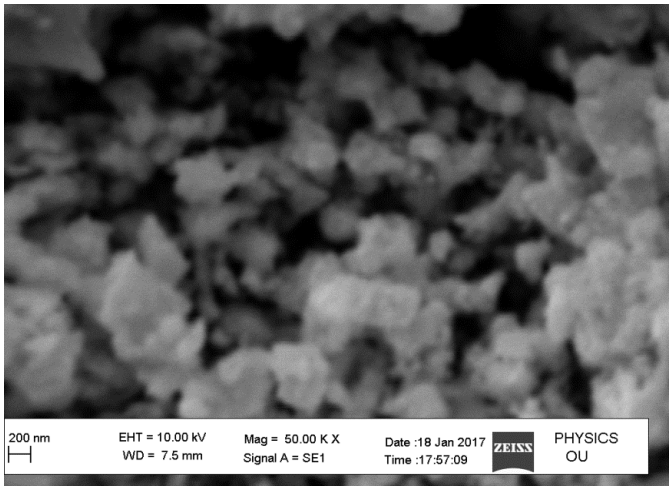
Stoichiometric amounts of the metal nitrates along with citric acid were dissolved in distilled water. Molar ratio of metal nitrates and citric acid was taken as 1:1 [13]. The resulting clear solution was mixed and heated at 80^OC with continuous stirring by using a magnetic stirrer with hot plate. The pH value of the solution was controlled at 7 by addition of ammonia drop by drop. The mixed solution was heated at about 100^0C with uniform stirring and evaporated to obtain a highly viscous gel. The resultant gel was further heated on a hot plate maintained at a temperature of 180^0C to 200^0C. The gel ignited on drying due to self combustion with the evolution of gases giving rise to a dark gray ash as product. This resultant ash powder samples were sintered for 4hours at different temperature like 300^OC, 400^OC, 500^OC, and 600^OC. The powdered materials were characterized by various techniques like powder x-ray diffraction for phase conformation, surface morphology by field emission scanning electron microscopy & energy dispersive spectroscopy (FESEM & EDS)(XL30). Dynamic light scattering (DLS) (HoribaSZ100) was performed on samples suspended in ethanol using a YD-laser (532 nm) and the mean value of the obtained histogram was considered the average particle size. Fourier transform infrared spectroscopy (FTIR) (Magna Nicolet550) was performed for wavelengths 300-500cm^{-1} to confirm the spinel formation.

Results and discussions:

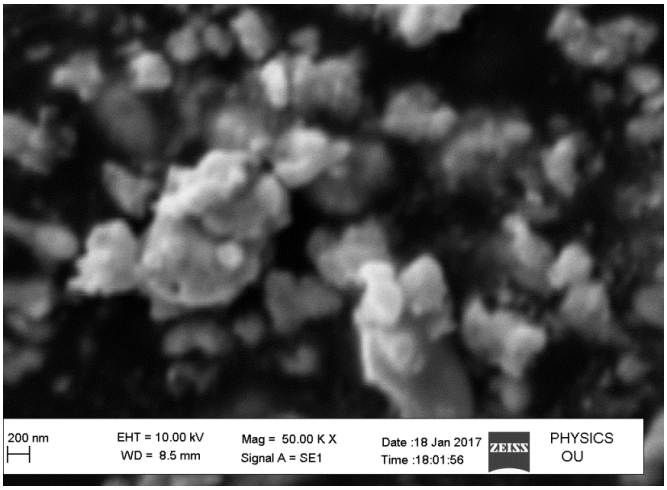
Structural analysis:

The structural characterization of $ZnBi_2O_4$ nanoparticles were carried out by using CuK_α radiation of wavelength 1.5405 A˚ at room temperature by continuous scanning in the range of Bragg's angles 5^O to 80^O in steps of 2^O/min to investigate the phase and crystalline

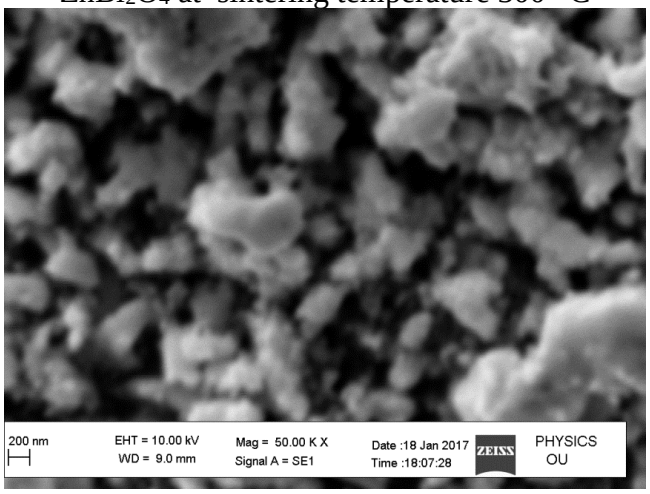




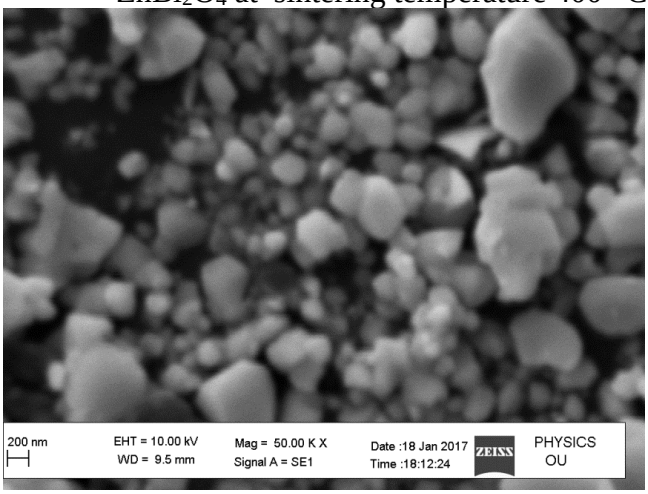
$ZnBi_2O_4$ at sintering temperature 300^O C



$ZnBi_2O_4$ at sintering temperature 400^O C



$ZnBi_2O_4$ at sintering temperature 500^O C



$ZnBi_2O_4$ at sintering temperature 600^O C

Fig.2. SEM images of $ZnBi_2O_4$ at sintering temperature

size. The recorded X-ray diffractograms, sintered at temperature 300^{O}C, 400^{O}C, 500^{O}C, and 600^{O}C are as shown in the Figure 1. This confirmed the formation of single phase structure.

The average crystallite size, was determined by using Debye Scherer's formula

$$t = \frac{0.9\lambda}{\beta cos\theta} \quad (1)$$

Where

'λ' is the wavelength of the X-ray used for diffraction

And

'β' is the full width half maximum (FWHM) in radians.

Surface morphology & particle size analysis:

The surface morphology of the $ZnBi_2O_4$ nano particles sintered at temperature 300^{O}C, 400^{O}C, 500^{O}C, and 600^{O}C was examined by FESEM and particle size by dynamic light scattering equipment. The surface morphology of the samples are as shown in Fig. 2, which indicates appearance of spongy porous like structure with the presence of The globule shaped particles were confirmed to be $ZnBi_2O_4$

The energy dispersive X-ray spectroscopy provided along with the FESEM confirms the presence of Zn, Bi and O elements without any precipitating cations. As evident from Fig.3

The particle size was evaluated by dynamic light scattering analysis, which reveals the average particle size is in the order of 34 nm to 60 nm at different sintering temperature like 300^{O}C, 400^{O}C, 500^{O}C, and 600^{O}C which is good agreement with particle size calculated by Debye Sherrer's formula. The particle size distribution is as shown in the Fig.4.

As the sintering temperature is increases from 300^{O}C to 500^{O}C the particle size is increases from 34 nm to 60 nm and then decreases to 40 nm at to 600^{O}C. The variation of particle size with temperature is as shown in the Fig.5.

The size of the particles is observed to be increasing linearly with sintering temperature. It appears that that increase in size with temperature becomes rapid between 300^{O} C-500^{O} C and appears to be slowing down at 600^{o} C. While annealing generally decreases the lattice defects and strains, however it can also cause coalescence of crystallites those results in increasing the average size of the nanoparticles [14]

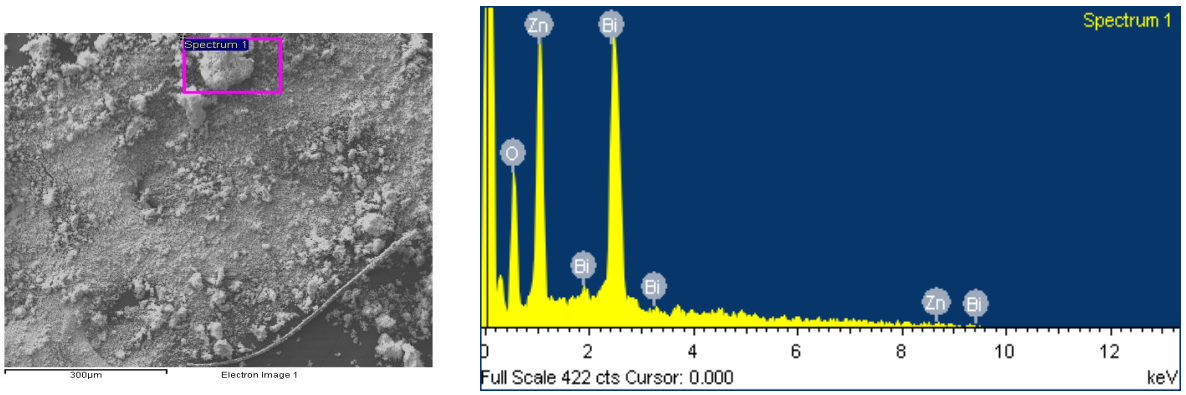
Fourier Transform Infrared Spectroscopy(FTIR) analysis:

According to Waldron [15], the spinel structure can be considered continuously bonded crystal, according to which, the atoms are bonded to all nearest neighbors by equivalent forces (ionic, covalent or Van der Waals). In spinel structure, the metals ions are situated in two different sub lattices designated tetrahedral (A-site) and octahedral (B-site) according to the geometrical configuration of the oxygen nearest neighbors. The IR spectra for the powder in the main form of $ZnBi_2O_4$ as prepared and annealed at different temperatures (300 ^{O}C, 400 ^{O}C, 500 ^{O}C and 600 ^{O}C) were recorded in the range of 500–3500 cm^{-1} are shown in Fig. 6. The absorption peak observed at low frequency within 500- 900 cm^{-1} confirms the vibration of metal oxide bond in the spinel structure. The band observed at around 578 cm^{-1} is the characteristic vibration of Zn-O bond in tetrahedral site, where as the band observed at 857 cm^{-1} is the characteristic of Zn-Bi bond at octahedral site.

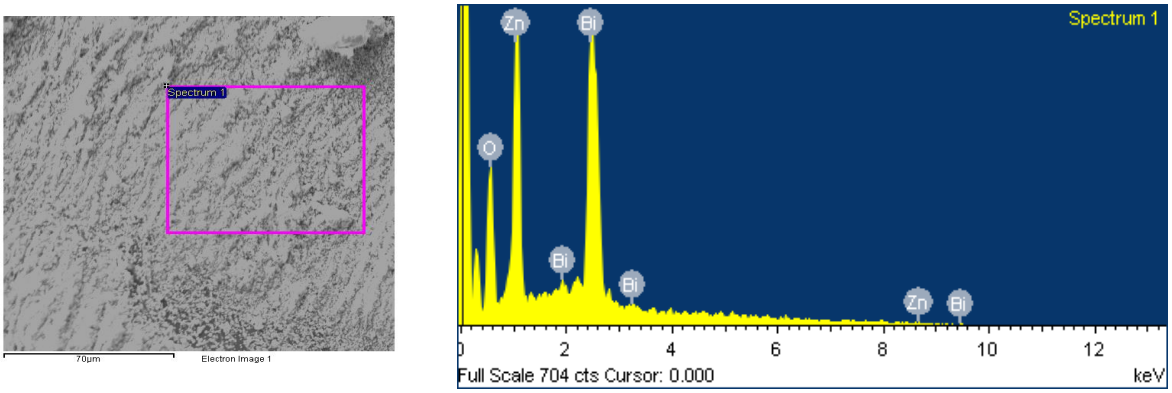
The absorption observed between 578 to 857 cm^{-1} confirms the formation of spinel structure of $ZnBi_2O_4$ nanoparticles.

Conclusions:

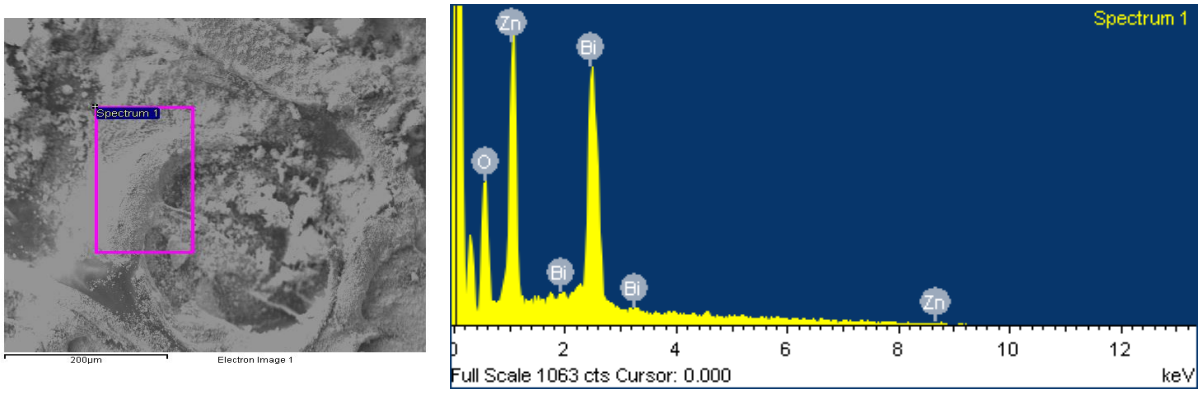
A series of Zinc Bismuth nanoparticles with chemical composition of $ZnBi_2O_4$ is prepared by low temperature auto combustion method and single phase were confirmed by XRD analysis. The surface morphology of the samples is studied by using Scanning Electron Microscopy with FESEM confirms the presence of Zn, Bi and O elements without any precipitating cations. The average particle sizes



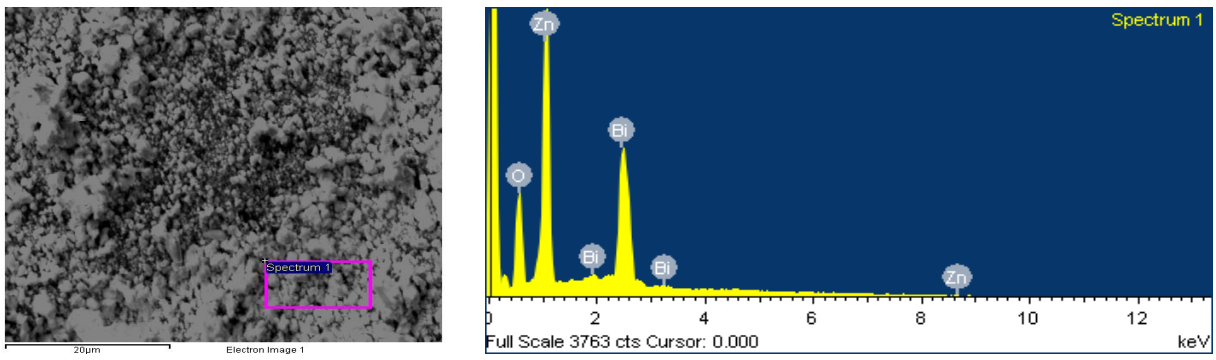
EDS Spectrum of $ZnBi_2O_4$ at sintering temperature 300^{O} C



EDS Spectrum of $ZnBi_2O_4$ at sintering temperature 400^{O} C

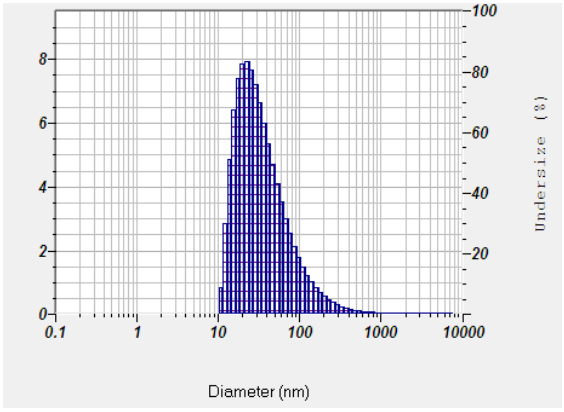


EDS Spectrum of $ZnBi_2O_4$ at sintering temperature 500^{O} C

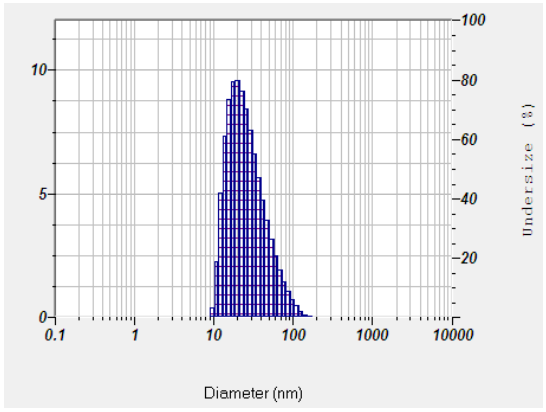


EDS Spectrum of $ZnBi_2O_4$ at sintering temperature 600^{O} C

Fig. 3. EDS spectrum of $ZnBi_2O_4$



Average particle size distribution of $ZnBi_2O_4$ at 500^OC



Average particle size distribution of $ZnBi_2O_4$ at 600^OC

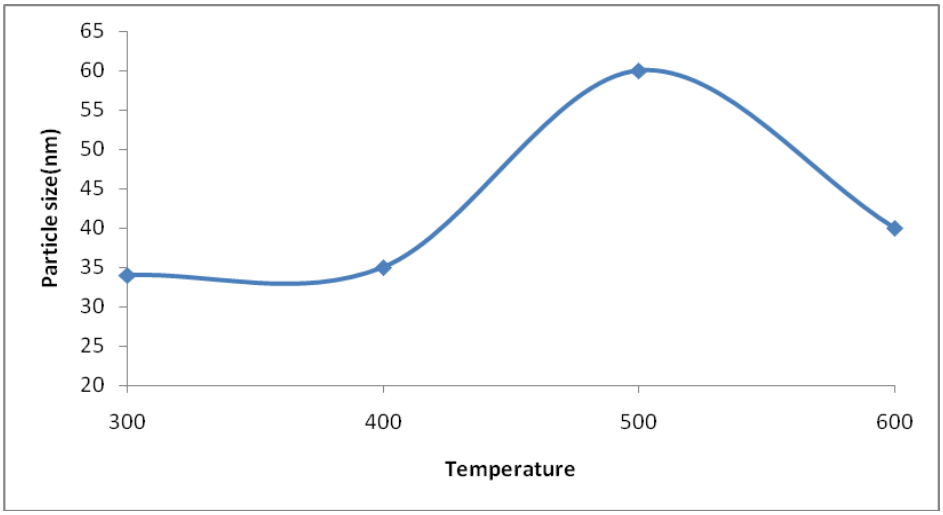


Fig.5. Variation of particle size with temperature.

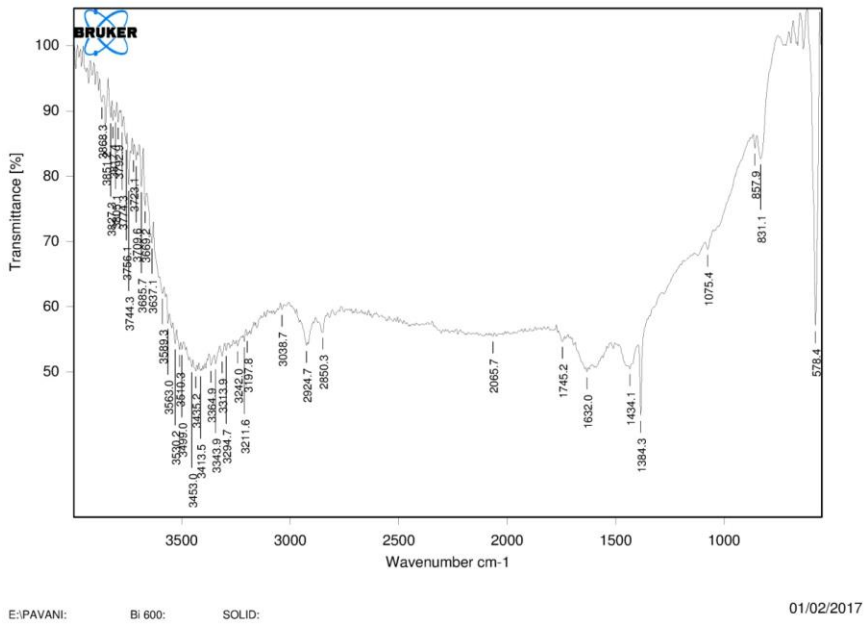


Fig. 6. FT-IR spectra of $ZnBi_2O_4$ sintered at 600^OC

of the samples are measured by using DLS technique and found to be in the order of 34 nm to 60 nm. The FT-IR spectroscopy confirms the formation of spinel structure

References:

[1] P. Suk, H.D.Wiemhofer, U. Guth, W. Gopel, M. Greenblatt, Oxide ion conducting solid Electrolytes based on Bi2O3, Solid State Ionics 89 (3–9) (1996) 179–196.

[2] M. Matsuoka, Nonohmic properties of zinc oxide ceramics, Jpn. J.Soc. Appl. Phys. 10 (6) (1971) 736–746.

[3] G.S. Devi, S.V. Manorama, V.J. Rao, and SnO2:Bi2O3 based CO sensor: laser-Raman, Temperatures programmed desorption and X-ray photoelectron spectroscopic studies, Sens. Actuators B 56 (1/2) (1999) 98–105.

[4] FanY, Lu X, Zhang H, Zhao L, Chen J. Destruction of poly chlorinated aromatic Compounds by spinel- type complex oxides. Environmental Science and Technology. 2010;44: 3079–84.

[5] Shuangming Chen, Yanfei Wu, Peixin Cui, Wangsheng Chu, Xing Chen, Ziyu Wu. Cation distribution in ZnCr2O4 nano crystals investigated by X-ray absorption fine structure spectroscopy. Journal of Physical Chemistry C. 2013; 117(47):25019–25025

[6] B. V Tirupanyam, Ch Srinivas, S. S. Meena, S. M. Yusuf, A. Satish Kumar, D. L. Sastry, and V. Seshubai, J. Magn. Magn. Mater., 392 (2015) 101-106.

[7] S. Dey, S. K. Dey, S. Majumder, A. Poddar, P. Dasgupta, S. Banerjee, and S. Kumar, Physica B: Condensed Matter, 448 (2014) 247-252.

[8] Meng, Yuying, Deyang Chen, Yitao Sun, Dongling Jiao, Dechang Zeng, and Zhongwu Liu. Applied Surface Science, 324 (2015) 745-750.

[9] Anwar, Humaira, Asghari Maqsood, and I. H. Gul. J. Alloys Compd., 626 (2015) 410- 414.

[10] M. Hashim, S. Kumar, S. E. Shirsath, R. K. Kotnala, J. Shah, & R. Kumar, Materials

Chemistry and Physics, 139 (2013) 364-374

[11] Li, Dong-yun, Yu-kun Sun, Yang Xu, Hong-liang Ge, Qiong Wu, and Chong Yan, Ceram. Int., 41 (2015) 4581-4589.

[12] Liu M, Obi O, Lou J, et al., J Phys D: Appl Phys, 42 (2009) 045007.

[13] Zhenxing Yue, Ji Zhou,Longtu Li, Hongguo Zhang Zhilum Gui, J Magn Magn Matr 2000, 208, 55.

[14] T. P. Raming, A. J. A. Winnubst, C. M. Van Kats, P. Philipse, Journal of Colloid and Interface Science 249, 346-350, (2002).

[15] Waldron RD (1955) Phys Rev 99:1727