Original Research Paper

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Physics

SYNTHESIS AND CHARACTERIZATION OF BISMUTH FERRITE NANOPARTICLES

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ABSTRACT A multiferroic material, Bismuth Iron Oxide (BiFeO3, BFO) has been synthesized by sol – gel method. The prepared samples were characterized by X-ray diffraction of powder (XRD), UV analysis, Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDAX). The surface morphology studies confirm the growth of bismuth ferrite nanoparticles with their diameters in the range of 81nm. The XRD analysis concludes the rhombocentered structure of synthesized nanoparticles. Topographical studies by SEM micrograph showed high level of agglomeration tendency in the sample and EDAX studies confirmed the elemental compositions in the sample. The BFO sample shows a decreasing trend of energy band gap Eg with increasing sintering temperatures was also observed. This absorption characteristics and narrow energy band gap has made pure phase BFO crystallites, a novel candidate in photocatalysis as well as photovoltaic applications. With change in synthesis conditions a transition from amorphous nature to crystalline behavior is observed.

KEYWORDS : Sol-gel technique, XRD, SEM, EDAX

INTRODUCTION:

Multiferroic materials are of particular interest due to the co-existence of ferromagnetic and ferroelectric properties [1,2]. Among various multiferroics, bismuth iron oxide ($BiFeO_3$, BFO) is the only material that shows both ferroelectric and antiferromagnetic properties at room temperature [3,4]. However, synthesis of phase pure BFO is difficult due to volatile nature of Bi_2O_3.Bismuth iron oxide ($BiFeO_3$, BFO) posses rhombohedral perovskite structure. Bismuth iron oxide is a potential candidate due to its high ferroelectric Curie temperature of 820˚C and antiferromagnetic Neel temperature of 370˚C. Magnetic properties in BFO are due to the presence of uncompensated spins at iron site. In BFO ferromagnetic coupling arises within the plane whereas, antiferromagnetic coupling arises between the spins of adjacent planes. Due to DM (Dzyaloshinsky-Moriya) interaction canted ferromagnetism arises. This is used in wide range of applications such as spintronics, data storage and magnetic sensor devices owing to their ferroelectric and ferromagnetic behavior [5,6]. In recent years, BFO NPs successfully tested for the degradation of triphenyl methane dyes and azo dyes due to their photocatalytic behavior.

Though BFO was discovered in 1960, recently there is a renewed interest because of its possible novel applications in the field of radio, television, microwave and satellite communications, audio-video and digital recording and, as permanent magnets. So far, bismuth ferrite powders have been prepared by Solid state reaction method, hydrothermal method, Chemical route method, Co-precipitation method and Autocombustion method. Most of the mentioned procedures need high temperature treatments (>800°C). Due to the requirement of nanosized oxides and in order to avoid bismuth volatilization the developing of low temperature synthesis methods is essential. Previous studies have demonstrated that synthesis of BFO NPs through a traditional solid-state method produces poor reproducibility and causes formation of coarser powders as well as Bi_2O_3/$Bi_2Fe_4O_9$impurity phase [7,8].

Synthesis of BiFeO3 materials by SOL-GEL method

AR grade of the starting materials Bismuth nitrate [$Bi(NO_3)_3.5H_2O$], Ferric nitrate [$Fe(NO_3)_3.9H_2O$], Acetic Acid, 2-methoxy ethanol and Ethylene glycol were used for synthesis of nanoparticles. The samples are prepared by sol – gel combustion route. The fuel chosen for the synthesis is mixture of 2 – methoxy ethanol and ethylene glycol. 2-methoxy ethanol is added with ethylene glycol and stirred. It is reported that, solvent with pH:1 results best prepared sample, so the mixture of fuels are added to acetic acid drop wise with continuous stirring till the pH meter reading showed pH:1. It was noted that 26.6 ml of acid is mixed with 50 ml of fuel mixture in order to obtain pH:1. This value is considered as standardization for other samples as well. After continuous stirring of the fuel and acid mixture, the stoichiometric amount of Bi(NO3)3 .5 H2O salt is added and stirred. Then Fe(NO3)3.9H20 salt is added to the mixture. Now the colour of the solution changes from colourless to brick red then red and finally blackish red. After continuous stirring the solution is heated with stirring at a temperature of 70˚C. After heating and stirring a fluffy gel is obtained with intense evolution of brown fumes. Now the gel is kept in hot air oven at 70˚. The powder collected is then calcined for 3 hours at a temperature of 600˚C. After cooling, the sample is collected from the furnace and is grinded by agate - mortar. The grinded powder is now ready for necessary characterization. The samples before calcinations and after calcinations are shown in Fig. 1(a) and 1(b).



Fig 1(a)BFO nanoparticles –Before Calcination



Fig 1(b)BFO nanoparticles –After Calcination

Characterization:

X-ray Diffraction (XRD) analysis:

X-ray diffraction analysis for the synthesized compound has been carried out to find the formation of the compound and confirmation of crystal structure. The powder XRD pattern of the Bismuth Iron Oxide is deployed in Fig.2

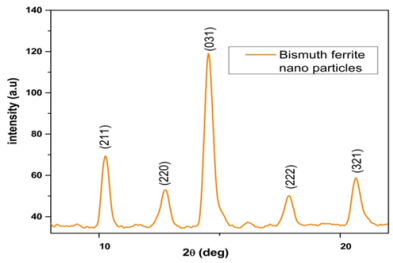


Fig 2Powder XRD pattern of BFO nanoparticles

The prominent peaks in xrd plot are indexed to various (hkl) planes of BFO, indicating formation of BFO. Besides these prominent peaks, some other peaks of low intensity are also observed, which do not belong to BFO. The average crystallite size of BFO was 420nm, determined using Scherrer equation with high intensity peak at 2θ = 14.86°. It is obvious from the XRD studies that BFO nanoparticles are highly crystallized and exhibit a single-phase perovskite structure. Non-perovskite phases such as $Bi_2Fe_4O_9$ and Bi_2O_3/Fe_2O_3 are not detected in XRD analysis. The commonly observed byproducts like $Bi_2Fe_4O_9$, $Bi_{24}Fe_2O_{39}$ located at 2θ =10.97° and α- and β-Bi_2O_3 formed by polynuclear species are removed and hence XRD studies proved the synthesized $BiFeO_3$ is highly crystalline in nature and is of high purity.

Scanning Electron Microscope (SEM):

The morphological studies of BFO were done by Scanning Electron Microscope, showing agglomerated particles with mean size of about 81 nm was observed. The SEM images of BFO nano particles at different magnifications are shown in Fig, 3(a) and 3(b). The agglomeration of BFO nanoparticles formed due to evaporation and has highly interconnected agglomerated particles with less than 100nm. Sol-gel techniques are adopted by Gautam et.al. gave agglomerated clusters of spherical particles. Also, it is found that solid state route provides polyhedral particles with reduction in particle size when doping concentration increases. Thus, it indicates that the agglomeration of BFO nanoparticles leads to decrease in particle size which is well in agreement with the previous reports [9,10].

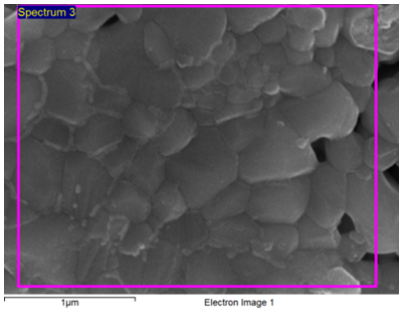


Fig 3(a)SEM image of BFO nanoparticles at 1µm magnification

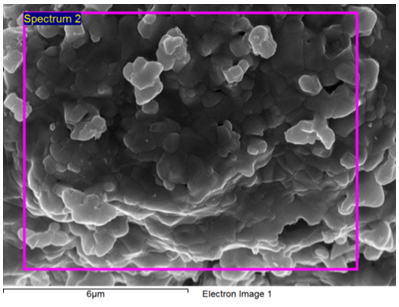


Fig 3(b)SEM image of BFO nanoparticles at 6µm magnification

Energy Dispersive Spectroscopy (EDAX):

Energy dispersive X-ray analysis (EDAX) is a micro-analytical technique for characterizing the elements present in the given sample. It uses the characteristic spectrum of X-rays emitted by the nanoparticle after excitation by high-energy electrons. EDAX gives the conformation of element percentage in the sample. The EDAX spectrum of BFO nanoparticles are shown in Fig. 4.

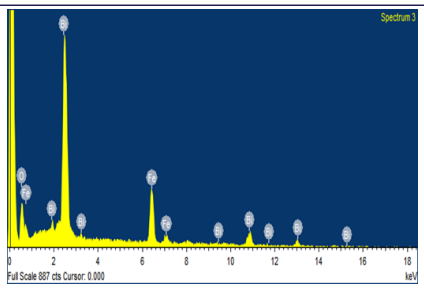


Fig 4EDAX image of BFO nanoparticles

CONCLUSION:

In the reported study, bismuth ferrite (BiFeO3) nanoparticles are successfully synthesized by Sol-Gel method. The sythesized bismuth ferrite (BiFeO3) nanoparticles were characterized by X-Ray Differaction (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDAX). The XRD characterization results indicate the rhombo centered structure of bismuth ferrite nanoparticles and the SEM analysis reveals that the diameter of bismuth ferrite (BiFeO3) nanoparticles increases with calcination temperature. The surface morphology studies confirm the growth of bismuth ferrite nanoparticles with their diameters in the range of 81nm.Topographical studies by SEM micrograph showed high level of agglomeration tendency in the sample and EDAX studies confirmed the elemental compositions in the sample.

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