

# **STUDIES ON MULTIFUNCTIONAL PROPERTIES OF DOPED MULTIFERROICS**

*Synopsis of the thesis submitted in fulfillment of the requirement for the Degree of*

## **DOCTOR OF PHILOSOPHY**

By

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## SYNOPSIS

Multiferroics are characterized by the presence of two and more switchable states, such as polarization, magnetization and strain [1-2]. Multiferroic materials are attractive not only due to information storage devices but also for their high electrical permittivity and high magnetic permeability. This property can be used to replace the inductor and capacitor in resonant circuits with a single component, which are further useful in miniaturizing portable cellular technologies. Strong coupling between the polarization and magnetization in multiferroics would allow these materials to be used in multistage data storage devices or switch the magnetic properties with an electric field and vice versa.

In general, magnetoelectric phenomenon exhibits coupling between magnetization and electric field or polarization and magnetic field. Induction of polarization (P) by applied magnetic field (H) or vice-versa has been observed long back in 1888 in an experiment carried out by Rongten where dielectric material gets magnetized when placed in an electric field [3]. Later in 1894, Curie recommended the possibility of magnetoelectric effect in some crystals [4]. In 1961, Cr<sub>2</sub>O<sub>3</sub> was recognized as first magnetoelectric material, which has shown magnetoelectric coupling coefficient of the order of 10<sup>-12</sup> s/m [5]. The first multiferroic material discovered was nickel iodine boracites (Ni<sub>3</sub>B<sub>7</sub>O<sub>13</sub>I) with magnetic ordering below T<sub>N</sub> (~ 60 K) and ferroelectric one below T<sub>C</sub> (~ 400 K) [6-7]. The boracites are also unfeasible materials for device applications as they have large unit cell and grow in needle shapes, which exhibit magnetoelectricity only at extremely low temperatures [8]. Although, importance of these materials was not realized by that time however, search began with approach of replacing some of the B-site cations with d<sup>0</sup> character such as Ti, by magnetic cations Fe<sup>3+</sup> with d<sup>n</sup> category, where n ≠ 0, in ABO<sub>3</sub> type oxides. There were materials already discovered with B-site ordered ferroelectric ferrimagnetic Pb(Fe<sub>2/3</sub>W<sub>1/3</sub>)O<sub>3</sub> and B-site disordered ferroelectric antiferromagnetic Pb(Fe<sub>1/2</sub>Nb<sub>1/2</sub>)O<sub>3</sub>. Further, way back in 1990, information technology (IT) started booming and importance of computers and their applications in IT was realized. Following these developments again, it was realized that multiferroic materials may play key role in enhancing data storage capacity and hence may exhibit superior properties than already existing materials for ferroic property alone like BaTiO<sub>3</sub>

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and  $\text{PbTiO}_3$ . Thereafter, research in this field started picking up worldwide but the progress was little bit slow upto 2000. By that time, the whole world had realized the importance of information technology. Soon after this, scientists across the world focused their research interest in the field of multiferroics.

Perovskite oxides in general do not have the same mechanism for the origin of spontaneous polarization. In  $\text{BaTiO}_3$ , shift of center Ti ion is responsible for ferroelectricity while in  $\text{PbTiO}_3$  the lone-pair electrons of Pb ion along with the shift of Ti-ion both are responsible for spontaneous polarization to occur below  $T_C$  [9]. In case of magnetic perovskites, magnetization comes from unpaired d-electrons of the B - site cations like  $\text{Fe}^{3+}$ ,  $\text{Mn}^{3+}$ ,  $\text{Cr}^{3+}$  etc. Thus, existence of magnetism in these materials is caused by spin order [10-11], and magnetic exchange interaction [12] or charge order [13]. Based on these ideas, new oxides have been theoretically predicted to be multiferroic, which further await direct experimental verification [14-16]. Meanwhile, the world wide attention was drawn to study  $\text{BiFeO}_3$  (BFO). Smolenskii's group found it difficult to grow  $\text{BiFeO}_3$  into single crystals [17]. Also in the form of ceramics samples, BFO samples are found to be highly conducting. High conductivity in this specimen has been explained in term of oxygen vacancies and mixed variable valence states of Fe. For perovskites of  $\text{ABO}_3$  type, on one hand, ferroelectric ceramics like  $\text{BaTiO}_3$  have B-site cation with  $d^0$  character which mainly contributes to the spontaneous dipole moment caused by shift of centre Ti-ion from centre position to off center position below  $T_c$ , while on the other hand, magnetization originates from  $d^n$  character ( $n \neq 0$ ) with unpaired electrons in d-orbital like  $\text{Fe}^{3+}$  [18]. In view of this,  $\text{BiFeO}_3$  is thought to be one of the few existing multiferroics where spontaneous polarization arises due to lone pair electrons of S-orbitals of  $\text{Bi}^{3+}$  ions and magnetization arises due to unpaired electrons of d-orbitals of  $\text{Fe}^{3+}$  ions, thus coexisting ferroelectricity and magnetism in the same materials.

So far,  $\text{BiFeO}_3$  (BFO) is the only single phase material that shows multiferroic phenomenon at room temperature. It possesses rhombohedrally distorted perovskite crystal structure with  $R3c$  space group symmetry, high magnetic transition temperature ( $T_N \sim 370^\circ\text{C}$ ) with G-type antiferromagnetic (AFM) behavior and high ferroelectric Curie temperature ( $T_C \sim 830^\circ\text{C}$ ) [19-20]. In BFO, ferroelectricity originates due to the stereochemically active  $6s^2$  lone pair electrons

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of  $\text{Bi}^{3+}$  ion at A-site while magnetic behavior appears due to  $\text{Fe}^{3+}$  ions at B-site, though the magnetization is low due to antiferromagnetic order of cycloid spin structure [21]. In devices based on the magnetoelectric effect, a large magnetic moment is required in addition to the strong ferroelectricity. In BFO, the presence of incommensurate cycloid spin structure, with a periodicity of 62 nm along [111], results in the cancellation of macroscopic magnetization and hence leading to very small magnetic moment [21]. Moreover, a large leakage current density caused by charge defects, nonstoichiometry and secondary phases make it difficult to observe a well-saturated ferroelectric hysteresis loop. These properties of BFO hinder its use in electronic devices. In order to improve electrical and magnetic properties, partial substitution of rare-earth ions at  $\text{Bi}^{3+}$ -sites or/and transition metal ion at  $\text{Fe}^{3+}$ -sites have been carried out [22-25]. Actually, these substitutions have already been found to be effective to modify ferroelectric or the magnetic properties to the some extent in recent years. However, still few issues like leakage current, defects formation etc could not be resolved well and therefore, there is a huge scope to improve electrical and magnetic properties by compositional tuning i.e. by substitution of foreign element in  $\text{BiFeO}_3$ .

With this background in mind, the objective of the present work is to study the effect of substitution on structural, electrical, magnetic and optical properties of  $\text{BiFeO}_3$ . The literature survey, experimental procedure for synthesis and characterization of specimen samples, results and discussion of research work have been organised into seven chapters of this thesis. Chapter wise discussions are given below:

**Chapter 1:** This chapter contains a brief introduction of dielectric properties, ferroelectricity, magnetism and multiferrocity. Different types of multiferroic materials, the origin and history have been discussed. In addition to this, recent developments and motivation to carry out research in this field of multiferroics have been described in details.

**Chapter 2:** The details of sample preparation method, experimental techniques used in due course of the present work have been discussed in this chapter.

**Chapter 3:** In this chapter, synthesis and studies on the effect of praseodymium (Pr) substitution on the structural, electrical, magnetic and optical properties of BFO have been carried out. This system is abbreviated as BPFO. The important inferences are followed as below:

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- Compositions with  $x \leq 0.20$  in the system  $\text{Bi}_{1-x}\text{Pr}_x\text{FeO}_3$  (BPFO) have been prepared by solid state ceramic method. All the samples show rhombohedral structure with R3c symmetry. Pr substitution for compositions with  $x \geq 0.15$  has been found to eliminate the impurity phases.
  - The occurrence of dielectric anomalies at around  $375^\circ\text{C}$  in dielectric plots and magnetic anomalies (AFM to PM) in temperature-dependent magnetization plots at  $\sim T_N$  ( $370^\circ\text{C}$ ) for  $x \leq 0.20$  indicate magnetoelectric coupling in these materials. Bismuth evaporation, which creates oxygen vacancies in pure BFO sample seems to be suppressed with increasing  $x$  and hence reducing the dielectric loss.
  - M-H hysteresis loops for these samples have revealed that Pr substitution induces weak ferromagnetism, leading to the enhanced remanent magnetization with increasing  $x$  in these samples. Further P-E hysteresis loops infer that the value of remnant polarization increases with Pr concentration.
  - Conductivity of these samples has been found to decrease with increasing  $x$ . Decrease in conductivity with  $x$  has been attributed to the suppression of defects in these ceramics which is also supported by decrease in dielectric loss. From these observations it is proposed that Pr substitution controls Bi-evaporation and hence reduces defects formation in these materials.
  - FTIR spectra showed two broad absorption peaks at  $560$  and  $430\text{ cm}^{-1}$ , which may be assigned to the stretching and bending of Fe–O and Bi–O bonds due to buckling in  $\text{BO}_6$  octahedra. Splitting of the absorption peak again around  $430\text{ cm}^{-1}$  for  $x = 0.15$  and  $0.20$  is attributed to the tilting of  $\text{FeO}_6$  octahedra. PL spectra for all these samples show a bandgap of  $\sim 2.67\text{ eV}$ . Observation of two weak emissions at  $545$  and  $564\text{ nm}$  in PL spectra may be attributed to the electronic transitions of  $\text{Pr}^{3+}$  states.

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**Chapter 4:** In this chapter, synthesis and studies on the effect of yttrium (Y) substitution on the structural, electrical, magnetic and optical properties of BFO (abbreviated as BYFO system) have been carried out. The important outcome is followed as below:

- Composition with  $x = 0.00, 0.05, 0.10, 0.15$  and  $0.20$  in the system  $\text{Bi}_{1-x}\text{Y}_x\text{FeO}_3$  (BYFO) ceramics have been synthesized by solid state reaction method. All the samples show rhombohedral structure with  $R3c$  space group symmetry. Lattice parameters determined by using software (POUDRIX), are found to decrease with increasing concentration, which may be due to small ionic radii of  $\text{Y}^{3+}$  ion as compared to  $\text{Bi}^{3+}$  ions.
- The average grain size in all the samples is found to be less than  $4\mu\text{m}$ .
- Dielectric measurements of all the samples have been carried out from room temperature to  $500^\circ\text{C}$  at several frequencies. Dielectric behavior may broadly be divided in two dielectric anomalies, though these anomalies are not very consistent with  $x$ , which may be due to high leakage problem. Dielectric anomaly at low temperature may be ascribed to the magnetic transition in BFO.
- The remnant magnetization also increases with Y concentrations.  $\text{Fe}^{3+}$  ions are the origin of the magnetic anisotropy, which leads to weak ferromagnetism in these samples. It has been observed from P-E loop that the value of remnant polarization increases with Y concentration. Bismuth evaporation seems to create oxygen vacancies in all the samples.
- Conductivity data has been plotted with  $1000/T$  at few selected frequencies. Conductivity behavior of all the samples with temperature is almost similar to BPFO system. It has been observed from these plots that conductivity increases with increasing temperature however, it is decreasing with increasing  $x$ . Activation energy determined in different temperatures regime according linear Arrhenius relation, reveals that conductivity in low temperature regime is attributed to localized electron hopping via defects. Whereas, at high temperature, conduction occurs by charge carrier released during oxidation of defects. Decrease in conductivity with  $x$  has been

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attributed to the suppression of defects in these ceramics which is also supported by decrease in dielectric loss.

- The presence of two vibrational modes at  $442\text{ cm}^{-1}$  and  $545\text{ cm}^{-1}$  in the FTIR spectra correspond to the bending and stretching of Bi-O and Fe-O bonds respectively. Shift of absorbance peak at  $442\text{ cm}^{-1}$  with  $x$  may be attributed to the distortion in  $\text{FeO}_6$  octahedra. PL spectra of compositions with  $x \leq 0.20$  show blue emission at 458 nm, indicating the optical band gap of 2.67eV. The emissions at 561 and 584 nm in yellow region may be attributed to the electronic transitions of  $\text{Y}^{3+}$  states. The optical band gap in the blue region along with yellow emissions find applications in optoelectronic devices.

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**Chapter 5:** In this chapter, synthesis and studies on the effect of praseodymium (Pr) and titanium (Ti) substitution on the structural, electrical, magnetic and optical properties of BFO (abbreviated as BPFTO) have been carried out. The important outcome is described below:

- Pr and Ti co-substituted  $\text{BiFeO}_3$  (BPFTO) ceramics with compositions with  $x \leq 0.20$  have been synthesized in the form of solid solutions. XRD patterns of all these samples illustrate single phase formation. Rietveld refined XRD data has confirmed structural phase transition from rhombohedral (R3c) with  $x \leq 0.10$  to orthorhombic (Pnma) phase with  $0.10 < x \leq 0.20$ . Lattice parameters have been determined. A volume of the unit cell has been found to decrease with increasing  $x$ .
- Dielectric measurements from room temperature to  $500^\circ\text{C}$  at few frequencies revealed some anomalies that infer more than one dielectric processes; the first one is due to relaxation of complex defect dipoles in the intermediate temperature region and other one is due to magnetic transition temperature of pure BFO.
- Appearance of open M-H loop at the origin with increasing  $x$ , except  $x = 0.00$  reveals improved magnetization. Increase in magnetization with  $x$  has been attributed to the partial destruction of spiral spin structure, which is caused by breaking of Fe-O-Fe

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chain in BFO due to insertion of  $\text{Ti}^{4+}$  ions. P-E hysteresis loops revealed that remnant polarization increases with increasing  $x$  i.e. increasing concentration of Pr and Ti in BFO.

- Conductivity vs.  $1000/T$  plots has shown that the conductivity increases with increasing temperature which has been divided in three temperature regions. Further, conductivity decrease with increasing concentration of Pr and Ti. Decrease in conductivity with  $x$  has revealed the suppression of defects in these ceramics which is also supported by decrease in dielectric loss.
- FTIR spectra of these samples show the presence of broad absorption band in the wave number range 400 – 800  $\text{cm}^{-1}$  which is due to overlapping of Fe-O and Bi-O vibration groups. UV-visible spectra in the wavelength range 400 – 700 nm reveals that band gap changes from 2.26 eV for  $x = 0.00$  to 2.08 eV for  $x = 0.20$ , which explores the utilization of these materials for optoelectronic devices in the visible range.

*(This work have been published in Journal of Magnetism and Magnetic Materials, vol. 349, pp. 264-267, 2014)*

**Chapter 6:** In this chapter, synthesis and studies on the effect of yttrium (Y) and titanium (Ti) substitution on the structural, electrical, magnetic and optical properties of BFO (abbreviated as BYFTO) have been carried out. The important inferences are followed as below:

- Y and Ti co-substituted  $\text{BiFeO}_3$  (BYFTO) ceramics with compositions  $x \leq 0.24$  have been synthesized in the form of solid solution. XRD patterns of these samples have shown single phase formation. Change in XRD profile with  $x$  indicates structural transformation. Rietveld refinement of XRD data has confirmed structural phase transition from rhombohedral (R3c) with  $x \leq 0.16$  to orthorhombic (Pnma) phase with  $x = 0.24$ . Volume of the unit cell has been found to decrease with increasing  $x$ .
- Dielectric plots ( $\epsilon_r$  and  $\tan \delta$  vs  $T$ ) have shown few dielectric anomalies at temperature 200°C and 350°C which infer two dielectric processes; the first one seems to be due to relaxation of complex defect dipoles in the intermediate temperature region and other one is due to characteristic magnetic transition temperature ' $T_N$ ' in pure BFO.

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- M-H hysteresis loops at room temperature illustrated, that weak ferromagnetism arises with increasing Y and Ti in BFO. Appearance of open M-H loop with increasing x, except x = 0.00 reveals improved magnetization. Increase in magnetization with x is attributed to the partial destruction of spiral spin structure, which is caused by breaking of Fe-O-Fe chain in BFO due to insertion of Ti<sup>4+</sup> ions. P-E hysteresis loops have revealed that remnant polarization increases with co substitution of Y and Ti in BFO
  - Conductivity vs. 1000/T plots has shown that the conductivity increases with increasing temperature which has been divided in three temperature regions. Further conductivity decrease with increasing concentration of Y and Ti. Decrease in conductivity with x has been a Synopsis-8 suppression of defects in these ceramics which is also supported by decrease in dielectric loss.
  - FTIR spectra of these samples show the presence of broad absorption band which in the wave number range 400 – 800 cm<sup>-1</sup> is due to overlapping of Fe-O and Bi-O vibration groups. UV-visible spectra in the wavelength range 400 – 700 nm reveals that band gap changes from 2.40 eV for x = 0.00 to 2.32 eV for x = 0.24, which explores the utilization of these materials for optoelectronic devices in the visible range.

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**Chapter 7:** This chapter presents a comparative study of the above systems and summary of the results and suggestions for further work.

- XRD patterns of all these samples have shown single phase formation. BPFO and BYFO systems have shown rhombohedral structure symmetry for all the samples with  $x \leq 0.20$ . Whereas BPFTO and BYFTO have shown structural transformation with increasing x. Lattice volumes have been found to decrease with increasing x in BFO.
- A small change in dielectric constant ( $\epsilon_r$ ) and dielectric loss ( $\tan \delta$ ) has been observed in A-sited doped samples like BPFO and BYFO systems, whereas, a significant increase in  $\epsilon_r$  and decrease in  $\tan \delta$  has been obtained in co doped BPFTO and BYFTO systems. This concludes that co-doping has been found to be more effective to control

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defect formation in these materials. Similar to the above, remanent polarization and magnetic polarization have been found to be enhanced with x in co-substituted samples of BPFTO and BYFTO systems.

**Conclusions:** The prime objective to study the impact of doping on structural, dielectric, magnetic and optical properties of bismuth ferrite has been carried out which clearly suggests that substitution of Pr and Ti in BFO (BPFTO system) and Y and Ti in BFO (BYFTO system) has not only produced structural changes but also a significant decrease in dielectric loss with enhanced polarization and magnetization. In addition to this, a complex behavior of conductivity data with temperature as well as with composition can be used for modeling of band conduction mechanisms in these complex oxide materials for optical device applications.

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## **Ph.D. thesis is based on these publications**

### ***Papers in International SCI Journals***

1. **Singh Vikash**, Sharma Subhash, Dwivedi R. K., Kumar Manoj, Kotnala R. K., “Multiferroic and Optical Properties of Pr-Substituted Bismuth Ferrite Ceramics”, Phys. Status Solidi A, vol. 210 (7), pp. 1442-1447, 2013. (**Thomson Reuters, I.F. = 1.469, H index = 72, H5-index = 32, Published by WILEY-VCH Verlag Gmbh, Indexed in SCI and SCOUPS**)
2. **Singh Vikash**, Sharma Subhash, Dwivedi R. K., Kumar Manoj, Kotnala R. K., Mehra N. C., Tondan R. P., “Structural, Dielectric, Ferroelectric and Magnetic Properties of Bi<sub>0.80</sub>A<sub>0.20</sub>FeO<sub>3</sub> (A = Pr, Y) Multiferroics”, J. Supercond Nov Magn., vol. 26, pp. 657-661, 2013. (**Thomson Reuters, I.F. = 0.930, H index = 26, H5-index = 18, Published by Springer, Indexed in SCI and SCOUPS**)
3. **Singh Vikash**, Sharma Subhash, Dwivedi R. K., Kumar Manoj, “Structural and Optical Properties of Pr Doped BiFeO<sub>3</sub> Multiferroic Ceramics”, AIP Conf. Proc., vol. 1512, pp. 462-463, 2013. (**H index = 29, Published by American Institute of Physics, Indexed in SCI and SCOUPS**)
4. **Singh Vikash**, Sharma Subhash, Kumar Manoj, Dwivedi R. K., “Synthesis and Optical Properties of Y<sup>3+</sup> Doped BiFeO<sub>3</sub> Multiferroics”, AIP Conf. Proc., vol. 1536, pp. 1027-1028, 2013. (**H index = 29, Published by American Institute of Physics, Indexed in SCI and SCOUPS**)

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5. **Singh Vikash**, Sharma Subhash, Kumar Manoj, Kotnala R. K., Dwivedi R. K., “*Structural transition, magnetic and optical properties of Pr and Ti co-doped BiFeO<sub>3</sub> ceramics*”, Journal of Magnetism and Magnetic Materials, vol. 349, pp. 264-267, 2014. (**Thomson Reuters, I.F.** = 2.002, **H index** = 111, **H5-index** = 43, **Published by** Elsevier, **Indexed** in SCI and SCOUPS)
  6. **Singh Vikash**, Sharma Subhash, Jha P. K., Kumar Manoj, Dwivedi R. K., “*Effect of Y<sup>3+</sup> substitution on structural, electrical and optical properties of BiFeO<sub>3</sub> ceramics*”, Ceramics International, vol. 40, pp. 1971-1977, 2014. (**Thomson Reuters, I.F.** = 2.086, **H index** = 56 , **H5-index** = 37, **Published by** Elsevier, **Indexed** in SCI and SCOUPS)
  7. **Singh Vikash**, Sharma Subhash, Kumar Manoj, Dwivedi R. K., “*Optical Properties of Y and Ti co-substituted BiFeO<sub>3</sub> Multiferroics*”, AIP Conf. Proc., vol. 1591, pp. 622-624, 2014. (**H index** = 29, **Published by** American Institute of Physics, **Indexed** in SCI and SCOUPS)

## **Other publications not included in Ph.D. thesis**

### ***Papers in International SCI Journals***

1. Sharma Subhash, Singh Vikash, Prakesh Om, Dwivedi R. K., “*Effect of Processing on Dielectric Properties of (0.95)PbZr<sub>0.52</sub>Ti<sub>0.48</sub>O<sub>3</sub>-(0.05) BiFeO<sub>3</sub>*”, Appl Phys A, vol. 112, pp. 975-984, 2013.
2. Sharma Subhash, Singh Vikash, Dwivedi R. K., “*Structural, Optical and dielectric Properties of (0.95)PbZr<sub>0.52</sub>Ti<sub>0.48</sub>O<sub>3</sub>- (0.05)BiFeO<sub>3</sub> solid solution*”, AIP Conf. Proc., vol. 1536, pp. 849-850, 2013.
3. Sharma Subhash, Singh Vikash, Dwivedi R. K., “*Structural, vibrational and dielectric studies of (0.95) Pb (Zr<sub>x</sub>Ti<sub>1-x</sub>) O<sub>3</sub>-(0.05) BiFeO<sub>3</sub> nanoceramics*”, AIP Conf. Proc., vol. 1591, pp. 880-882, 2014.
4. Sharma Subhash, Singh Vikash, Kotnala R. K., Dwivedi R. K., “*Effect of Zr/Ti ratio on structural, vibrational, magnetic and dielectric properties of (0.95) PbZr<sub>x</sub>Ti<sub>1-x</sub>O<sub>3</sub>-(0.05) BiFeO<sub>3</sub> ceramics*”, Journal of materials Science: Materials in Electronics, vol. 25, pp. 1915-1921, 2014.

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### ***Abstracts Presentations in International Conferences***

1. Singh Vikash, Sharma Subhash, Dwivedi R. K., Manoj Kumar, “*Structural and Optical Properties of Pr Doped BiFeO<sub>3</sub> Multiferroic Ceramics*”, 57<sup>th</sup> DAE Solid State Physics Symposium (DAE-2012), December 3-7, 2012, IIT, Mumbai, India. (Poster Presentation)
2. Singh Vikash, Sharma Subhash, Kumar Manoj, Dwivedi R. K., “*Synthesis and Optical Properties of Y<sup>3+</sup>Doped BiFeO<sub>3</sub> Multiferroics*”, International Conference on Recent Trends in Applied Physics and Material Science (RAM-2013), February 1-2, 2013, Govt. College of Engineering and Technology, Bikaner, India (Poster presentation).
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