

## Enhanced multiferroics properties of strontium substituted bismuth ferrite prepared by auto combustion method

S. Iqbal<sup>a,\*</sup>, M. U. Subhani<sup>b</sup>, H. Anwar<sup>b</sup>, Y. Jamil<sup>b</sup>, H. M. Rafique<sup>a,\*</sup>

<sup>a</sup> Department of Physics, University of the Punjab, Quaid-e-Azam Campus  
Lahore-54590, Pakistan

<sup>b</sup> Department of Physics, University of Agriculture Faisalabad-38040 Pakistan

A low cost and simple auto combustion method was used to prepare the  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  ( $x=0.0, 0.04, 0.08, 0.12, 0.16$ ) multiferroics samples. X-ray diffraction patterns confirms the integration of BFO pure phase nanoparticles with a perovskite structure of  $\text{ABO}_3$  type having space group  $R3c$ . Spherical structure with little agglomeration was observed during the morphological studies of the prepared samples.  $\text{Sr}^{2+}$  substituted at the A-site produces expansion in Raman modes and increased variations in cation site occupancy. Impedance analyzer was used to examine the frequency dependent dielectric parameters ( $\epsilon'$ ,  $\tan\delta$ ), electric conductivity ( $\sigma_{ac}$ ), impedance ( $Z$ ) and electric modulus ( $M'$  and  $M''$ ) of the samples in frequency between 20 Hz and 20 MHz. Maximum value of dielectric constant was observed at very low frequency for  $x=0.0$  samples and it decreases by increasing concentration. Resonance peak was observed in tangent loss for high content of strontium. Maximum value of Impedance was observed at low frequency and it decreases by increasing frequency which is due to the grain and grain boundary contributions. It is observed that ac conductivity of the samples is found to be frequency dependent and it varies with doping of Sr in BFO. Improved polarization was examined in P-E loop of Sr doped BFO nanoparticles.

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**Keywords:** Auto combustion method, Multiferroics, characterization, Dielectric, P-E loop

### 1. Introduction

Multiferroics materials are those materials that show two ferroic properties in a single phase. These ferroic properties include ferromagnetism, ferroelectricity, ferrotoroidicity and ferroelasticity. Materials which show the combination of properties of ferromagnetism and ferroelectricity are Magneto-electric (ME) multiferroics. ME effect is the simultaneously collaboration between magnetism and electricity in a single phase. This ME effect is the basics of ME MFs and gives an extra degree of freedom i.e. controlling of electric dipoles and magnetic spins by magnetic and electric field, respectively. ME multiferroics are rare in nature because the microscopic behavior of ferroelectricity and magnetism are significantly in these materials and have been discussed by Hill [1] [2]. ME materials have a wide range of properties, and their use in numerous fields. These are quantum electromagnets, storage-media, magnetic field sensors, television, optical fibers, switches transducers, satellite communication, microwave actuators, multiple state memories, spintronic, microelectronic, and high-density ferroelectric RAM devices make them general marketing products [3] [4] [5]. Among active MFs, bismuth ferrite (BFO) is the reflection of both ferroelectric and G-type anti-ferromagnetic and properties at the same time. At room temperature BFO has the rhombohedrally distorted perovskite structure with space group.

Preparation of BFO in pure phase is a hard task, and to overcome this effort, scientists used various preparation methods such as coprecipitation [6], solid state reaction [7], hydrothermal [8], sol-gel auto combustion method [9], and micro-emulsion [10]. Nowadays, the auto combustion process is widely used because of its different advantages over the other preparation process such as low cost, easy to use, and homogeneity etc [11]. Jaiprakash et al. used solid state reaction process to prepare  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  and to examine the effect of various contents of Sr on the

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\* Corresponding authors: shahidiqbl786@gmail.com

structure, magnetic and dielectric properties. Single phase and a hexagonal structure with space group R3c was observed during the XRD studies. By increasing the content of Sr, unit cell volume decreases. [12]. According to Li et al.  $\text{Mg}^{2+}$  doped  $\text{BiFeO}_3$  has improved ferroelectric and magnetic properties and these materials are appropriate for magnetoelectric and data storage device applications [13] [14].

In the present work, we have prepared the  $\text{Sr}^{2+}$  doped  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  ( $x = 0, 0.04, 0.08, 0.12$  and  $0.16$ ) and examined structural, morphological, dielectric, and ferroelectric properties of  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  compounds for their practical applications.

## 2. Materials and Methods

### 2.1. Sample Preparation and equipment

Sol-gel auto combustion method was used to prepare the multiferroics  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  for  $x=0.0, 0.04, 0.08, 0.12, 0.16$ . Iron nitrate nonahydrate, Bismuth nitrate hexahydrate, Strontium nitrate, nitric acid, citric acid and ammonia solution was used as a starting materials to prepare multiferroics material. All the nitrates are dissolved in distilled water. A little amount of nitric acid was used to dissolve the bismuth nitrate in distilled water. Mixing all the nitrates solution and continue stirring for 1 hour. Add fuel agent citric acid in the prepared solution and maintain the pH of solution at 7 by adding ammonia solution. After this, the solution was placed at  $90^\circ\text{C}$  on hot plate for continue stirring and heating until solution convert into gel. When the gel was formed the temperature of hot plate increased to  $300^\circ\text{C}$ . The gel burnt and ash was obtained. Pestle and mortar were used to grind the ash. This powder was sintered in furnace for 3 h at temperature  $600^\circ\text{C}$ . Now the final product was obtained and ready for the characterization.

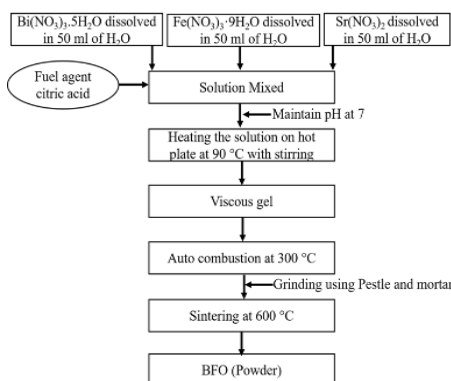


Fig. 1. Synthesis of Sr doped Bismuth ferrites.

X-ray diffractometer was used to examine the structural properties. Morphological properties were examined with the help of JEOL-JSM 5910 model. Impedance analyzer was used to perform the dielectric measurements. Automatic P-E loop tracer was used for ferroelectric studies.

## 3. Results and discussion

The XRD pattern of Sr substituted Bi ferrites with different content  $x = 0, 0.04, 0.08, 0.12$ , and  $0.16$  sintering at temperatures  $600^\circ\text{C}$  are shown in Fig. 2.

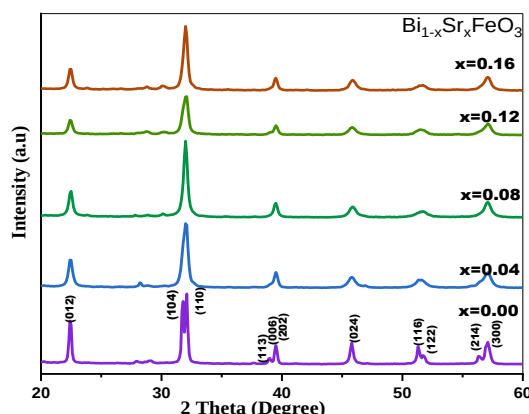


Fig. 2. X-ray diffractions patterns of Sr doped bismuth ferrites at different concentrations.

The perovskite structure with R3c space group is confirmed by XRD pattern. The diffraction peaks of XRD such as (012), (104), (110), (113), (006), (202), (024), (116), (112), (214), and (300) represent the hkl plans respectively. All peaks in the XRD pattern is exactly the same with the ICDD card no. 01-071-2494. Twin peak was observed at (104) and (110) plane. In XRD patterns around  $32^\circ$  is shown where the peak at (104) shifts to a higher angle and combines with peak (110) peak to produce an extended peak. The intensity of this peak also increases by increasing the dopant Sr except  $x=0.12$  sample. Phase pure samples were obtained without traces of any other phases [14]. Table 1 shows the calculated value of structural parameters such as the average crystallite size, lattice constant, volume, and X-ray density. The values shown that the lattice constant  $a$  increases, and decrement in values of  $c$  and volume of unit cell by increasing the amount  $\text{Sr}^{2+}$  in BFO except  $x=0.12$  sample. The variation in values is due to the ionic radii of  $\text{Bi}^{3+}$  (1.03 Å) and  $\text{Sr}^{2+}$  (1.12 Å) and due to the formation of oxygen vacancies to compensate charge imbalance. Debye Scherrer's formula was used to calculate the average crystallite size  $\langle D \rangle$  of prepared nanoparticles.

The average crystallite size of the synthesized nanoparticles was calculated using Debye–Scherrer's formula. The value of  $\langle D \rangle$  lies between 19 and 25 nm. Which is due to Sr content in BFO. The other calculations were also done such as lattice constant which was found in the range 5.5779–5.5932 Å while the volume of the unit cell was found in the range of 371.49–373.83 Å<sup>3</sup>. The X-ray density of the samples was found in the range 7.72 g/cm<sup>3</sup> to 8.34 g/cm<sup>3</sup>.

Table 1. Crystallite size, lattice constants ( $a=b$ ,  $c$ ) volume and x-ray density of Sr-doped Bi ferrites against concentration.

| Concentration (x) | Average Crystallite size (nm) | Lattice constants (Å) |         | Volume (Å <sup>3</sup> ) | X-ray density (g/cm <sup>3</sup> ) |
|-------------------|-------------------------------|-----------------------|---------|--------------------------|------------------------------------|
|                   |                               | a=b                   | c       |                          |                                    |
| 0.00              | 25.99                         | 5.5876                | 13.9034 | 373.83                   | 8.34                               |
| 0.04              | 19.77                         | 5.5892                | 13.7762 | 372.76                   | 8.19                               |
| 0.08              | 20.53                         | 5.5911                | 13.7611 | 372.64                   | 8.04                               |
| 0.12              | 19.78                         | 5.5779                | 13.7858 | 372.59                   | 7.86                               |
| 0.16              | 20.06                         | 5.5932                | 13.7159 | 371.49                   | 7.72                               |

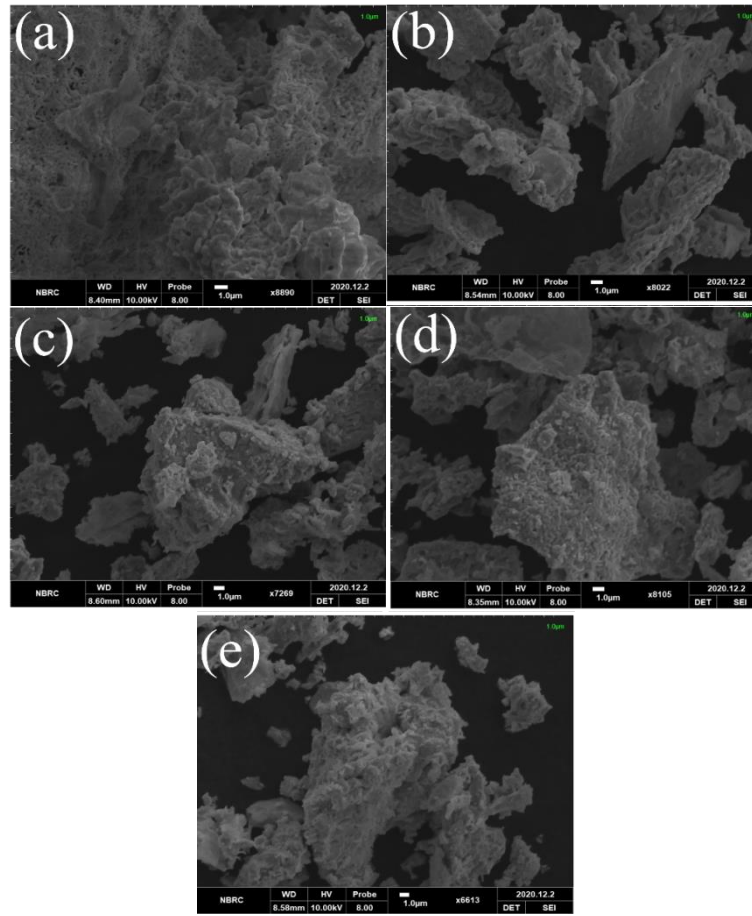


Fig. 3. SEM micrographs of all  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  samples (a)  $x=0.00$ , (b)  $x=0.04$ , (c)  $x=0.08$ , (d)  $x=0.12$  (e)  $x=0.16$ .

Fig 3 shows the SEM images of  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  for concentration ( $x=0, 0.04, 0.08, 0.12$  and  $0.16$ ) samples prepared by using sol-gel auto combustion method. The SEM analysis of BFO for  $x=0$  sample shows grains different sizes that have spherical structure and agglomeration. The shape of the crystalline samples appears to be as flake. The SEM micrographs clearly show that materials have porous nature. It can be seen the little porous in the micrographs and also has some shaded areas due to voids. The micrographs of all the samples also revealed that the porosity of the samples decreased as the Sr doping was increased [9].

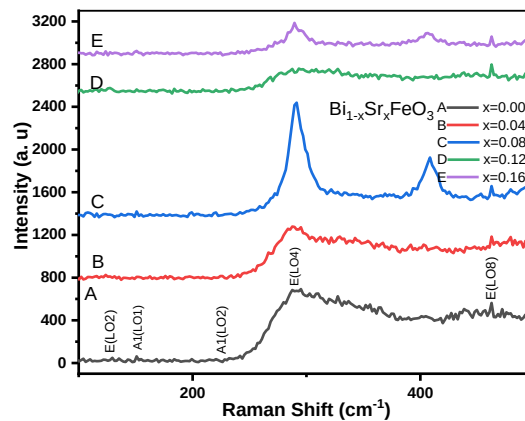


Fig. 4. RT Raman spectra of Sr doped Bismuth ferrites.

Fig. 5 shows the Raman spectra for all Sr doped bismuth ferrites samples to distinguish the effects of strontium on atomic vibration frequencies. The Raman modes of R3c rhombohedral structure for bismuth ferrite can be summarized using the following equation.

$$\Gamma = 4A_1 + 9E \quad (1)$$

All  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  for  $x=0, 0.04, 0.08, 0.12$  and  $0.16$  concentrations show the same raman spectra and modes, representing the perovskite structure with space group R3c. Bismuth atoms contribute to low frequency modes less than  $170 \text{ cm}^{-1}$ . Iron atoms provides modes between  $140\text{--}260 \text{ cm}^{-1}$  [35]. Oxygen movements dominates most of the Raman modes beyond  $260 \text{ cm}^{-1}$ . Three weak and lowest vibrations of the Raman can be caused by mode E(LO2) near  $120 \text{ cm}^{-1}$ , mode  $A_1(\text{LO1})$  near  $150 \text{ cm}^{-1}$ , and mode  $A_1(\text{LO2})$  near  $220 \text{ cm}^{-1}$  [15]. The atomic vibrations close to  $220 \text{ cm}^{-1}$  mainly from the  $A_1$  mode of the  $\text{FeO}_6$  oxygen octahedra. The two high frequency Raman modes E(LO4) near  $280 \text{ cm}^{-1}$  and E(LO8) modes near  $480 \text{ cm}^{-1}$  [15], closely related to atomic vibrations between the oxygen and the A-site atom [16].

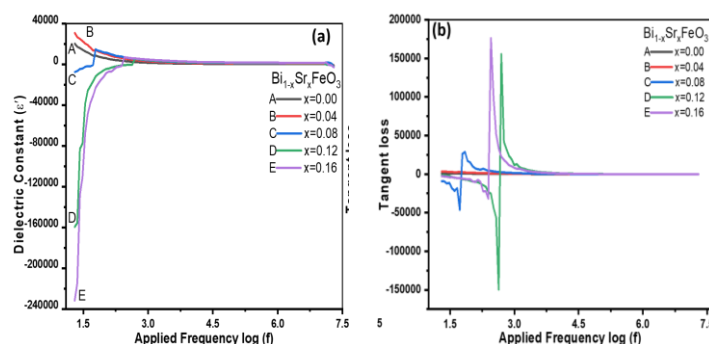


Fig. 5. Graph of (a) Dielectric constant ( $\epsilon'$ ) and (b) Tangent loss Versus frequency log (f).

Fig. 5(a) demonstrates the behavior of dielectric constant ( $\epsilon'$ ) against frequency in the range 20 Hz to 20 MHz for  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  samples.

$$\epsilon' = \frac{Cs}{Co} \quad \text{Where} \quad Co = \frac{A\epsilon_o}{d} \quad (2)$$

Equation (1) was used to measure the  $\epsilon'$  of all the samples. Where  $C_s$  is the capacitance in series,  $A$  and  $d$  is the area and pellet thickness, respectively,  $\epsilon_o$  is the permittivity of free space. From the fig it was shown that maximum value of  $\epsilon'$  was observed at low frequency for sample having low concentration ( $x=0.00, 0.04$ ) and negative value of  $\epsilon'$  was detected for remaining samples. Large value of dielectric constant at low frequency is due to many factors such as oxygen vacancies, interfacial dislocations, grain boundary defects and majority of  $\text{Fe}^{2+}$ . By increasing frequency the value of  $\epsilon'$  decreases, which is due to the polarization and dielectric relaxation. Ionic, Electronic, interfacial and dipolar are the polarization types that look like to total polarization and the impact can be described by Maxwell's Wagner effect [17] [18].  $\tan\delta$  is a term used to explain the loss of electrical energy in the dielectric materials at various frequencies.

$$\tan\delta = \frac{1}{\omega CsRs} \quad (3)$$

Equation (2) was used to measure the tangent for all the Sr doped Bismuth ferrite samples. Fig 5 (b) shows the behavior of tangent loss versus frequency between 20 Hz to 20 MHz. Maximum tangent loss was observed at very low frequency for samples ( $x=0.00, 0.04$ ) and it decreases by increasing frequency.

At low frequency  $\tan\delta$  decreases faster than at higher frequency. Because more energy is needed for electron transfer from  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  leading in high loss [19]. Different factors cause the dielectric loss such as  $\text{Fe}^{2+}$  content, composition, stoichiometry, and annealing temperature etc. At very low frequency 20 KHz,  $\tan\delta$  decreases by adding high content of Sr doping content which is the same in earlier studies [20] [21]. For sample  $x=0.08$ , 0.12, and 0.16 resonance peak was noticed by increasing frequency from 40 Hz to 20 KHz. These resonance peaks could be attributed to electron hopping at the octahedral sites between ferric and ferrous ions, which produces the conduction mechanism. [22] [23].

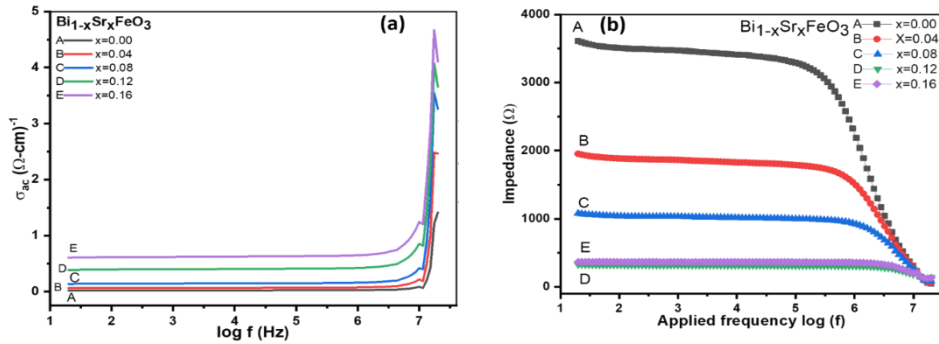


Fig. 6. Plot of (a) AC conductivity and (b) Impedance Versus frequency log (f).

Conductivity is the best method to examine the material electrical properties with the variation of composition, temperature and frequency. The ac conductivity is considered by the following formula

$$\sigma_{ac} = \omega \epsilon_0 \epsilon' \tan\delta \quad (3)$$

where  $\omega$  is the angular frequency and  $\epsilon_0$  is the vacuum permittivity [3]. Fig. 6 (a) shows the behavior of ac conductivity against frequency lies between 20 Hz to 20 MHz for Sr doped bismuth ferrite samples. A smooth frequency independent zone was observed at low frequency followed by dispersion at higher frequencies. Because of the availability of a long time period there is ions hopping from one point to the nearest site at low frequency. Ratio of successful to unsuccessful mechanism of hopping cause the dispersion behavior examined at low frequency.[24]. Fig. 6 (a) shows the increment in ac conductivity with various contents of strontium ( $\text{Sr}^{2+}$ ). This increment in ac conductivity was due to the ionic radii of Sr (1.18Å) and Bi (1.03 Å). Maximum values of ac conductivity were found due to rising charge carriers at B-site. This happened due to dense microstructure. Conduction occurs as a result of hopping of cations within  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$ , resulting in poor conductivity. As shown from Fig 6 by increasing frequency the ac conductivity increased which is due to the grain boundary and increases in cations hopping [25] [26] [27].

For polycrystalline material, complex impedance study is commonly used because it helps to separate grains from grain boundaries. Fig. 6 (b) shows the behavior of impedance (Z) with frequency and the doping contents. Maximum impedance was observed at low frequency. This high impedance confirms the dominant response of grain boundaries towards low frequency signals. By increasing frequency, the Z shows decreasing behavior which could be based on conductivity. In reality, impedance is the circuit resistance which displays the inverse trend to ac-conductivity. The high impedance in low frequency region confirmed the dominant response of grain boundaries towards low frequency signals. [28]

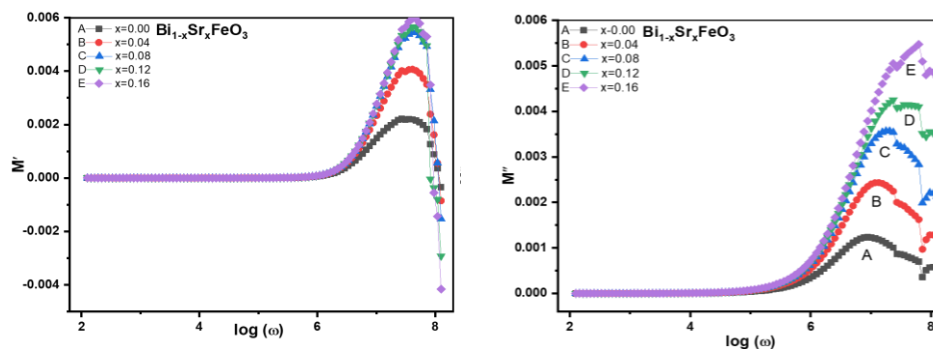


Fig. 7. Real ( $M'$ ) and Imaginary parts of Electric modulus for all samples of Sr doped Bismuth ferrites.

Fig. 7(a) shows the variation of real part of modulus  $M'$  with frequency. The value of  $M'$  at lower frequency is low. For all samples, the value of  $M'$  increases with an increase in frequency and at higher frequencies, it reaches a maximum constant value of  $M_\infty = 1/\epsilon_\infty$ . These observations may be attributed to the lack of restoring force that is responsible for movement of charges under the influence of an induced electric field. These aspects show the negligible contribution of electrodes polarization in the material [29,30]. Such behavior promotes the conduction because of long-range mobilities of charge carriers.

The variation of imaginary part of electric modulus as a function of frequency (at room temperature) is shown in fig 7(b). The virtual part denoted by  $M''$  shows a peak providing insights for charges transport mechanism, called conductivity relaxation [31]. The successful charge carriers hopping happened below the low- frequency side region of the peak. Whereas, the charge carriers above the peak frequency having short-ranged restricted motions were present inside the potential well. The peaks shifting towards high frequencies is an indication of a good correlation between the ion's mobility [3]. Furthermore, the presence of peak in the modulus spectrum indicates the conductivity relaxation [23].

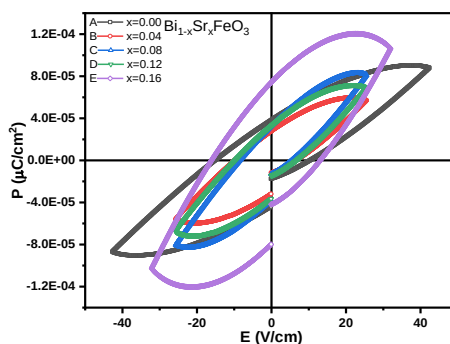


Fig. 8. Polarization hysteresis loop for all samples of Sr doped Bismuth ferrites.

Polarization and Electric field (P-E) loop were used to explain the materials ferroelectric property. Fig 9 demonstrate the P-E loop of Sr doped Bismuth ferrites for ( $x=0, 0.04, 0.08, 0.12$ , and  $0.16$ ) determined at room temperature. As shown in fig by applying field of  $40 \text{ V/cm}$  the sample shows rounded corner and will breakdown with further increasing of field. While all the samples do not indicate saturated hysteresis loops, which is related to the previous studies [32]. Partially saturated behavior of the loop shows the leaky nature and ferroelectric of the compounds. Most lossy behavior was observed in P-E loop for  $x=0.16$ . By applying further electric field it shows round shape with decreasing polarization owing to the break down voltage.[33]. Various defects including lack of crystallinity, imperfections, low density, high porosity, defects, formation of cation or anion, grain size and impurity phases effects are answerable for high leakage current and bigger electrical losses problems in BFO compounds [34]. Table 2 shows the value

maximum polarization ( $P_{\max}$ ), Coercive field ( $E_c$ ), and remanent polarization ( $P_r$ ) of all samples. The results are well agreement in the previous studies [11].

Table 2:  $P_{\max}$ ,  $E_c$ , and  $P_r$  Values for all Sr doped bismuth ferrite samples.

| Concentration (x) | $P_{\max}$ ( $\mu\text{C}/\text{cm}^2$ ) | $E_c$ (V/cm) | $P_r$ ( $\mu\text{C}/\text{cm}^2$ ) |
|-------------------|------------------------------------------|--------------|-------------------------------------|
| 0.00              | 8.80E-05                                 | 10.39        | 3.85E-05                            |
| 0.04              | 5.70E-05                                 | 6.61         | 2.79E-05                            |
| 0.08              | 8.11E-05                                 | 5.43         | 3.28E-05                            |
| 0.12              | 6.88E-05                                 | 6.77         | 3.29E-05                            |
| 0.16              | 1.06E-04                                 | 13.23        | 7.44E-05                            |

#### 4. Conclusions

In this research work Sr substituted  $\text{Bi}_{1-x}\text{Sr}_x\text{FeO}_3$  has been effectively prepared by using the auto combustion method and the prepared samples was sintered at temperatures 600 °C for 3 h. X-ray Diffraction Technique (XRD) and Raman spectra was used to check the microstructural characteristics. XRD and raman spectra of the prepared materials confirms the perovskite structure with Phase group R3c. Sherrer formula was used to find the average crystallite size and it lies between 19.77 nm and 25.99 nm. The morphological results have clarified the and growth of grain which was close in agreement with results of XRD. Maximum dielectric constant was observed at low frequency at it decreases by increasing frequency. The maximum value of  $P_{\max}$  and  $P_r$  is  $1.06 \times 10^{-4} \mu\text{C}/\text{cm}^2$  and  $7.44 \times 10^{-5} \mu\text{C}/\text{cm}^2$  respectively for  $x = 0.16$ . Variations in dielectric properties by doping make this material is very helpful for technological applications.

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