



# Tailored Synergy: Synthesis and In-Depth Structural Analysis of $x[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4] + (1-x)[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ Composites

Tandel RC<sup>1</sup>, Sunitha<sup>1</sup>, Bagal S<sup>1</sup>, Kamat C<sup>1</sup>, Kotekar S<sup>1</sup>, Naik S<sup>1</sup>, Patil S<sup>1</sup>, Kakati S<sup>2</sup>, Mathad SN<sup>2\*</sup>, Shirgaonkar DB<sup>3</sup>, Patil RK<sup>4</sup>, Rendale MK<sup>5</sup>, Deshpande SM<sup>6</sup> and Pujar RB<sup>1†</sup>

<sup>1</sup>P G Department of Studies and Research in Physics, PC Jabin Science College, India

<sup>2</sup>Department of Engineering Physics, KLE Institute of Technology, India

<sup>3</sup>Department of Physics, ARACS College, India

<sup>4</sup>KLE College of Engineering and Technology, India

<sup>5</sup>Department of Engineering Physics, KLS Gogte Institute of Technology, India

<sup>6</sup>Department of Chemistry, SKE Society's Govindram Seksaria Science College, India

\*Corresponding author: Shridhar N Mathad, Department of Engineering Physics, KLE Institute of Technology, India, Tel: 9886347873; Email: physicsiddu@kleit.ac.in

†Equally contributed towards this article

## Research Article

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## Abstract

M E Composites with composition  $x[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4] + (1-x)[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ , Where x varying from 0.1 to 0.4 in molar ratio, were prepared by standard double sintering ceramic technique. The presence of ferrites phase, namely  $[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4]$  and ferroelectric phase, namely  $[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ , were confirmed by X-Ray Diffraction analysis, whereas SEM micrographs were obtained to study the morphology of samples. The x ray diffraction patterns exhibit a set of well-defined ferrites and ferroelectric peaks. The tetragonality ratio c/a, of the ferroelectric phase remains the same in all the samples, the porosity varies from 20% to 30% while the average grain diameter lies in the range of 0.5  $\mu\text{m}$  to 2  $\mu\text{m}$ .

**Keywords:** Ferrites; Ferroelectrics; Porosity; Micrographs; Grain Diameter

## Introduction

Ferroelectrics and ferrites play crucial roles in various electronic applications, from nonvolatile materials to data storage. In the pursuit of advancing materials science, the exploration of multifunctional composites has gained significant attention. These composites, which combine different phases, offer a wide range of applications, from electronic devices to energy storage systems [1-4]. One promising avenue is the synthesis and characterization of ferrite-ferroelectric composites, leveraging the unique properties of both materials. Ferrite-ferroelectric composites

combine magnetic ferrites, typically composed of iron oxide compounds, with ferroelectrics, which exhibit spontaneous electric polarization. This combination results in materials with enhanced electromagnetic and ferroelectric properties [5-9]. Key aspects of these composites include synthesis, electromagnetic and ferroelectric properties, magnetoelectric coupling, tunable microwave devices, energy harvesting, and lead-free ferroelectrics [10-17].

There are two types of multiferroics: single-phase, where one material possesses both ferroelectric and magnetic ordering, and composites, consisting of different

phases with ferroelectric and magnetic properties [18-21]. Examples include  $\text{BiFeO}_3$  and  $\text{BiMnO}_3$  for single-phase, and  $(\text{Ni,Zn})\text{Fe}_2\text{O}_4\text{-BaTiO}_3$ ,  $\text{BaSrTiO}_3\text{-(Ni,Zn)Fe}_2\text{O}_4$ ,  $\text{Ni(Co,Mn)Fe}_2\text{O}_4\text{-BaTiO}_3$ , and  $\text{CoFe}_2\text{O}_4\text{-BaTiO}_3$  for composites [22-25].

In this research, the focus is on integrating Ni-Cu-Co Ferrite with BST, aiming to exploit synergies from their complementary properties. Various compositions are explored to find the optimal blend with superior attributes for specific applications. The study involves synthesizing the  $x[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4] + (1-x)[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$  composites through the solid-state method, followed by in-depth characterization of their structural properties. Ultimately, the goal is to contribute valuable insights for the development of advanced materials with tailored functionalities and improved performance.

## Experiment

Both ferrite and ferroelectric phases were prepared through a standard solid state reaction method, using AR grade  $\text{NiO}$ ,  $\text{CuO}$ ,  $\text{CoO}$  and  $\text{Fe}_2\text{O}_3$  powders in molar ratio as starting materials (Ferrite). Whereas ferroelectric phases was prepared by same method, using AR grade  $\text{BaCO}_3$ ,  $3\text{SrCO}_3$ , and  $\text{TiO}_2$  as starting materials. The mixture was pre-sintered at  $800^\circ\text{C}$  for 10 hours in each case. ME Composites were synthesized by mixing 10 to 40 molar percentage of ferrite phase with 90 to 60 mole percentage of ferroelectric phase respectively. And presintered at  $1000^\circ\text{C}$  for 12 hours. These composites were pressed in to pellets about 1 gram, 2mm in thickness and 1cm in diameter and subjected to final sintering at  $1200^\circ\text{C}$  for about 15 hours in muffle furnace and then furnace cooled. A detailed Schematic synthesis method for ferrite and ferroelectric phase is shown in Figures 1 & 2.

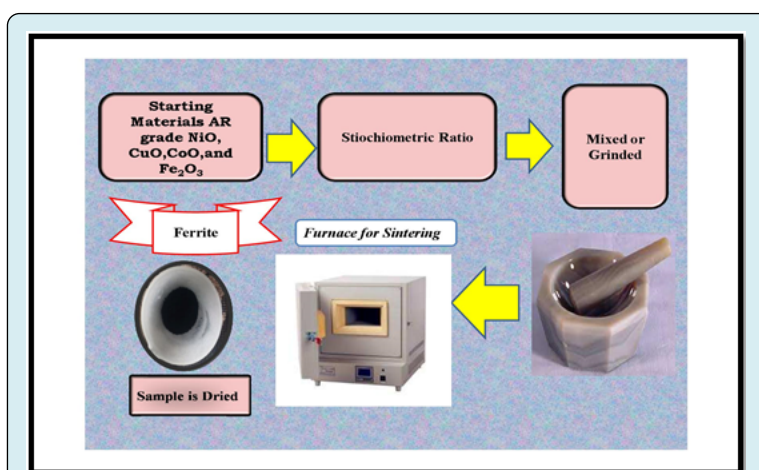


Figure 1: Schematic Synthesis of Ferrite Phase of sample.

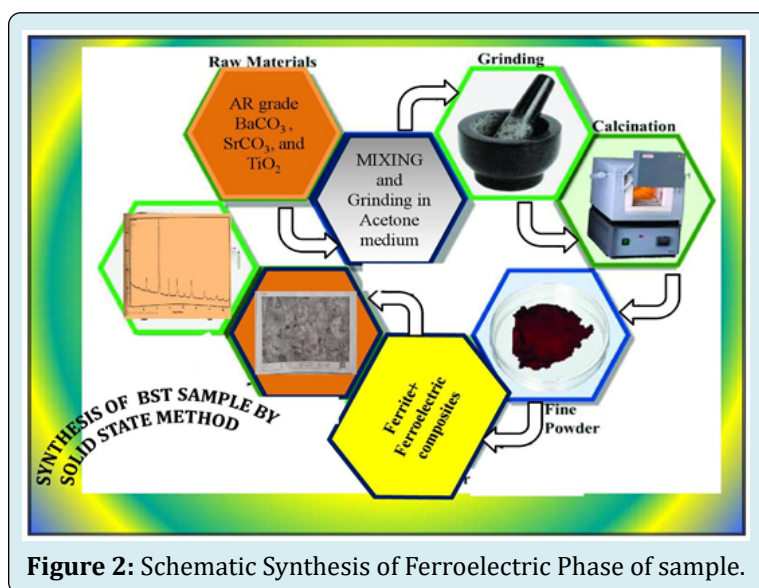


Figure 2: Schematic Synthesis of Ferroelectric Phase of sample.

## Characterization

The presence of crystalline phases and crystal structure of the composites and constituents phases were determined by powder X-Ray Diffraction, using Cu-K $\alpha$  monochromatic radiation of wavelength 1.5148Å, in a wide range of glancing angle 2 $\theta$  from 10° to 90° the micrographs of the samples were obtained to study the surface morphology, grain size and microstructure through SEM.

## Results and Discussion

X-Ray diffraction patterns of the Ni<sub>0.2</sub>Cu<sub>0.3</sub>CO<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub> represented from the Figure 3. X ray diffraction patterns

indexing by JCPD data ferrite and ferroelectric exhibits face centered cubic structure and tetragonal structure. The absence of extra line confirms the formation of single phase ferrites the calculated values of interplanar distances and lattice constant are in good agreement with those expected for spinel ferrites. The cyclic sum of miller indices is even number which confirms FCC Structure (Table 1). The same behaviour is observed in the present case. X ray diffraction pattern of Ba<sub>0.7</sub>Sr<sub>0.3</sub>TiO<sub>3</sub> ferroelectric phase (Figure 4) are indexed with the help of JCPD data. The observed doublet (002) (200); (103) (301); and (113) (311) without additional peaks confirms the formation of tetragonal perovskite structure of ferroelectric phase (Table 2).

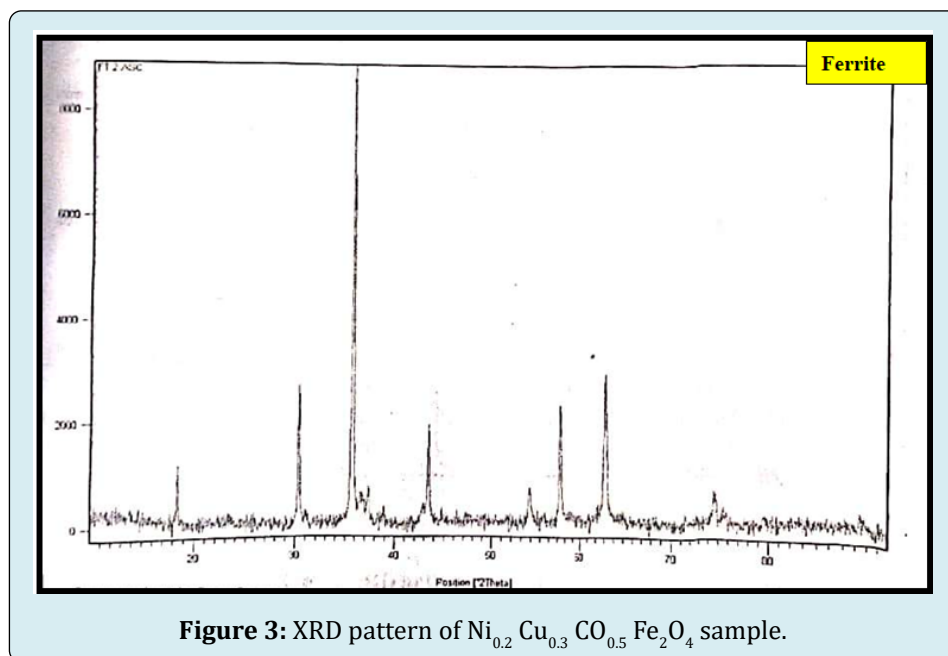


Figure 3: XRD pattern of Ni<sub>0.2</sub>Cu<sub>0.3</sub>CO<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub> sample.

| 2 $\theta$ | $\theta$ | sin $\theta$ | hkl | d <sub>obs</sub> (Å) | D <sub>cal</sub> (Å) | Lattice Constant |
|------------|----------|--------------|-----|----------------------|----------------------|------------------|
| 18.4454    | 9.2227   | 0.1602       | 111 | 4.8101               | 4.8146               | a=8.3389Å        |
| 30.308     | 15.154   | 0.2614       | 220 | 2.9491               | 2.9482               |                  |
| 35.7138    | 17.8569  | 0.3066       | 311 | 2.5141               | 2.5142               |                  |
| 37.1497    | 18.5748  | 0.3185       | 222 | 2.4202               | 2.4072               |                  |
| 43.3115    | 21.6557  | 0.369        | 400 | 2.089                | 2.0847               |                  |
| 53.9054    | 26.9527  | 0.4532       | 422 | 1.7008               | 1.7021               |                  |
| 57.4299    | 28.7149  | 0.4804       | 511 | 1.6046               | 1.6048               |                  |
| 74.598     | 37.299   | 0.6059       | 622 | 1.2722               | 1.2571               |                  |

Table 1: Lattice constant of Ferrite sample.

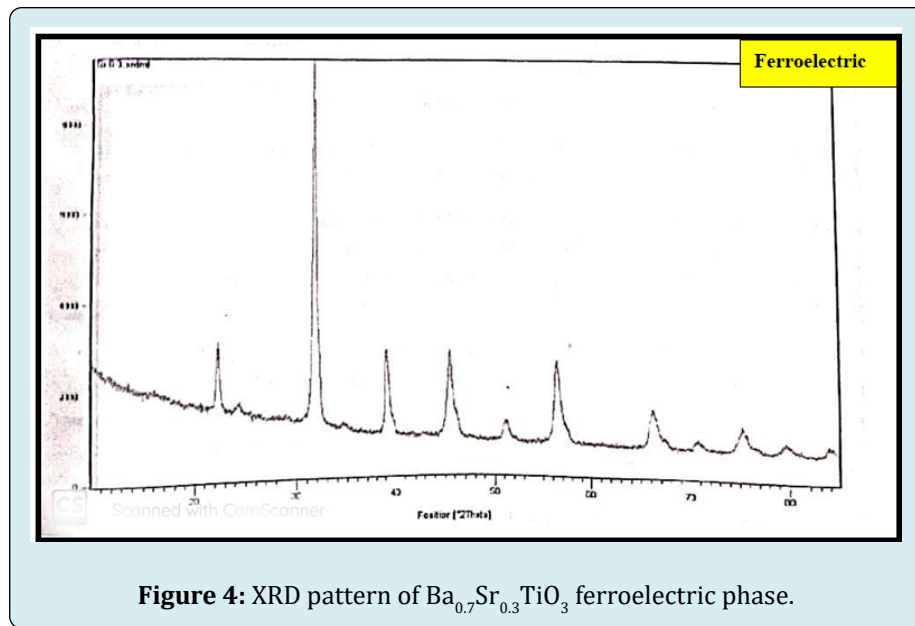


Figure 4: XRD pattern of  $Ba_{0.7}Sr_{0.3}TiO_3$  ferroelectric phase.

| $2\theta$ | $\theta$ | $\sin\theta$ | hkl | $d_{cal}(\text{\AA})$ | $d_{obs}(\text{\AA})$ | Lattice Parameter   |
|-----------|----------|--------------|-----|-----------------------|-----------------------|---|
| 22.243    | 11.1213  | 0.1929       | 1   | 3.9966                | 39.968                | $a=3.99463\text{\AA}$ $c=3.9986\text{\AA}$<br>(Tetragonality) $c/a=1.001$ |
| 31.678    | 15.8389  | 0.2729       | 110 | 2.8244                | 2.8246                |   |
| 39.035    | 19.5177  | 0.3341       | 111 | 2.3074                | 2.3075                |   |
| 45.487    | 22.7436  | 0.3866       | 200 | 1.994                 | 1.9941                |   |
| 51.184    | 25.5921  | 0.432        | 210 | 1.7846                | 1.784                 |   |
| 57.574    | 28.7872  | 0.4815       | 211 | 1.6008                | 1.6009                |   |
| 66.187    | 33.0935  | 0.546        | 210 | 1.4118                | 1.4119                |   |
| 70.707    | 35.3535  | 0.5786       | 103 | 1.3323                | 1.3323                |   |
| 75.339    | 37.6694  | 0.6111       | 301 | 1.0261                | 1.2615                |   |
| 79.623    | 39.8116  | 0.6402       | 222 | 1.204                 | 1.204                 |   |
| 83.834    | 41.9168  | 0.668        | 320 | 1.1539                | 1.153                 |   |

Table 2: Lattice constant of Ferroelectric sample.

The absence of intermediate peaks apart from  $x[Ni_{0.2}Cu_{0.3}CO_{0.5}Fe_2O_4] + (1-x)[Ba_{0.7}Sr_{0.3}TiO_3]$  ferrites and ferroelectric phases Figure 5 is attributed to the fact that no chemical reaction have been taken between the constituent phases during final sintering. The peaks exhibit both perovskite (110) and cubic (311) peaks which are the characteristics of ferroelectric and ferrites phase respectively. The lattice parameters in case of composites (Tables 3-6) are almost equal to those of constituents phases. This indicates the

absence of structural changes with varying molar portions [26,27]. However the intensity of peaks is found decrease with components. This is due to the capacity of ferrites phase to dissolve in to the spinel lattice [26,27]. The data on patterns is given in tables from 1 to 6. Figures 1&2 exhibit a set of well defined ferric and ferroelectric peaks [26,27]. The variation of X ray Density, actual density and porosity with doping is shown in Figure 6.

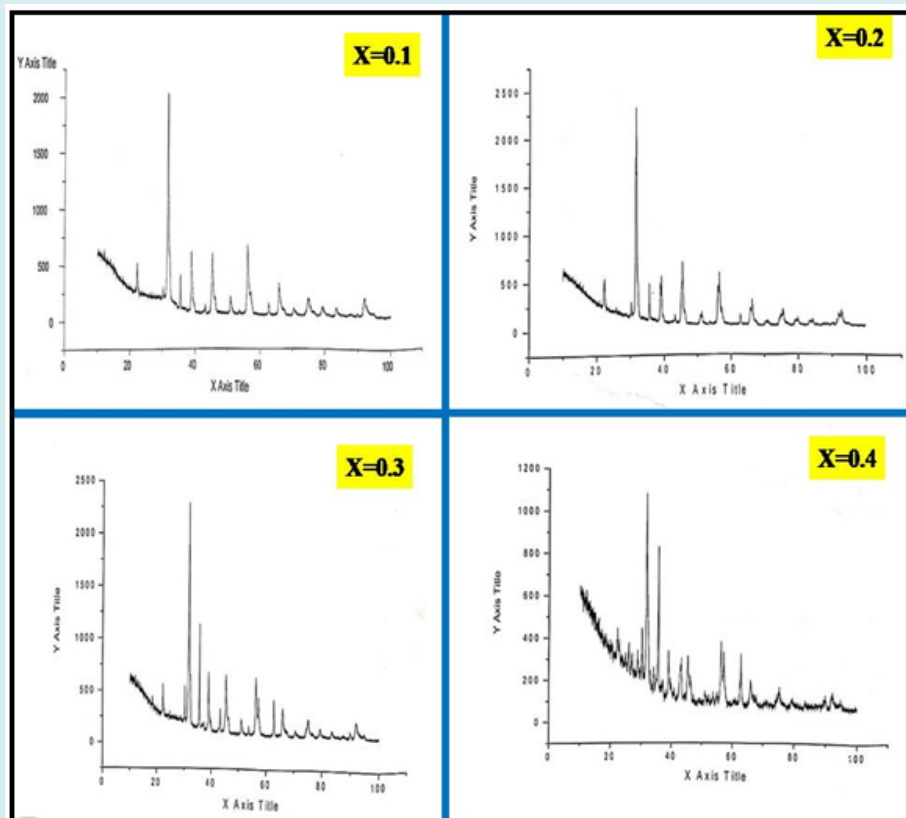


Figure 5: XRD patterns of  $x[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{CO}_{0.5}\text{Fe}_2\text{O}_4] + (1-x)[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$  ( $x=0.1, 0.2, 0.3$  and  $0.4$ ).

| $2\theta$ | $\theta$ | $\text{Sin}\theta$ | hkl    | $d_{\text{cal}}$ | Lattice Parameter  |
|-----------|----------|--------------------|--------|------------------|--|
| 22.156    | 11.078   | 0.1922             | *(100) | 4.0111           | Ferrite phase $a=8.3758\text{\AA}$                               |
| 30.146    | 15.073   | 0.26               | 220    | 2.965            |  |
| 31.574    | 15.787   | 0.2721             | *(101) | 2.8331           | Ferroelectric phase<br>$a=8.3758\text{\AA}$ $c=4.0096\text{\AA}$ |
| 35.535    | 17.7675  | 0.3052             | 311    | 2.5259           |  |
| 38.901    | 19.4505  | 0.3329             | *(111) | 2.3157           | (tetragonality) $c/a=1.0009$                                     |
| 42.624    | 21.312   | 0.3634             | 400    | 2.1214           |  |
| 45.242    | 22.621   | 0.3846             | *(002) | 2.004            |  |
| 50.971    | 25.4855  | 0.43028            | *(210) | 1.7916           |  |
| 53.572    | 26.786   | 0.4507             | 422    | 1.7105           |  |
| 56.241    | 28.1205  | 0.4713             | *(211) | 1.6357           |  |
| 62.735    | 31.3675  | 0.5205             | 440    | 1.4811           |  |
| 65.965    | 32.9825  | 0.5444             | *(220) | 1.4161           |  |
| 70.589    | 35.2945  | 0.5778             | *(221) | 1.3342           |  |
| 75.077    | 37.5385  | 0.6093             | *(301) | 1.2652           |  |
| 79.395    | 39.6975  | 0.6387             | *(311) | 1.2069           |  |
| 83.713    | 41.8565  | 0.6673             | *(222) | 1.1553           |  |
| 92.366    | 46.183   | 0.7216             | 731    | 1.0683           |  |

Table 3: Lattice parameter of  $0.1[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{CO}_{0.5}\text{Fe}_2\text{O}_4] + 0.9[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ .

| 2 $\theta$ | $\theta$ | Sin $\theta$ | hkl    | d <sub>cal</sub> | Lattice Parameter                           |
|------------|----------|--------------|--------|------------------|---|
| 22.326     | 11.163   | 0.194        | *(100) | 3.982            | Ferrite phase<br>a=8.3638Å                  |
| 30.231     | 15.116   | 0.261        | *(220) | 2.956            |   |
| 45.616     | 22.808   | 0.388        | *(002) | 1.989            | Ferroelectric phase<br>a=8.39796Å c=3.9809Å |
| 51.362     | 25.681   | 0.433        | *(210) | 1.779            |   |
| 56.717     | 28.359   | 0.475        | *(211) | 1.623            |   |
| 62.888     | 31.444   | 0.522        | 440    | 1.478            |   |
| 66.05      | 33.025   | 0.545        | *(220) | 1.415            | (tetragonality)<br>c/a=1.0003               |
| 71.099     | 35.55    | 0.581        | *(221) | 1.326            |   |
| 75.638     | 37.819   | 0.613        | *(301) | 1.257            |   |
| 79.412     | 39.706   | 0.639        | *(311) | 1.207            |   |
| 84.376     | 42.188   | 0.672        | *(222) | 1.148            |   |
| 93.063     | 46.532   | 0.726        | *(731) | 1.062            |   |

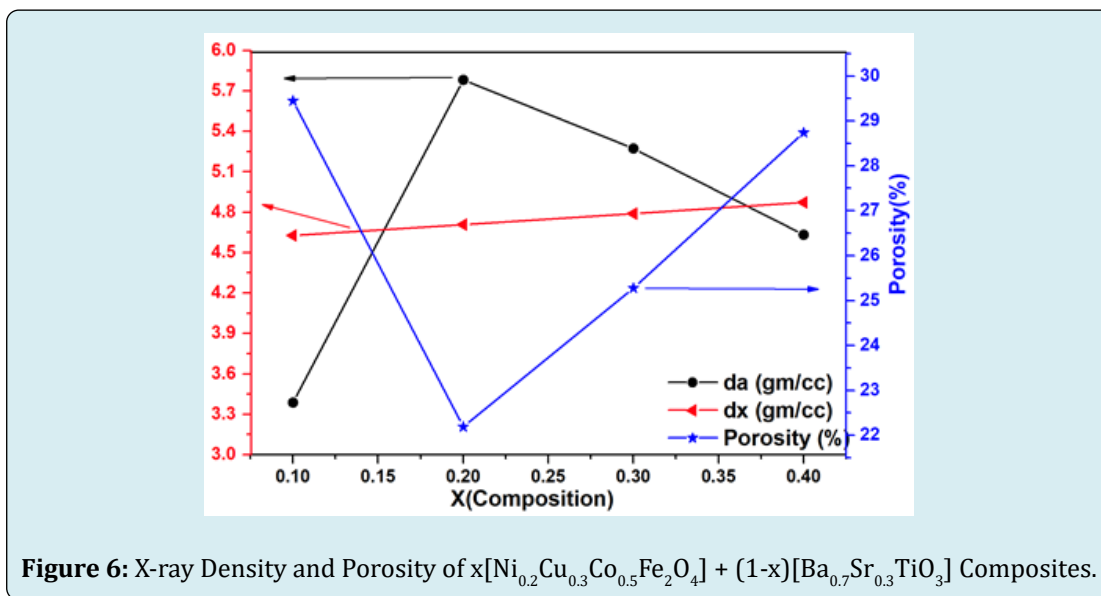
**Table 4:** Lattice parameter of  $0.2[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{CO}_{0.5}\text{Fe}_2\text{O}_4] + 0.8[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ .

| 2 $\theta$ | $\theta$ | Sin $\theta$ | hkl    | d <sub>cal</sub> | Lattice Parameter                          |
|------------|----------|--------------|--------|------------------|--|
| 22.14      | 11.07    | 0.192        | *(100) | 4.0172           | Ferrite phase<br>a=8.3747Å                 |
| 30.18      | 15.09    | 0.26         | *(220) | 2.9616           |  |
| 31.54      | 15.77    | 0.272        | *(106) | 2.8363           | Ferroelectric phase<br>a=4.0103Å c=4.0128Å |
| 35.55      | 17.776   | 0.3053       | *(311) | 2.5251           |  |
| 38.9       | 19.451   | 0.333        | *(111) | 2.3157           |  |
| 43.22      | 21.61    | 0.368        | *(400) | 2.0931           |  |
| 45.31      | 22.665   | 0.385        | *(002) | 2.0013           | (tetragonality)<br>c/a=1.0006              |
| 50.94      | 25.469   | 0.43         | *(210) | 1.7928           |  |
| 53.64      | 26.82    | 0.451        | *(422) | 1.7086           |  |
| 56.22      | 28.112   | 0.471        | *(211) | 1.636            |  |
| 62.8       | 31.402   | 0.521        | *(440) | 1.4797           |  |
| 66.02      | 33.008   | 0.545        | *(220) | 1.4150           |  |
| 70.5       | 35.252   | 0.577        | *(221) | 1.3356           |  |
| 75.3       | 37.649   | 0.611        | *(301) | 1.2621           |  |
| 79.57      | 39.783   | 0.64         | *(311) | 1.2047           |  |
| 83.7       | 41.848   | 0.667        | *(222) | 1.1557           |  |
| 92.2       | 46.098   | 0.721        | *(731) | 1.0699           |  |

**Table 5:** Lattice parameter of  $0.3[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{CO}_{0.5}\text{Fe}_2\text{O}_4] + 0.7[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ .

| 2 $\theta$ | $\Theta$ | Sin $\Theta$ | hkl    | d <sub>cal</sub> | Lattice parameter                        |
|------------|----------|--------------|--------|------------------|--|
| 22.19      | 11.095   | 0.192        | *(100) | 4.007            | Ferrite phase<br>a=8.3775Å               |
| 30.16      | 15.082   | 0.26         | *(200) | 2.963            |  |
| 31.57      | 15.787   | 0.272        | *(111) | 2.833            | Ferroelectric phase<br>a=4.007Å c=4.009Å |
| 35.54      | 17.768   | 0.305        | *(311) | 2.526            |  |
| 38.94      | 19.468   | 0.333        | *(111) | 2.313            |  |
| 43.2       | 21.601   | 0.368        | *(400) | 2.094            | (tetragonality)<br>c/a=1.0005            |
| 45.26      | 22.629   | 0.385        | *(002) | 2.003            |  |
| 51.01      | 25.503   | 0.431        | *(210) | 1.79             |  |
| 53.62      | 26.812   | 0.451        | *(422) | 1.709            |  |
| 56.26      | 28.129   | 0.472        | *(211) | 1.635            |  |
| 62.82      | 31.41    | 0.521        | *(440) | 1.479            |  |
| 65.9       | 32.949   | 0.544        | *(220) | 1.417            |  |
| 75.26      | 37.632   | 0.611        | *(301) | 1.263            |  |
| 79.24      | 39.621   | 0.638        | *(311) | 1.209            |  |
| 92.4       | 46.2     | 0.722        | *(731) | 1.068            |  |

**Table 6:** Lattice parameter of  $0.4[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4] + 0.6[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ .



**Figure 6:** X-ray Density and Porosity of  $x[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4] + (1-x)[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$  Composites.

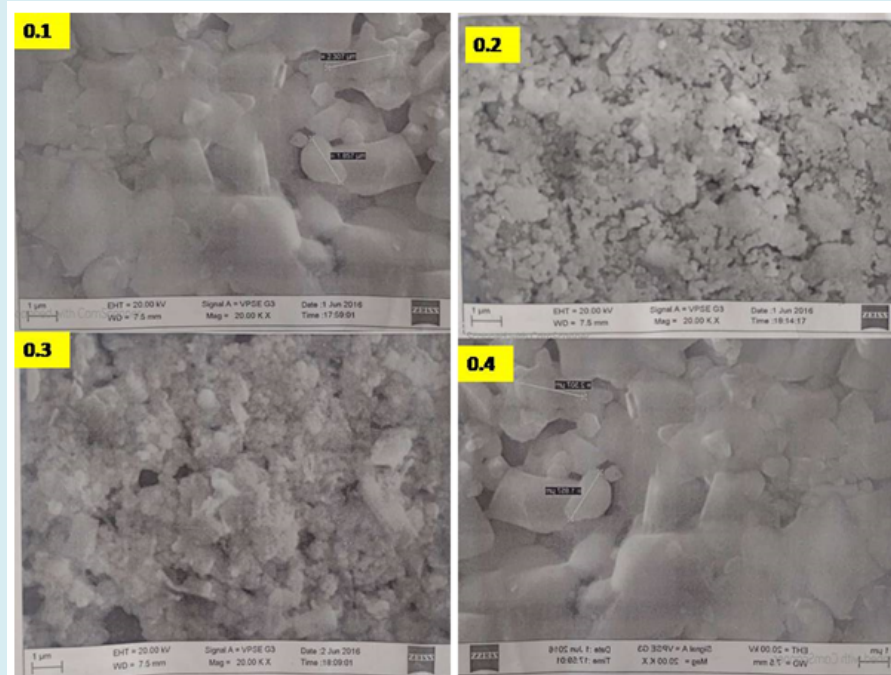
### SEM analysis

Scanning Electron Microscopy (SEM) is a powerful imaging technique that provides high-resolution, three-dimensional images of the surface morphology of ferrites & ferroelectric samples. The average grain size was estimated by Cotrell's method [28], which lies in between 0.8  $\mu\text{m}$  to 2.0  $\mu\text{m}$  and decrease with increase in mole percentage of ferrites phase (Figure 7) and cause for the decrease of mean free path of electrons. The porosity is an inherent phase associated

with the samples prepared by ceramic method. Porosity is a crucial aspect of materials prepared by ceramic methods, and it plays a significant role in influencing the properties and applications of these materials. In the present case porosity in composites varies from 20 to 30 percent (Figure 6). The grain growth in the ME composite is assigned to the presence of inclusions and pores in the solid solution which migrate to the grain boundary. The grain growth in the ME Composite depends on the particle size of individual phases and their distribution, homogeneity of chemical composition

and sintering conditions [26-28]. Increase in ferrite phase decrease the porosity and hence decrease the grain size. This leads to decrease in magnetization of the ME composites.

Because, of large grain and less effective in inducing ferrite and ferroelectric coefficients rather than smaller ones [26-28].



**Figure 6:** SEM images of  $x[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4] + (1-x)[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$  ( $x=0.1, 0.2, 0.3$  and  $0.4$ ).

## Conclusions

A simple and cost-effective solid-state technique was employed to synthesize both ferrite & ferroelectric samples. Phase of Ferrite sample cubic, Ferroelectric as tetragonal and further composites were analyzed through XRD analysis. The study focused on investigating variation of structural properties of  $x[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4] + (1-x)[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$  Composites. The presence of cubic phase of  $[\text{Ni}_{0.2}\text{Cu}_{0.3}\text{Co}_{0.5}\text{Fe}_2\text{O}_4]$ , ferroelectric phase (tetragonal)  $[\text{Ba}_{0.7}\text{Sr}_{0.3}\text{TiO}_3]$ , were confirmed by X-Ray Diffraction analysis and SEM average grain diameter lies in the range of  $0.5\mu\text{m}$  to  $2\mu\text{m}$ .

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