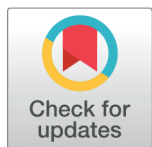


## ORIGINAL ARTICLE



# Thickness Dependent Dielectric Properties Of CFO-PZT-CFO Tri-layered Composites

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B S Kavitha<sup>1</sup>, H V Bhavana<sup>2</sup>, S S Bellad<sup>3\*</sup><sup>1</sup> Department of Physics, Government Science College (Autonomous) Hasan, India-573202<sup>2</sup> Department of Physics, Saptagiri NPS University, Bangalore, India-560057<sup>3</sup> Materials Science lab, Maharani Cluster University, Bangalore, India-560001

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\* Corresponding author.

[ssbellad1971@gmail.com](mailto:ssbellad1971@gmail.com)

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## Abstract

**Objectives:** The main objective of the present research is to synthesize  $\text{CoFe}_2\text{O}_4$  (CFO) and  $\text{PbZr}_{0.52}\text{Ti}_{0.48}\text{O}_3$  (PZT) materials in nanoform and to fabricate the tri-layered structures to study the dielectric properties. **Method:**  $\text{CoFe}_2\text{O}_4$  (CFO) and  $\text{PbZr}_{0.52}\text{Ti}_{0.48}\text{O}_3$  (PZT) materials are synthesized by using combustion method. Glycine ( $\text{C}_2\text{H}_5\text{NO}_2$ ) was used as fuel. X-ray diffraction (XRD) technique is used to study the crystal structure. Tri-layered structures of CFO/PZT/CFO are fabricated by co-pressing the CFO and PZT powders. The dielectric measurements are carried out on tri-layered pallet samples in the frequency range 10 Hz-10MHz. **Findings:** The XRD studies reveal the single phase formation in each case. The dielectric constant ( $\epsilon'$ ) decreases with increase in frequency of the applied AC field; rapidly at low frequency side and then remain unaltered at higher frequency region. The dielectric dispersion is observed at low frequency region and is attributed to the inhomogeneity in the microstructure of the samples. The rapid decrease of dielectric constant at lower frequency region with increase in frequency is caused due to failure of the hopping frequency of the charge carriers to follow the frequency of the applied AC electrical field. The variation of dielectric loss tangent with frequency follow the same trend as that of dielectric constant, however the samples with low thickness PZT layer show loss maxima in the frequency range 50 Hz-1000Hz. The AC electrical conductivity of the present composites remains unaltered up to 10 kHz frequency of the applied field and then increase abruptly beyond that limit because of increase in hopping frequency of the charge carriers. The linearity in  $\text{Log}(\sigma_{AC}-\sigma_{DC})$  vs.  $\text{Log}(\omega^2)$  plot indicates that the small polaron hopping mechanism is responsible for the conductivity process in these composites. **Novelty:** No reports are found in the literature on the co-axial pressing of CFO and PZT materials alternatively to make tri-layered structures. Also, no reports are found on the dielectric properties of the tri-layered structures of CFO and PZT.

**Keywords:** Ferrites; Ferroelectrics; Composites; Dielectrics; XRD

## 1 Introduction

Recent developments in science and technology are very advanced in general and in particular the progress in microelectronics industry is such that even the Moore's law is challenged. This is due to the invention of newer materials which are multifunctional in nature, yet simple in structure, helps in miniaturization, reduce the power consumption and possess high working speed<sup>(1)</sup>. This development prompted the search for multifunctional materials which possess important properties of different phases in one phase/material<sup>(2), (3)</sup>.

The interaction between the ferroelectric and ferromagnetic orders gives rise to the most interesting and much studied phenomenon called "Magnetoelectric Effect" (ME). The ME response in multiferroic systems is manifested by the appearance of an electric polarization (development of some small electric voltage across the material) upon applying a magnetic field  $H$  (direct ME effect) to the multiferroic material and/or the appearance of a magnetization,  $M$  upon applying an electric field (converse ME effect). The magnitude of the ME voltage (ME response) observed in the multiferroic materials is enormous and is several orders of magnitude higher than that in the single-phase ME materials<sup>(4)</sup>. In this process an applied magnetic field causes the magnetic phase to deform magnetostrictively (direct ME effect), transferring strain to the ferroelectric phase which undergo piezoelectric phenomenon leading to the electric polarization. The converse effect also take place in an analogous manner<sup>(5)</sup>.

Particulate composites [with (0-3) / (3-3) connectivity] are generally prepared by mixing ferromagnetic and ferroelectric materials taken in a proper ratio followed by a thorough grinding in acetone medium. Whereas, in case of laminated composites [with (2-2) connectivity] ferromagnetic and ferroelectric phases are pressed/tape casted alternatively to make bilayer/trilayer. Either ferromagnetic layer is sandwiched between two ferroelectric layers or a ferroelectric layer is sandwiched between two ferromagnetic layers. Compared to particulate composites, laminated multilayered composites have shown very strong magnetoelectric coupling<sup>(6)</sup>. The technique of sandwiching alternating layers of piezoelectric and magnetostrictive phases horizontally helps in the blockage of the leakage current that observed in 0-3 particulate composites. However, one of the disadvantage of the (2-2) connectivity is that it reduces the interfacial area as compared to (0-3) / (3-3) connectivity present in particulate composites leading to increased losses due to poor quality of interface<sup>(7)</sup>. One of the technique of fabricating the layered composites is that the magnetostrictive and piezoelectric sintered pellets are bonded using silver adhesive epoxy followed by a heating at a suitable temperature to ensure proper adhesion between phases layers. Some researchers do this using tape casting method or by co-sintering of pressed powders of each phase<sup>(8), (9)</sup>.

Coming to the applications of magnetoelectric composites, these find very large applications because of their unique properties. The important properties are the ME coefficient is the function of applied bias magnetic field, the ME coefficient is high at the mechanical resonant frequencies, the power loss (or dissipation) in ME composites influences the ME coupling and ME composites have a tunability feature i.e the resonant frequency, impedance and output voltage characteristics could be shifted with bias field. These characteristic features enable potential applications of ME composites in memory devices, gyrators, magnetic sensors, filters, resonators and energy harvest applications<sup>(5)</sup>.

Shara Sowmya et al. have reported the effect of layer architecture on magnetoelectric effect in bi-layer of BCZT/NFO and tri-layer of NFO/BCZT/NFO composites.<sup>(10)</sup> The present investigation investigates the thickness dependent dielectric properties of CFO-PZT-CFO tri-layered structures as there are no reports available on this topic in the literature. The thickness of the PZT layer is varied systematically and the corresponding dielectric properties are measured.

## 2 Methodology

### 2.1 Synthesis

Combustion method was used for the synthesis of piezomagnetic and piezoelectric phases which involves exothermic and self-sustaining thermally-induced anionic redox reaction of xerogel. The xerogel was obtained from aqueous solutions of metal nitrates (oxidizer) and organic complexant (reductant)<sup>(11)</sup>. Proportions between nitrates and complexant were calculated according to the valences of the reacting elements in order to make the ratio of oxidizer/ reductant equal to 1<sup>(12)</sup>. Here we have used nitrates as oxidizers and glycine as fuel, the ratio of oxidizers to fuel is taken according to stoichiometric proportion<sup>(5)</sup>. The reason behind choosing glycine ( $C_2H_5NO_2$ ) as the fuel is that it contains nitrogen which helps in the solubility of oxidizers and the end product will be a uniform mixture of all the metal ions. It also gives better control over the composition<sup>(11)</sup>. The ferrite phase  $CoFe_2O_4$  was prepared by taking stoichiometric amount of AR grade  $Co(NO_3)_2$  and  $Fe(NO_3)_3$  as oxidizers and glycine as the fuel in the ratio of 1:2. The metal nitrates and the fuel are mixed in a beaker of 1000ml, stirred rigorously and then kept on a hot plate present at 200°C and allowed to catch the fire. After burning allowed to cool down to room temperature. The so obtained ash after combustion was crushed and ground to a fine powder and then the powder was sintered at 500 °C for

4 hr to complete the crystallization. Ferroelectric phase  $\text{PbZr}_{0.52}\text{Ti}_{0.48}\text{O}_3$  (PZT) was prepared using Lead nitrate, Zirconium nitrate and Titanium dioxide as oxidizers and glycine was used as fuel. Similar procedure is adopted to prepare PZT as that of  $\text{CoFe}_2\text{O}_4$ .

## 2.2 Pelletization

We have used 1.5cm diameter steel die to make circular pellets. First the cobalt ferrite powder is poured in to the die and gently pressed by hand using press ram. Then, the PZT mixed with diluted PVA is poured in to the die and pressed gently with ram. Over the PZT layer again the cobalt ferrite powder is poured in to the die and pressed gently with ram to make the uniformity in the layer. The die then placed below the hydraulic press and applied a pressure of about 10 ton for 3 min. The pellets so obtained were sintered at 500°C for 2 hr and furnace cooled. Here, the thickness of the ferrite layer is kept constant whereas the thickness of the PZT layer is systematically increased. The table -1 below gives the details of the samples.

**Table 1.** Composites and their representation

| Sample Representation | Bottom Layer Thickness (mm)<br>$\text{CoFe}_2\text{O}_4$ | Middle Layer Thickness (mm)<br>PZT | Top Layer Thickness (mm)<br>$\text{CoFe}_2\text{O}_4$ |
|-----------------------|----------------------------------------------------------|------------------------------------|-------------------------------------------------------|
| CPC1                  | 2                                                        | 0.5                                | 2                                                     |
| CPC2                  | 2                                                        | 1.0                                | 2                                                     |
| CPC3                  | 2                                                        | 1.5                                | 2                                                     |
| CPC4                  | 2                                                        | 2.0                                | 2                                                     |
| CPC5                  | 2                                                        | 2.5                                | 2                                                     |
| CPC6                  | 2                                                        | 3.0                                | 2                                                     |

## 2.3 Characterization

To study the crystal structure, X-ray diffraction technique was used using Rigaku X-ray diffractometer with  $\text{CuK}\alpha$  radiation ( $\lambda=1.5406 \text{ \AA}$ ) on powder samples of ferrite and ferroelectric samples.

## 2.4 Dielectric measurements

The dielectric measurements were carried out on tri-layered pellet samples at room temperature using Wayne Kerr 6500B impedance analyzer from 10 Hz to 10 MHz. The top and bottom surfaces of the pallet were gently polished and applied conducting silver paste on both the surfaces. Thin pure copper sheets of 2cm x 2cm were placed on either side of the silver pasted surfaces of the pallet and this assembly was kept in a sample holder. By varying the frequency of the input voltage the capacitance ( $C_p$ ), loss tangent ( $\tan\delta$ ) and AC resistance ( $R$ ) are measured. Other dielectric properties were calculated using these three measured parameters.

# 3 Results and Discussion

## 3.1 X-Ray diffraction study

The synthesized powder of cobalt ferrite and PZT were examined by using the X-ray diffraction technique to understand their crystal structure. The X-ray diffraction pattern of the cobalt ferrite and PZT obtained are as shown in the Figure 1 and Figure 2 respectively.

The X-ray peaks are the characteristics of the ferrite (JCPDS Card # 22-1086 for cobalt ferrite) phase. By observing the dominant peaks in the XRD patterns at particular  $2\theta$  values it is confirmed that the cubic spinel structure of ferrite phase is completely formed. The dominant (311) peak of spinel structure of ferrite phase is rightly placed at  $2\theta$  value of  $36^\circ$ . The high intensity of all the peaks in ferrite phase indicates that the crystallization process is completed. The broadening of the peaks at base line also reveal that the particle size is sufficiently small. For face centered cubic structure, the cyclic sum of Miller indices viz.,  $(h+k)$ ,  $(k+l)$  and  $(l+h)$  is an even integer. This fact is clearly observed in all the samples<sup>(13)</sup>.

It is clear from the Figure 2 that the crystal clear perovskite structure is formed in the sample with characteristic dominant peak (110) precisely placed at its proper  $2\theta$  value. All other peaks are also placed precisely at their usual  $2\theta$  values. The high intensity of the peaks indicates the completion of crystal structure formation. Unidentified phases such as  $\text{ZrO}_2$  or  $\text{PbO}$  are not seen in the XRD pattern. The specific ratio of (Zr/Ti) was deliberately fixed at (52/48) as the material exhibit the morphotropic

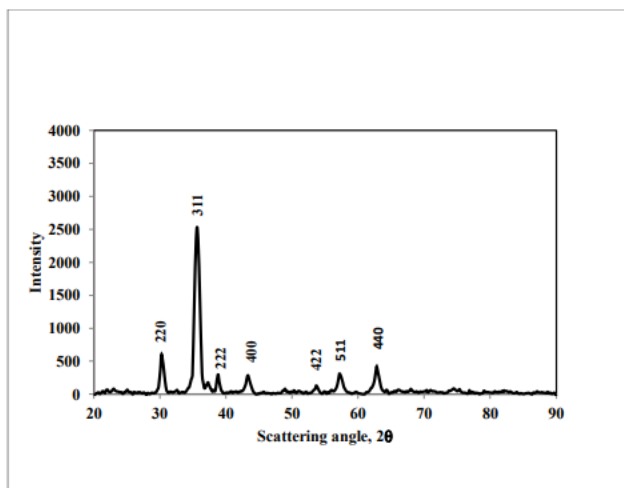


Fig 1. X-Ray diffraction pattern of  $\text{CoFe}_2\text{O}_4$

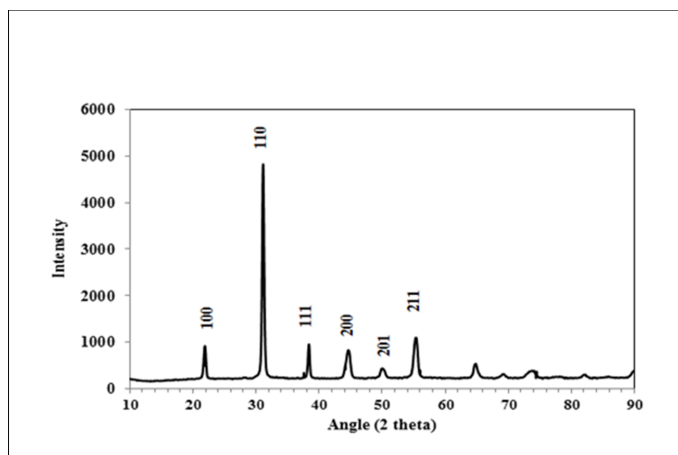


Fig 2. X-Ray diffraction pattern of PZT.

phase boundary at this particular ratio so that the tetragonal and rhombohedral phases co-exists and the material shows high values of polarizability and piezoelectric constants. Distinct splitting in the (111) peak is not observed indicating that the tetragonal phase is not formed<sup>(14)</sup>.

### 3.2 Dielectric Studies

The variation of dielectric constant ( $\epsilon'$ ) with frequency for the present tri-layered CFO/PZT/CFO composites is as shown in the Figure 3. It is clear from the figure that the dielectric constant decreases rapidly with increase in frequency at low frequency region and then remains almost independent of frequency at higher frequency region. The dielectric dispersion observed at low frequency side is attributed to the Maxwell-Wagner interfacial type polarization and is in good agreement with the Koops phenomenological theory<sup>(15)</sup>. Space charge polarization could also occur around grain boundaries due to inhomogeneous structure and that works in favour of dielectric dispersion<sup>(16)</sup>,<sup>(17)</sup>. It is also observed from the Figure 3 that the dielectric constant increases with increase in the thickness of the middle PZT layer. This is due to the fact that cobalt ferrite is a low dielectric constant material whereas PZT is a very high dielectric constant material<sup>(18)</sup>. Therefore it is obvious to expect this result.

The decrease of dielectric constant with increase in frequency can be explained as follows- At very low frequency region the space charge polarization and the interfacial polarization are active. However, when the frequency increases, these two polarization mechanisms fail to follow the external ac field and starts lag behind the field. Thus, the value of dielectric constant rapidly falls when the frequency of the applied field increases. When the frequency of the applied ac electric field exceeds a certain value, these two polarization mechanism even ceases. At higher frequency range the dielectric constant becomes independent of the applied electric field, because, at this region only ionic and electronic polarization mechanisms are active but their contribution is very low to the phenomenon of dielectric<sup>(19)</sup>. We can treat this tri-layered structure as a parallel plate capacitor with a dielectric medium in it. As the conductivity of CFO layer is much higher than the conductivity of PZT layer and CFO layers are sandwiching the PZT layer to make a parallel plate capacitor. The capacitance of the capacitor increases  $\epsilon$  times the capacitance without dielectric, the increase of dielectric constant with the thickness of the PZT layer in the present case is justified.

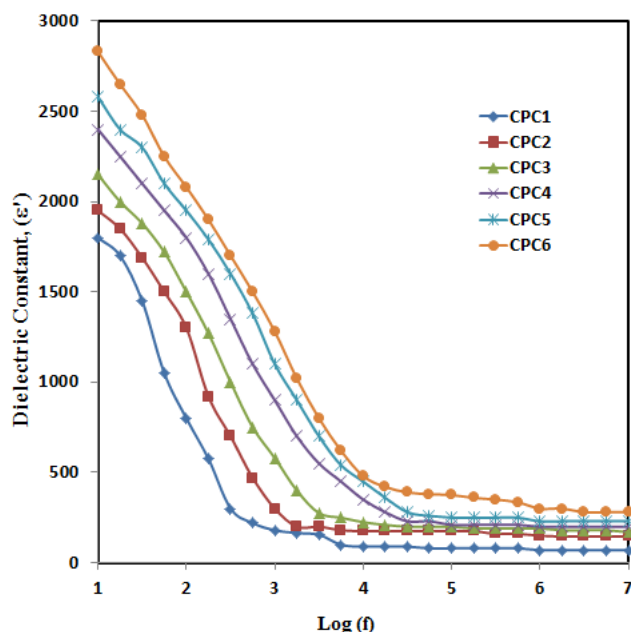


**Fig 3.** Variation of dielectric constant with frequency for CFO/PZT/CFO tri-layered composites.

The variation of dielectric loss tangent ( $\tan\delta$ ) with applied ac frequency is as shown in the Figure 4 for the present CFO/PZT/CFO tri-layered composites. It is observed from the Figure 4 that the tendency of showing loss maxima is seen in the samples with low thickness of PZT layer in the composites in the frequency range 10Hz-1kHz. This tendency found to be less observed in the samples with higher thickness of PZT layer in the composites. The loss maxima seen very clearly in the sample CPC1 (PZT thickness =0.5 mm) and least seen in the sample CPC6 (PZT thickness =3.0mm). It is also observed that the loss maxima shift towards low frequency side as the thickness of the PZT layer increases. With the increase in the frequency of the applied ac electrical field a overall decrease in the dielectric loss is observed. A similar type of dielectric behaviour is reported to similar systems<sup>(20), (21)</sup>.

Both the dielectric constant and electrical conductivity phenomenon are basically electrical transport properties and it could be assumed that the same mechanism is responsible for both. A strong correlation between conduction mechanism and the dielectric behavior has been reported for ceramics<sup>(22), (23)</sup>. The electronic exchange between ions such as  $\text{Fe}^{2+}/\text{Fe}^{3+}$ ,  $\text{Co}^{2+}/\text{Co}^{3+}$  results in local displacements, which determine the polarization in ferrites/ ceramics. When the hopping frequency of electrons between these ions matches with the frequency of the applied ac electrical field the resonance takes place leading to the maximum dielectric loss. The shifting of dielectric maxima towards low frequency side is attributed to the increase in the resistivity of the composites due to increase in the thickness of the PZT layer which reduces the number of electrons available for hopping mechanism<sup>(20), (23)</sup>. The overall decrease of dielectric loss with increase in frequency is explained in analogous to the decrease of dielectric constant with increase in frequency.

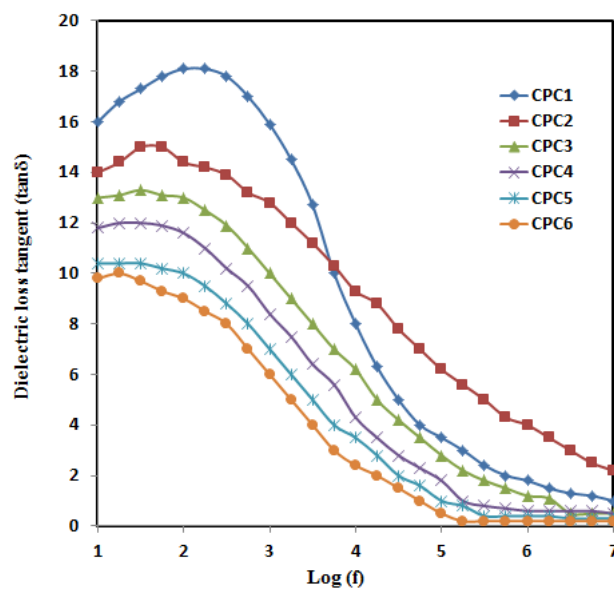


Fig 4. Variation of dielectric loss tangent ( $\tan\delta$ ) with frequency for CFO/PZT/CFO tri-layered composites.

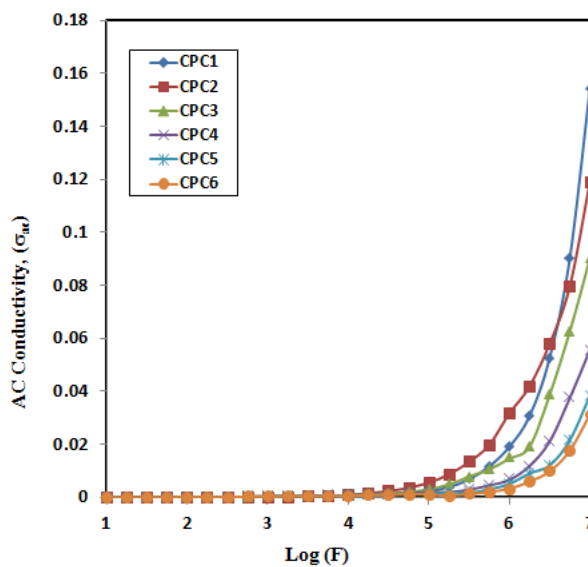


Fig 5. Variation of AC conductivity with frequency for CFO/PZT/CFO tri-layered composites.

The variation of AC conductivity with frequency is shown in the Figure 5. It is clear from the figure that the AC electrical conductivity of the present composites studied remains unaltered up to 10kHz of the applied AC field frequency and beyond that the conductivity rises abruptly. In this tri-layered structure, PZT layer is sandwiched between two CFO layers. The conductivity of CFO layers is much higher than that of PZT layer. The conductivity in CFO layers depends upon the hopping frequency of charge carriers between  $\text{Fe}^{2+}/\text{Fe}^{3+}$  and  $\text{Co}^{2+}/\text{Co}^{3+}$ . Whereas the conductivity in PZT depends upon the availability of free charge carriers and stress/strain condition of the PZT layer. Since the stress/strain conditions do not change much under the influence of the external AC field and generally the number of free charge carriers in PZT are less as compared to CFO layer, the contribution of PZT layer to the conduction mechanism is very less. Therefore, it is the hopping mechanisms of charge carriers in CFO layers which contribute much to the conduction process. Thus, the abrupt increase in the conductivity beyond 10kHz frequency of AC field is attributed to the abrupt increase in the hopping frequency of charge carriers in the CFO layers<sup>(19), (24)</sup>.

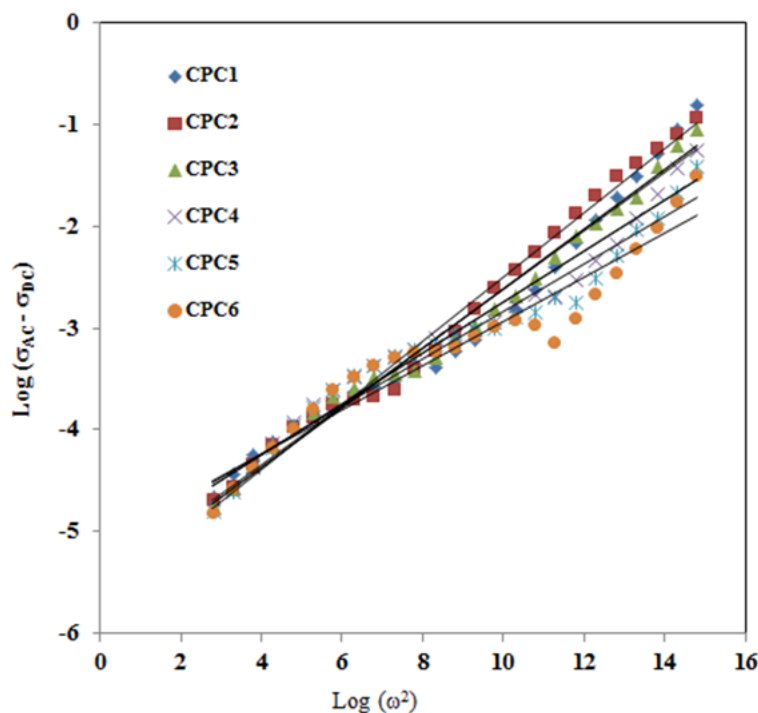


Fig 6. Variation of  $(\text{Log}(\sigma_{AC} - \sigma_{DC}))$  with  $\text{Log}(\omega^2)$  for CFO/PZT/CFO tri-layered composites.

Figure 6 shows the plot of  $(\text{Log}(\sigma_{AC} - \sigma_{DC}))$  with  $\text{Log}(\omega^2)$  for CFO/PZT/CFO tri-layered composites. The result is a straight line in all the samples studied. This proves that the conduction mechanism in these samples is of small polaron type<sup>(24)</sup>.

## 4 Conclusion

Cobalt ferrite and PZT materials are synthesized using combustion method. XRD patterns of CFO and PZT show the single phase formation. Dielectric constant ( $\epsilon'$ ) decreases with increase in frequency of applied AC electrical field and is attributed to the failure of hopping frequency of charge carriers to follow the frequency of applied field. The dielectric dispersion observed at low frequency region is due to the space charge at the grain boundaries as well as at interface of the layers. Dielectric loss follows the same trend of the dielectric constant, however some frequency maxima found at lower frequency due to the resonance of hopping and applied field frequency. AC conductivity increases abruptly beyond 100 kHz of frequency of applied field due to the increase in hopping frequency of charge carriers and it is also confirmed that the conduction process is of small polaron type.

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