

Electric and Magnetic Properties of [(NCZF)_{1-x}(Na(ac.ac))_x] Nanocomposite

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Abstract

The pure spinel phase of Ni_{0.45}Co_{0.2}Zn_{0.35}F₂O₄ [NCZF] was prepared by coprecipitation method. The nanocomposite [(NCZF)_{1-x}(Na(ac.ac))_x] ($x = 0\%$, 20%, 40%, 60%, 80%, 100%) was prepared by mixing two phases: ferrite phase and ferroelectric phase. The magnetic characters of composites performed using vibrating sample magnetometry (VSM) at room temperature with maximum applied field up to 2 kG. For smaller particle size < 30.89 nm, the coercivity increases rapidly with increasing particle size and reaching maximum value of 99.9 G at 30.89 nm and then decrease with particle size for larger particles > 30.89 nm. The saturation magnetization M_s decreases linearly with increasing particle size. The effect of temperature on the electrical resistivity of the different composites samples was studied. It is noted that, the resistivity decreases with rising temperature to have a minimum value at ferroelectric transition temperature, above this value the resistivity increases. The dielectric constant increases with increasing temperature for all composites. The dielectric loss $\tan(\delta)$ as a function of temperature sharply with temperature reached to maximum value at ferroelectric phase transition then decrease again.

Keywords

Ferroelectric, Ferrite, Composites, DC Conductivity

1. Introduction

Nowadays technology and science are focusing on three global problems [1]: energy, environment and health. Magnetic materials can contribute in all of these cases; cubic spinel ferrite is a group of technologically important magnetic materials having applications from microwave to radio frequencies. Structural,

electrical, and magnetic properties of these materials effectively depend upon their stoichiometry methods of synthesis, and cationic distributions [2]. The ability to control magnetism with an electric field has drawn wide research interest due to the potential it holds in lowering the power consumption of magnetic devices [3]. The coupling between electric and magnetic fields in matter was initially discovered by Rontgen in 1888 [4] when he found that a moving dielectric placed in an electric field has become magnetized, and then was theoretically described by Curie in 1894 [5] on the base of crystal symmetry considerations. The discovery of the phenomenon of ferroelectricity was made by Valasek in 1920 [6]. Fox and Scott [7] have also shown that ferroelectricity can produce magnetic field and vice versa. This has provoked interest in study and understanding of “multiferroics” and “magnetoelectric” materials. **Magnetoelectric materials** are the materials, which are simultaneously ferrimagnetic and ferroelectric. Such materials can be used for all the potential applications. In addition, these materials also show the magnetoelectric effect [8]. The **magnetoelectric effect** (ME) defined by the electric field (E) induced under application of a magnetic field (H) or vice versa. Electronic and magnetic materials can be found everywhere in modern technology and industry. These ME materials could potentially be used for fabricating new types of sensors, actuators and data storage devices. Magnetoelectric materials are an important class of materials for such applications, and can offer potential revolutionary device. **Table 1** shows some composite multiferroic materials.

The aim of our study is to fabricate new magnetoelectric nanocomposite, which has a high sensitivity to any change in electric field.

2. Experimental

Sample Preparation

$\text{Ni}_{0.45}\text{Co}_{0.2}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$ ferrite particles was prepared by chemical precipitation of $[\text{NiCl}_2, \text{CoCl}_2, \text{ZnCl}_2]$ and $[\text{FeCl}_3]$ (1:2 molar ratio) by addition 25% ammonia

Table 1. Composite multiferroic materials [31] [32].

Material	Characteristic	Examples	α ($\text{mV cm}^{-1}\text{Oe}^{-1}$)
Particulate Ceramic Composite		BTO/CFO	130
	A variety of ferroelectric/ferrite compositions have been fabricated consisting of the ferroelectric phases, such as BaTiO_3 (BTO),	CuFO/BTO	425
	$\text{PbZr}_{(1-x)}\text{Ti}_x\text{O}_3$ (PZT), (PVDF),	CFO/BTO	200
	PbTiO_3 (PT) and ferromagnetic phases such as CoFe_2O_4 (CFO),	PZT/CFO	--
	Terfenol-D($\text{Tb}_{1-x}\text{D}_x\text{Fe}_2$),	$\text{Pb}_{0.95}\text{Sr}_{0.05}\text{Ni}_{0.06}$	--
	NiFe_2O_4 (NFO), CuFe_2O_4 (CuFO),	$\text{Zr}_{0.49}\text{Ti}_{0.4}\text{O}_3/\text{CFO}$	--
	LaMnO_3 (LMO),	Terfenol-D/PVDF	70
		$\text{BTO}/\text{Ni}_{0.94}\text{Co}_{0.01}\text{Cu}_{0.05}\text{Fe}_2\text{O}_4$	1430
		$\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Nb}_2\text{O}_6/\text{CFO}$	0.64

solution [9]. The volume of reaction mixture have been mixed using magnetic stirring during continuous slow addition of 25 ml ammonia solution and the heating continued for thirty minutes. The powder precipitate decanted in and washed with 500 ml distilled water [10]. Sodium acetylacetonate prepared by dissolving 40 g. (1 mole) of sodium hydroxide in 50 ml. of water and adding to this 200 ml. of methanol. This solution is added, slowly with hand stirring, to 100 g. (1 mole) of acetyl acetone contained in a 500-ml. flask. The creamy-white crystalline salt separates from solution immediately. The flask is stoppered and cooled in ice (or in a refrigerator) for 2 hours or overnight. The sodium salt is collected on a Büchner funnel and washed with two small portions of cold methanol. After the salt is air dried, it dried further either by allowing it to stand in a vacuum desiccator at room temperature or by heating it in a vacuum oven at 100° for 3 hours. The anhydrous product, which is stable, can be stored indefinitely in a stoppered jar [11]. $\text{Ni}_{0.45}\text{Co}_{0.2}\text{Zn}_{0.35}\text{Fe}_3\text{O}_4$ [NCZF] and Sodium Acetylacetonate [Na(ac.ac)] were mixed with the percentage according to formula $[(\text{NCZF})_{1-x}(\text{Na}(\text{ac}.\text{ac}))_x]$, ($x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$). The composites mixed and ground very well for 12 hours using agent mortar. Finally, all samples were ground and pressed at room temperature into tablets under of 10 Ton/cm² of diameters 1 cm and 0.4 cm thickness.

3. Results and Discussion

3.1. Vibrating Sample Magnetometer (VSM)

Figure 1 shows the magnetic hysteresis loop of nanocomposite samples at room temperature with maximum applied up to 2 kG. The magnetic properties such as saturation magnetization (M_s) and coercivity (H_c) calculated from the hysteresis loop. The insets of the figure show the expanded region around the origin with different field ranged $0:\pm 600$ G or in order to make the coercivity more visible. For the pure ferrite sample, the coercivity at room temperature as derived from M-H loops was 89 G while for $x = 40\%$, $[(\text{NCZF})_{1-x} + (\text{Na}(\text{ac}.\text{ac}))_x]$ composite was 99 G. The saturation magnetization M_s is at room temperature 25.5 emu/gm for the pure ferrite. The relatively large coercivity of this pure ferrite sample are constant with the pronounced growth of magnetic anisotropy caused the inhibiting the alignment of the magnetic moment along the applied field direction 24.26 G [12].

The coercivity of nanoparticle also studied as a function of particle size at room temperature as shown in the **Figure 2** which shows that the coercivity increases with increase the particle size reaching maximum value of 99.9 G at 30.89 nm and then decreases for large particle size.

Figure 2 shows also the saturation magnetization M_s decrease linearly with increase in particle size. This is due to the following:

It is well established that the permeability of polycrystalline ferrite consists of two different mechanisms. The magnetic domain rotational component and the domain wall motion are sensitive to both the ferrite grain size and the post

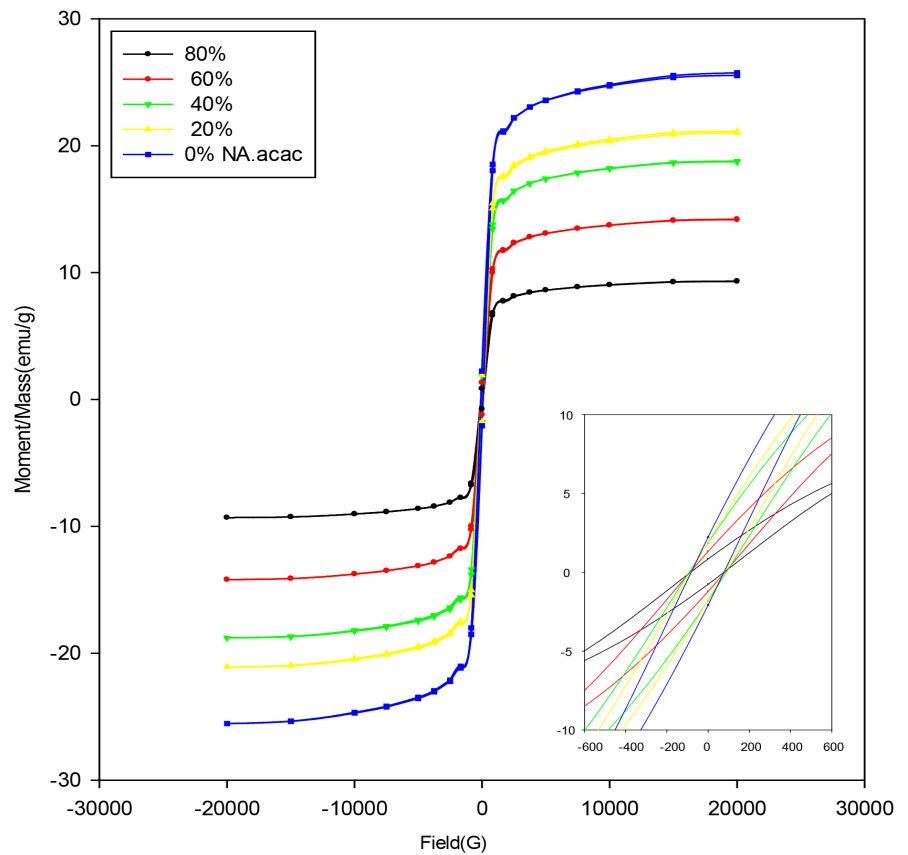


Figure 1. The vibrating sample magnetometry (VSM) at room temperature with maximum applied up to 2 kG, for composite samples $[NCZF_{(1-x)} + Na(ac.ac)_x]$, $x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$.

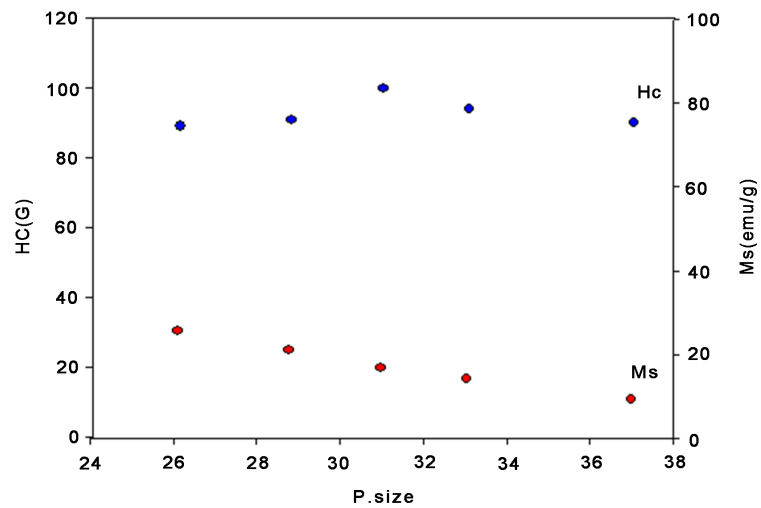


Figure 2. The coercivity of nanoparticle (H_c) and the saturation magnetization (M_s) studied as a function of particle size at room temperature.

sintering density. The grain size is more important parameter affecting the magnetic properties of ferrites.

Globus [13] [14] assumed that the permeability due to the wall motion is like-

ly to be linearly dependent on the grain size. But in the present work, the decrease of M_s with increasing particle size can be explained by taking into consideration the cation distribution and domain wall motion. The presence of Na(ac.ac) contents retard the domain wall motion in the composite under the magnetic field, which decreased the interaction of A-B in ferrites and hence the decrease of M_s of the composites.

3.2. The Electrical and Thermoelectrically Studies

3.2.1. Ferro-Electric Hysteresis Loops of Composite Samples

The ferroelectric hysteresis loops were display using Sawyer-Tower modified circuit, illustrated in **Figure 3**. It is observed that the inclination of hysteresis loop increases with increasing Na.ac.ac content in the composite.

Figure 4 illustrates the effect of Na(ac.ac) contents content on the polarization. The increase of the inclination (polarization) with increasing $x\%$ content is due to the following: In previous work the ferroelectric hysteresis loop of Na(ac.ac) is displayed [15].

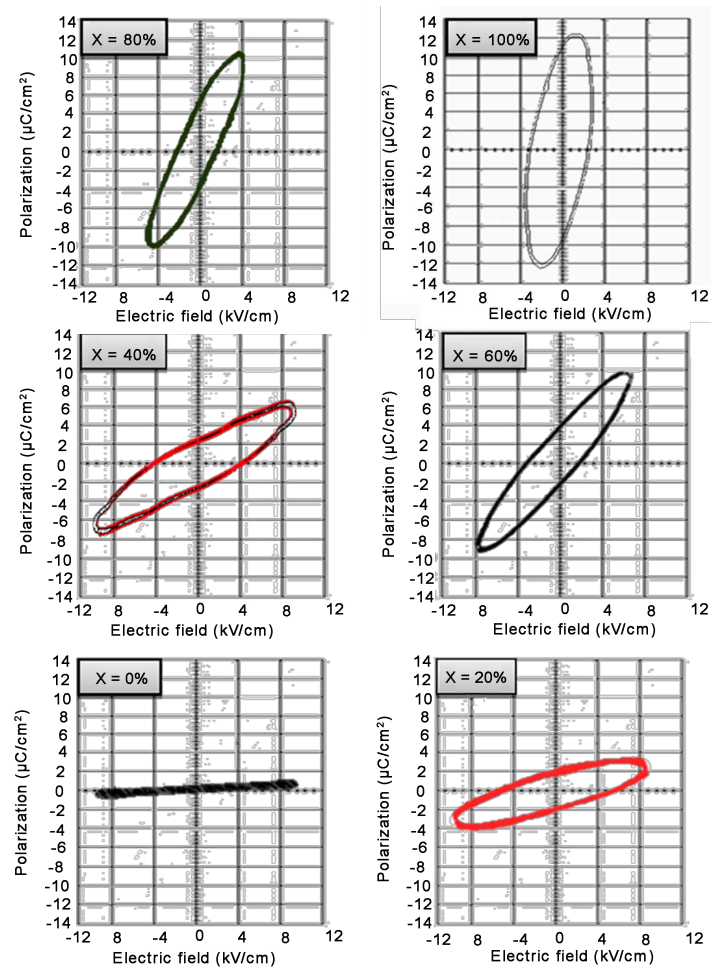


Figure 3. The ferroelectric hysteresis loops where display on the cathode ray oscilloscope for composite samples $[\text{NCZF}_{(1-x)} + \text{Na(ac.ac)}_{(x)}]$, $x = 0\%$, 20%, 40%, 60%, 80%, 100%.

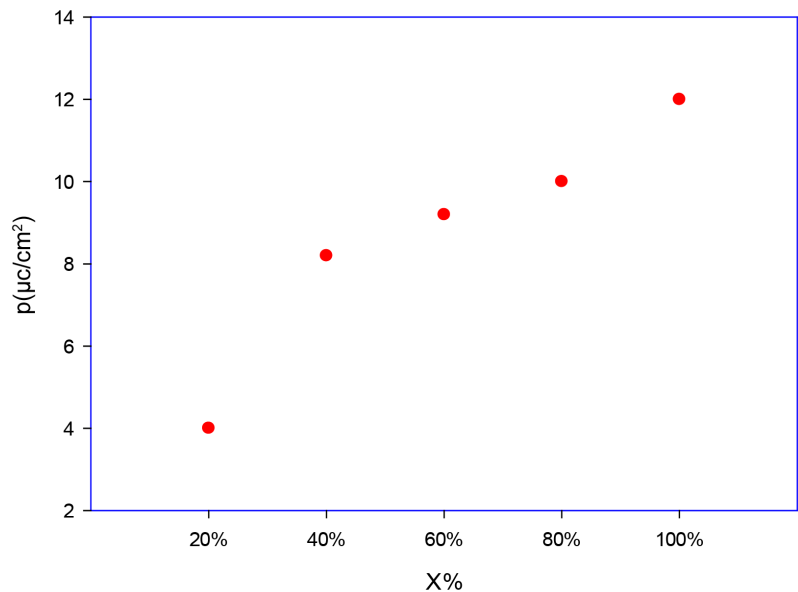


Figure 4. The effect of Na(ac.ac) contents on the polarization of the composite samples.

Therefore the increase x% content in the composite increases number of 180° and 90° domains in the direction of the applied ac field. This domain is correlated with the induced polarization on the sample surface giving rise to polarization. It is obvious that role of ferrite in the composite is antiferroelectric material and this confirmed the polarization of the composite is due to the piezoelectric Na(ac.ac).

3.2.2. The Pyroelectric phenomena of Composite Samples

Figure 5 shows that The variation of pyroelectric charge of composite sample $[(\text{NCZF})_{1-x} + (\text{Na}(\text{ac.ac}))_x]$, $x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$. The pyroelectric charge increases by increasing temperature up to maximum value at nearly transition temperature T_c of Na(ac.ac) phase. The reason of increase in pyroelectric charge by heat treatment might be due to the displacement of ion by thermal agitation, leading to the increase of ionic distance (metal-oxygen bond). The change in distance increases the dipole moment resulting; the increase of polarization to attain maximum at T_c . Above T_c the decrease in pyroelectric charge is nearly reached to zero at 120°C. This decrease may be due to phase transition from ferroelectric to paraelectric state. The pure ferrite sample is not pyroelectric samples due to the absence of ferroelectric phase. The presence of ferrite phase in composite shifts the transition temperature to higher value of about 90°C [16] whereas for pure Na(ac.ac) the transition temperature of about 60°C. **Figure 6** shows that the value of pyroelectric charge increases to maximum value by increasing Na(ac.ac) content. These phenomena may be due to the increase of dipole moment by increasing ferroelectric phase content. The peak value of pyroelectric charges is attributed to change of crystal structure from orthorhombic to triclinic phase.

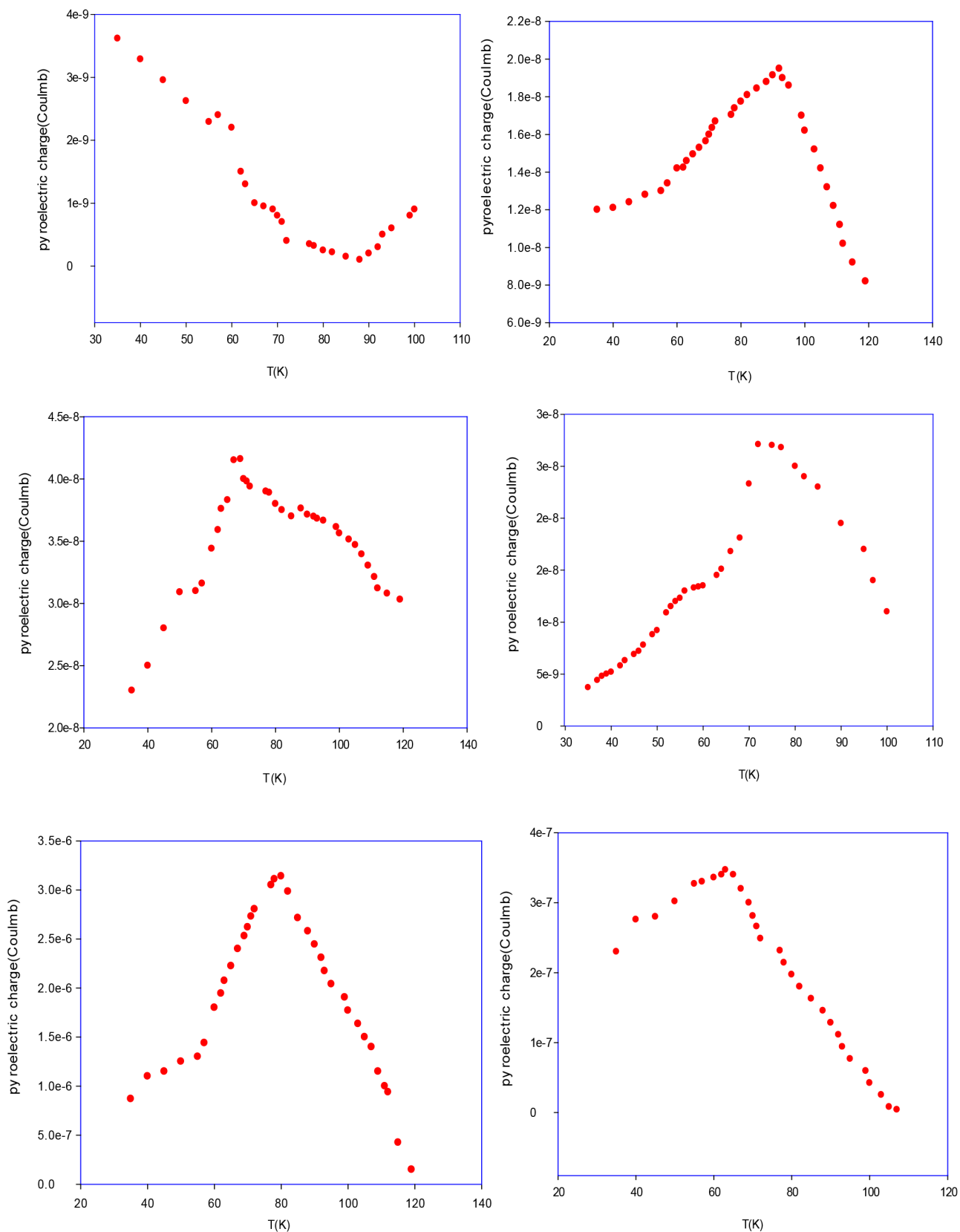


Figure 5. The variation of pyroelectric charge as a function of temperature of composite $[NCZF_{(1-x)} + Na(ac.ac)_{(x)}]$, $x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$.

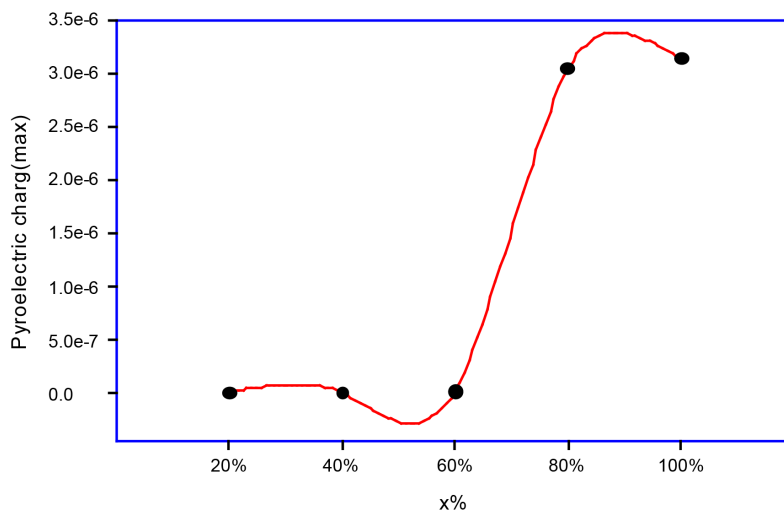
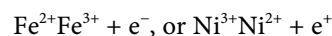


Figure 6. The pyroelectric charge (maximum value) as a function of x% for composites $[\text{NCZF}_{(1-x)} + \text{Na}(\text{ac.ac})_{(x)}]$, $x = 20\%, 40\%, 60\%, 80\%, 100\%$.

3.2.3. Effect of Temperature on the Electrical Resistivity of the Composites

Figure 7 illustrates the effect of temperature on the electrical resistivity of the different composites $[\text{NCZF}_{(1-x)} + \text{Na}(\text{ac.ac})_{(x)}]$, $x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$ $\text{Na}(\text{ac.ac})$. It is noted that the resistivity decreases with rising temperature to have a minimum value at transition temperature of the composite, above the transition temperature the resistivity increases, This can be explain as follows, in general, spinel ferrites behave with temperature as semiconductor but their conductivity is better being described by localized electron model rather than collective band model. The conductivity is commonly attributed to presence of Fe^{2+} ions [17], and sometimes-in Ni containing ferrites to the presence of Ni^{2+} also. The conductivity arise due to the mobility of the extra electron which comes from Fe^{2+} (or sometimes extra hole in positive charge containing ferrites), through the crystal lattice. The conductivity by hopping mechanism taking place between divalent and trivalent ions of the same element present on equivalent crystallographic site (B-sites) is expressed as follows [18] [19]:



Some evidence of existence of divalent metal ions on the octahedral sites is found in the IR spectra of the ferrites used to prepare the present composites [20].

In pure ferrite ($x = 0\%$) there are two transition temperature which is characteristic behavior of the present ferrite. The high is always attributed to the magnetic phase transition of ferrite from ferrimagnetic state to paramagnetic state [21]. The low transition temperature is attributed to the spin reorientation phase transition below Curie temperature [22]. This leads to the change in the conduction mechanism [23]. The minimum value of resistivity at the transition temperature as indicated in **Table 2** is due to transition of the crystal structure of $\text{Na}(\text{ac.ac})$ in the composite from orthorhombic to triclinic phase [22].

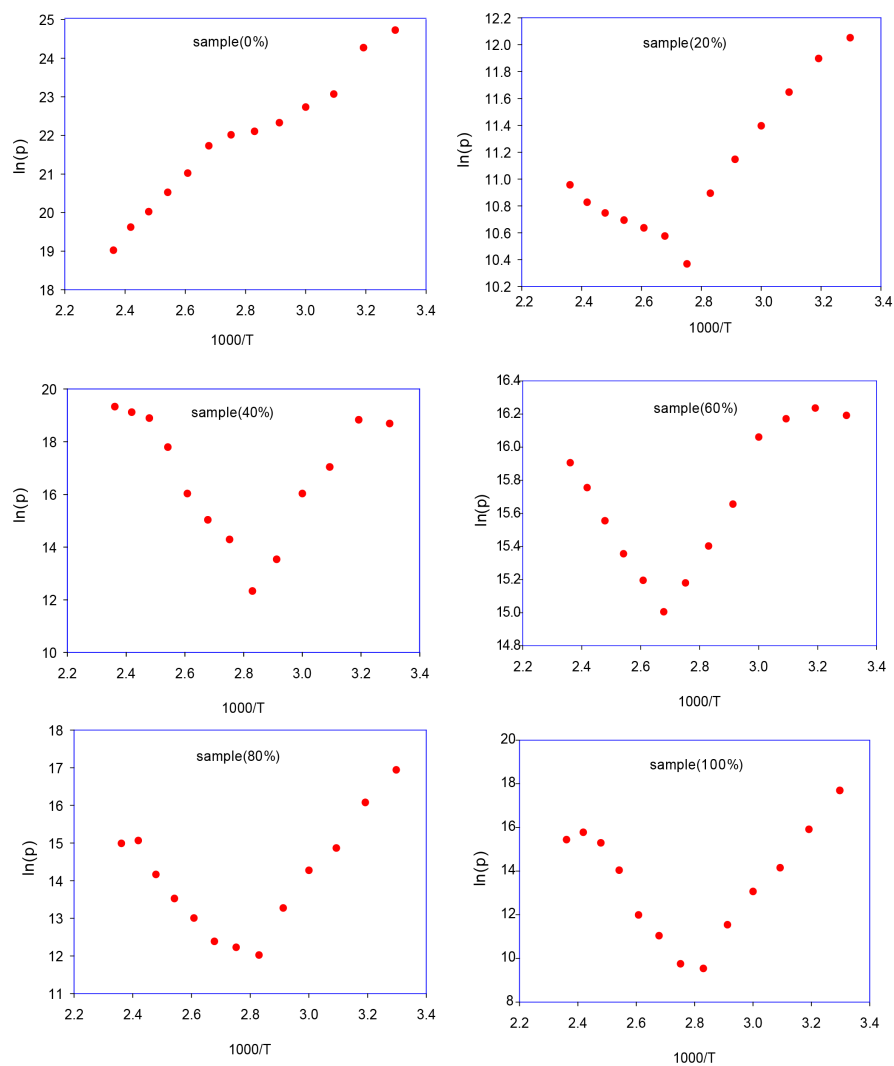


Figure 7. The relation of DC resistivity on reciprocal temperature for composite samples $[\text{NCZF}_{(1-x)} + \text{Na}(\text{ac.ac})_{(x)}]$, $x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$.

Table 2. The activation energy for each transition temperature for composite samples $[\text{NCZF}_{(1-x)} + \text{Na}(\text{ac.ac})_{(x)}]$, $x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$.

X% Na(ac.ac)	Ferroelectric T_C (k)	Activation energy (ev)
Pure ferrite	314	0.35
	373	0.37
20% Na(ac.ac)	370	0.38
40% Na(ac.ac)	363	0.43
60% Na(ac.ac)	357	0.54
80% Na(ac.ac)	363	0.62
100% Na(ac.ac)	353	0.76

The increase of electrical resistivity above the transition temperature is due to conduction of charge carrier is correlated with the band conduction mechanism. The activation energy is listed in previous table for each composite before the

transition temperature with indicate the increase of Na(ac.ac) content increases the activation energy leading to increase the electrical resistivity of the composites.

3.2.4. Effect of Temperature on the Dielectric Constant of the Composites

The effect of temperature on the dielectric constant on the present composites is shown in **Figure 8**. It's obvious that the dielectric constant increases with increasing temperature for all composites. This can be explained on the basis that as the temperature increases the hopping between Fe^{2+} and Fe^{3+} ions on the octahedral-site it thermally activated, this electron hopping causes local displacement in the direction of the electrical field and as a result the dielectric polarization increase therefore dielectric constant increase [24] [25].

The dielectric constant of the composite increase than that of ferrite only as the temperature increases. It is known that Na^+ cation are weakly bounded with acetylacetonate ($\text{CH}_3\text{-C-CH}_2\text{-C-CH}_3$) anions. This facilitate to those ions to jump from

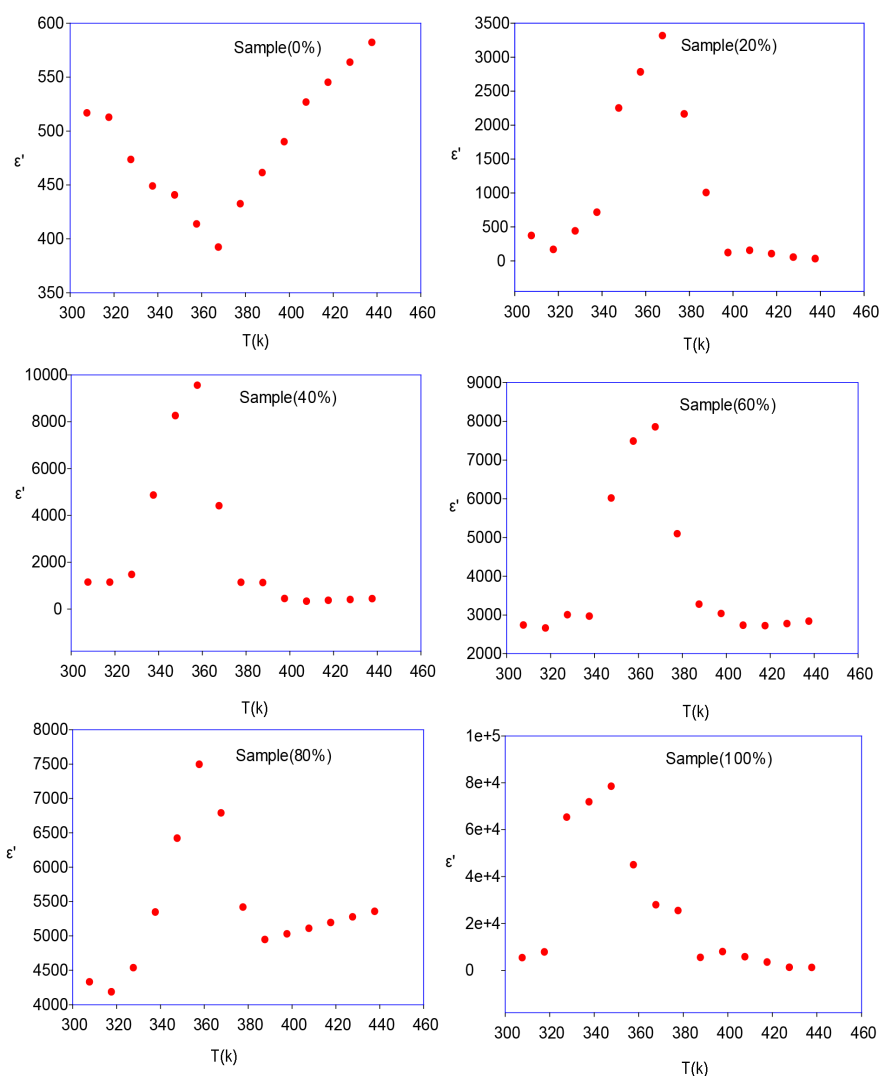


Figure 8. The dielectric constant (ϵ') as a function of temperature for composite samples $[\text{NCZF}_{(1-x)} + \text{Na}(\text{ac.ac})_{(x)}]$, $x = 0\%$, 20% , 40% , 60% , 80% , 100% .

site to the existed vacancies in the crystal. This gave rise to dielectric polarization in the present composite beside the hopping conduction of ferrite. The peak value of the present composite with temperature due to the transition of Na(ac.ac) crystalline from orthorhombic to triclinic phase above the transition temperature [26] for each composite (20% - 100% Na(ac.ac)) as shown in **Figure 8**. However beyond transition temperature the ions and electron are less oriented towards the field direction in the triclinic phase of paraelectric state of the composite and hence the dielectric constant decrease.

3.2.5. Effect of Temperature on the Dielectric Loss of the Composites

The dielectric loss as a function of temperature is noticed in **Figure 9**. It is obvious that the dielectric loss $\tan(\delta)$ increases sharply with temperature up to 40% Na(ac.ac). This peak value decreases again for $x > 40\%$. The increase in $\tan(\delta)$ with temperature can be explained on the basis that as the temperature increases the hopping electrons between Fe^{2+} and Fe^{3+} ions on the octahedral sites

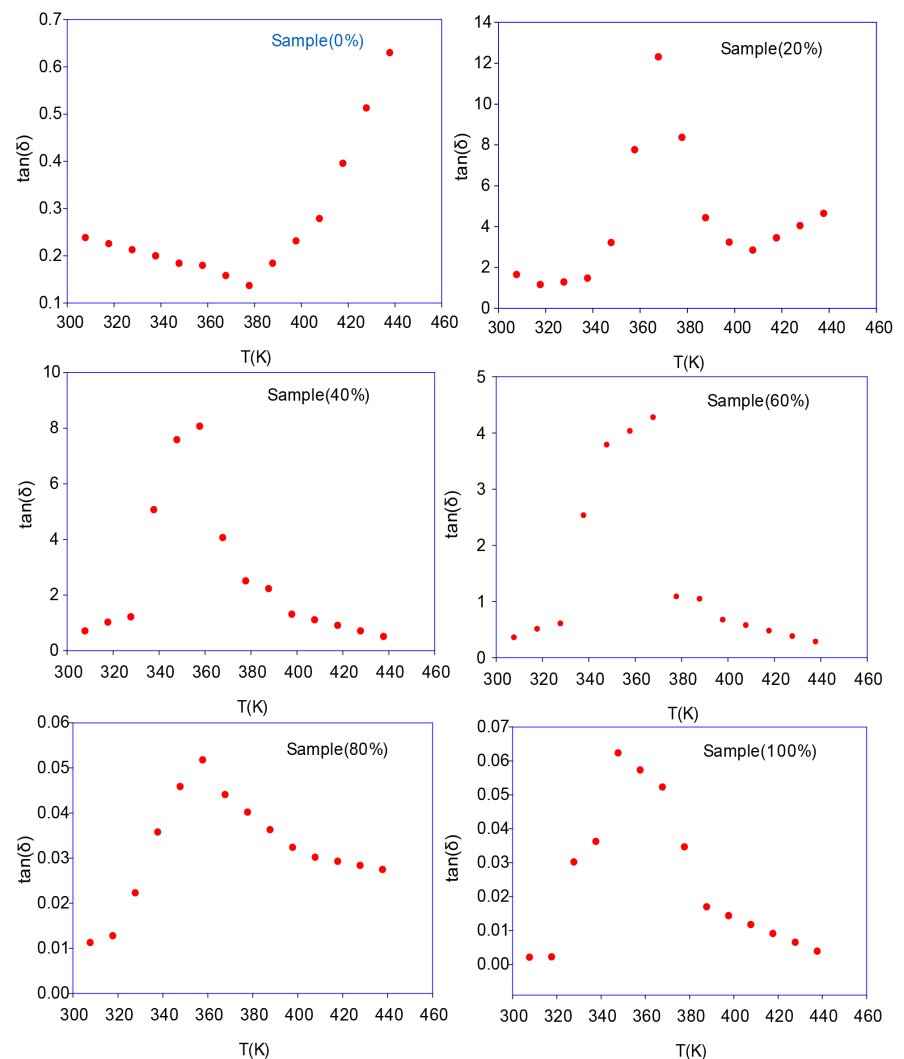


Figure 9. The dielectric loss as a function of temperature for the composite $[(\text{NCZF})_{1-x} + (\text{Na(ac.ac)})_x]$.

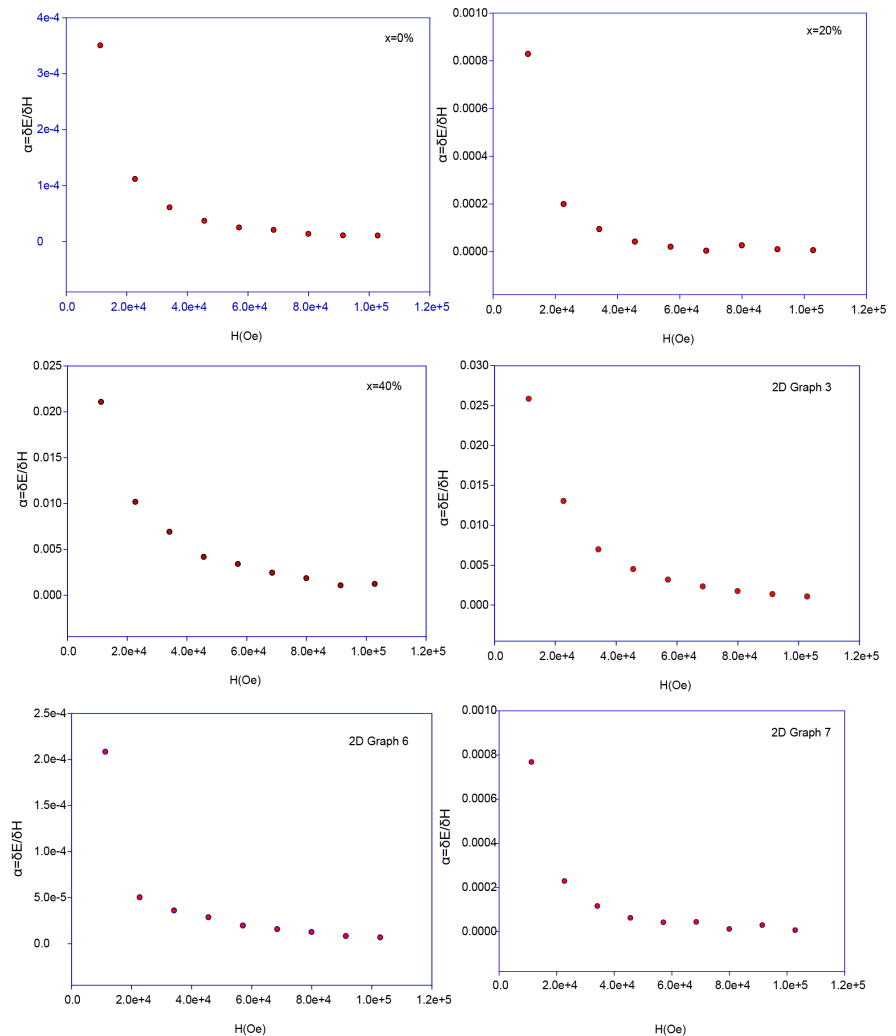


Figure 10. The dependence of ME coefficient on the applied magnetic field of various Na(ac.ac) contents for composite samples $[\text{NCZF}_{(1-x)} + \text{Na}(\text{ac.ac})_{(x)}]$, $x = 0\%, 20\%, 40\%, 60\%, 80\%, 100\%$.

is thermally activated. These electrons caused a local displacement in the direction of the external applied field. The loss peak occurs when the applied field is in phase with the dielectric and verifying the condition $W\tau = 1$ is stats field where $W\tau = 2\pi f$, f is the frequency of the applied field and τ the relaxation time. This is related to jump probability per unit time ρ , by an equation $\tau = \rho/2$. At the temperature 370 K the composite is transferred from ferroelectric to paraelectric states of Na(ac.ac). The jump probability increases at low temperature and the decreases of higher temperature. This explain the increase the value of $\tan(\delta)$ at the high temperature of composite, The decrease of $\tan(\delta)$ with increasing Na(ac.ac) above 40% may be due to decrease of the jumping probability of Na ions.

3.3. The Effect of Magnetization

Magnetolectric Effect (ME) of the Composites

ME is property of composite and is absent in their constituent phases. The de-

formation of ferrite phase causes the polarization of piezoelectric phase whereas electrical polarization material causes change in the magnetization of ferrite phase due to mechanical coupling of piezomagnetic and piezoelectrical phase. **Figure 10** indicates the dependence of ME coefficient on the applied magnetic field of various Na(ac.ac) contents. It is observed the ME coefficient decreases by increase of magnetic field. This may be due to low resistivity of ferrite phase than that the ferroelectric Na(ac.ac) phase because of Na(ac.ac) is a piezo and ferroelectric material [27] [28]. Hence the charges developed in piezoelectric phase leak out through low resistance path. It is observed also ME coefficient decreased as Na(ac.ac) increased. The behavior of the present result is in agreement to those in previous work. These composites can be used as sensor in detecting electromagnetic waves [29] [30].

4. Conclusion

The addition (Na(ac.ac)) has considerable effect on electric and magnetic properties of both ferrite and (Na(ac.ac)). The presence of Na(ac.ac) content decreases the A-B exchange interaction; which leads to the decrease of saturation magnetization M_s . The presence of Na(ac.ac) as a piezoelectric material has an important role in increasing polarization and dielectric constant, while the presence of ferrite phase prevents the reorientation of dipole moment in the same direction and freezes the rotation of dipole moment in the easy direction. The increase of Na(ac.ac) content increases the activation energy leading to increasing electrical resistivity of the composites. ME coefficient has a considerable value compared with present composite but decreased as Na(ac.ac) increased. These composites can be used as sensor in detecting electromagnetic waves.

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