SYNTHESIS AND CHARACTERIZATION OF COBALT BASED FERRITE

ALI A. ATI

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To my parents, with love and gratitude.

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ABSTRACT

Magnetoelectric (ME) materials have the ability to convert magnetic energy into electrical energy and/or vice versa. This work involves the study of structural, electrical and magnetic properties of $(f)Ni_{(1-x)}(Co/Mn)_xFe_2O_4/Pb(Mg_{0.33}Nb_{0.67})_{0.67}Ti_{0.33}O_3$ nanocomposites, which have been successfully synthesized by chemical co-precipitation method. The presence of both phases in the composites were confirmed by using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and vibration sample magnetometer (VSM). The variations of dielectric constant and loss tangent as a function of frequency as well as temperature were studied using two-point probe impedance analyzer. Temperature dependent dielectric constant shows diffused phase transition in magnetoelectric nanocomposites. The DC electrical resistivity measurements were carried out within the temperature range of 300 – 923 K. Variation of magnetoelectric voltage coefficient traces the path of magnetostriction as a function of magnetic field. All composites show peak behavior in magnetic field dependent on magnetoelectric voltage coefficient. The magnetoelectric (ME) powder nanocomposite system of $(f)\ Ni_{(1-x)}(Co/Mn)_xFe_2O_4+(1-f)\ Pb(Mg_{0.33}Nb_{0.67})_{0.67}Ti_{0.33}O_3$ (with $x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0$) and $f = 0.15$, has been successfully studied. The magnetoelectric coefficient for all the composites were measured using static magnetoelectric set up. All magnetic field dependent of magnetoelectric measurements show peak behaviour, which can be explained on the basis of magnetic field dependent variation of magnetostriction and piezomagnetic coefficient behavior. The strong compositional dependent of magnetoelectric voltage coefficient is a common feature for ferrite base nanocomposites. In this study the magnitude of the magnetoelectric coefficient is found to be higher with increasing amount of ferrite phase in nanocomposites samples. The magnetoelectric studies show that high resistive magnetic phase with high piezomagnetic coefficient in low magnetic field region is helpful to enhance the magnetoelectric coupling. The present data suggest that the magnetoelectric interaction depends on the magnetostriction behaviour, piezomagnetic coefficient, resistivity, content of constituent phases and connectivity between the phases.

ABSTRAK

Bahan magneto-elektrik (ME) mempunyai keupayaan untuk menukar tenaga magnetik kepada voltan elekktrik dan/atau sebaliknya. Kajian ini melibatkan sifat-sifat struktur, elektrik dan magnetik bagi nanokomposit $(f)\mathrm{Ni}_{(1-x)}(\mathrm{Co/Mn})_x\mathrm{Fe}_2\mathrm{O}_4/\mathrm{Pb}(\mathrm{Mg}_{0.33}\mathrm{Nb}_{0.67})_{0.67}\mathrm{Ti}_{0.33}\mathrm{O}_3$ yang berjaya disintesis menggunakan kaedah pemendakan kimia. Kehadiran semua fasa di dalam komposit telah dikenal pasti menggunakan kaedah pembelauan sinar-X (XRD), mikroskop electron imbasan pancaran medan (FESEM) dan magnetometer getaran sampel (VSM). Variasi pemalar dielektrik dan tangen kehilangan (tanδ) sebagai fungsi frekuensi serta fungsi suhu telah dikaji menggunakan penganalisis impedans dengan penduga dua titik. Kelakuan pemalar dielektrik bersandar suhu menunjukkan pembauran fasa di dalam nanokomposit magneto-elektrik. Pengukuran kerintangan elektrik DC telah dijalankan dalam julat suhu 300 – 923 K. Variasi pekali voltan magneto-elektrik telah menunjukkan magnetostriksi sebagai fungsi medan magnet. Semua komposit menunjukkan ciri-ciri puncak di dalam medan magnetik adalah bersandar kepada pekali voltan magneto-elektrik. Serbuk nanokomposit magneto-elektrik untuk sistem $(f)\mathrm{Ni}_{(1-x)}(\mathrm{Co/Mn})_x\mathrm{Fe}_2\mathrm{O}_4 + (1-f)\ \mathrm{Pb}(\mathrm{Mg}_{0.33}\mathrm{Nb}_{0.67})_{0.67}\mathrm{Ti}_{0.33}\mathrm{O}_3$ (dengan $x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0$) dan $f = 0.15$, telah berjaya disediakan. Pekali magneto-elektrik untuk semua komposit telah diukur menggunakan aturan magneto-elektrik pegun. Semua pengukuran bagi medan magnet bersandar magneto-elektrik menunjukkan ciri-ciri puncak, yang dijelas berasaskan medan magnet bersandar terhadap kelakuan magnetostriksi dan pekali piezomagnet. Kebergantungan kuat pekali voltan magneto-elektrik terhadap komposisi adalah cirri lazim untuk nanokomposit berasas ferit. Dalam kajian ini magnitud pekali magneto-elektrik didapati meningkat dengan peningkatan amaun fasa ferit di dalam sampel nanokomposit. Kajian magneto-elektrik menunjukkan bahawa fasa magnet kerintangan tinggi dengan pekali magnetik piezo yang tinggi di dalam medan magnet rendah dapat membantu dalam meningkatkan gandingan magneto-elektrik. Data semasa menunjukkan interaksi magneto-elektrik bergantung kepada kelakuan magnetostriksi, pekali piezomagnet, kerintangan, kandungan juzuk fasa dan hubungan antara fasa.

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LIST OF ABBREVIATIONS

DC Resistivity	-	Electric resistivity measurements
EDX	-	Energy dispersive X-ray
E-P	-	Electric poling
FESEM	-	Field emission scanning electron microscopy
FT-IT	-	Fourier transform Infrared
FWHM	-	Full with at half maximum
LCR	-	Inductance capacitance resistance
ME	-	Magnetoelectric Measurements
P-E	-	Polarization electric felid measurement
SEM	-	Scanning electron microscope
VSM	-	Vibrating sample magnetometer
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

(dE/dH) - Magnetoelectric voltage coefficient

$(dE/dH)_H$ - Magnetoelectric voltage coefficient

ΔE - Activation energy

μ_0 - Permeability

A - Area

a - Area

Å - Angstrom

a, c - Lattice parameter

Ba - Barium

Bi -

BSTO - Barium strontium titanate

C - Capacity

C_0 - Capacitance of condenser

Ca - Calcium

CBH - Correlated barrier hopping

Co - Cobalt

C_o - Stander capacitor

Cp - Capacity

Cr_2O_3 - Chromium(III) oxide

Cu K_α - Copper K-alpha line

$CuFe_2O_4$ - Copper ferrite

d - Interplaner distance

D - Vertical distance

d - Thicknesses of grain boundary

d_{33} - Piezoelectric coefficient

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CHAPTER 1

INTRODUCTION

1.1 Overview

The identification of novel materials with better properties or new dispensation techniques to improve the performance of existing materials, along with the inexpensive advantages, is always a substance of attention to researchers. The desire to produce novel smart materials is strongly dependent on the availability of suitable materials with enhanced properties [1, 2]. Each ceramic material has its own properties, which makes it useful for human beings. There are certain classes of materials such as, magnetic and ferroelectric materials, which infuse many aspects of modern science and technology. In this case, these materials are prepared today by various techniques in different form. Therefore, it is essential to know the origin and mechanism of magnetic and electrical for every combination and form of the magnetic and electrical materials, which is used for specific applications [3-5]. As mentioned before, each material has its unique property, which increases its importance in useful applications of these materials.

It is known that, a piezoelectric material has ability to convert mechanical energy into electrical energy and vice versa. Similarly, magnetostrictive materials can convert magnetic energy into mechanical energy [6, 7]. New generation of devices need such type of smart materials, which can convert magnetic energy into electrical energy or vice versa. Such a conversion is possible in a new category of materials called magnetoelectric (ME) materials [8, 9]. Very few naturally occur single phase materials like Cr_2O_3, show magnetoelectric phenomena, whereas a combination of ferroelectric-ferrite materials generate ME effect extrinsically. Such a combination of individual phases having its own characteristic properties shows a new material property, which is absent in their parent phases. The demerit of the single phase magnetoelectric materials is that, it's magnetoelectric effect at room temperature is very weak and not usable in practical applications [10]. The main advantage of composite material is that, one can improve the magnetoelectric phenomena by a combination of the best characteristics of ferrite-ferroelectric materials.

Recently, many research groups paid attention for the improvement of magnetoelectricity in different composites, to fulfill the necessary requirements for device applications. As for the trend towards advanced technology, a good addition of multi-functions into a single material organization then becomes very attractive. It is expected that new generation of devices using composite materials that combine magnetic, ferroelectric and magnetoelectric (ME) properties in an effective and intrinsic manner have broad potential applications. The co-existence of magnetism and electricity will produce new physical phenomena (magnetoelectric effect), which offer possibilities for new device functions [11-13].

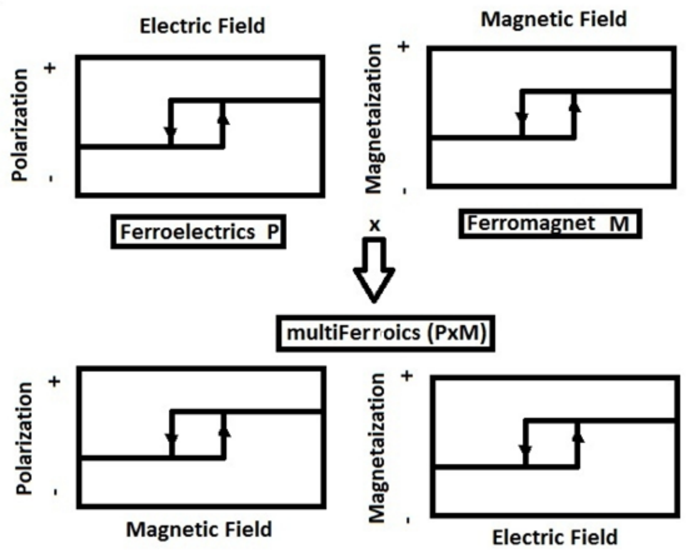

Figure 1.1 A sketch representation of ferroelectricity and ferromagnetism integration as well as the mutual control between them in multi-ferroics (ferrite and ferroelectric materials)

Multi-ferroic (ferrite and ferroelectric materials) offers excellent ferroelectric polarization (electric field hysteresis) and magnetization (magnetic field hysteresis) [13-15]. It is represented in Figure 1.1 where all magnetoelectric (ME) materials are multi-ferroics in nature, and hence the coupling interaction between the two order parameters becomes prime important. The coupling of the ferroelectricity and magnetism (either ferromagnetic or ferrimagnetic) in magneto-electricity indicates an option that influences the magnetic properties over electric fields in vice versa manner. Thus, the material is suited for many state memory parts or unique memory requests.

In the case of magnetoelectric (ME) nanocomposites, despite of many materials mixtures and structures surveyed, poor performances on the magnetoelectric (ME) yields were due to the reduced dielectric, electrical and ferroelectric features. In addition, there are poor reproducibility and large scattering of functional properties data [16, 17]. Comprehensive investigations considering the different aspects such as, the influence of nanocomposition, preparation routes, nanostructural properties such as interface doping and degree of connectivity, magnetic/ferroelectric properties of the parent phases are still lacking.

1.2 Magnetoelectric (ME) Effect in Nanocomposites

Neither ferroelectric nor magnetic phase has the magnetoelectric (ME) influence except in the composites of these two phases, which can be used to create magnetoelectric (ME) behavior from materials, which do not show the magnetoelectric (ME) outcome. This is conveniently achieved by using a mixture of magnetic and ferroelectric composites.

Consider a particulate of ME nanocomposites as shown in Figure 1.2, where the magnetostrictive particles are distributed in the ferroelectric grains.

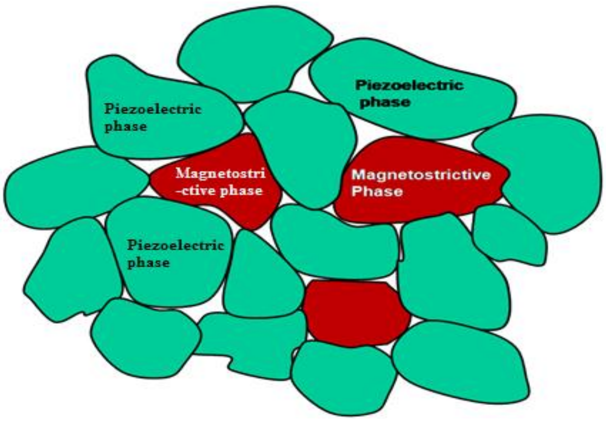

Figure 1.2 Schematic representation of particulate ME nanocomposites

The magneto-electric influence on nanocomposites material term a product property [18, 19], was from the results of cross interaction on different orderings of two separate composite phases. This two-step process was observed from the ME effect in nanocomposites, which is explained using block diagram in Figure 1.3.

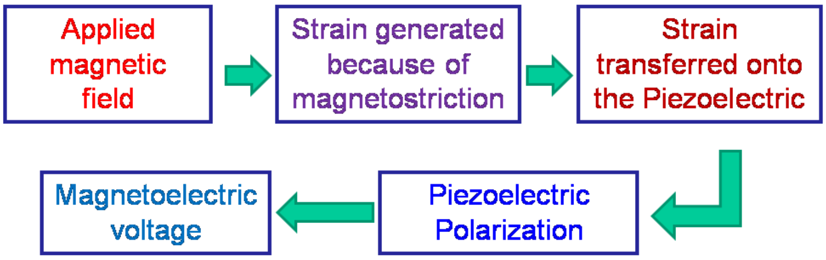

Figure 1.3 Block diagram indicating sequential steps of magnetoelectric effect in nanocomposites

When a magnetic field is applied to ferrite-ferroelectric composites, the magnetic phase changes its shape magnetostrictively i.e. ferrite grains are strained. The strain is then transferred to the ferroelectric phase, which exerts stress on it, resulting in an electric polarization due to piezoelectric effect. Thus, the magneto-electric effect in composites is extrinsic (i.e. in product, unlike in single phase, where it is intrinsic), depending on the composite nanostructure and coupling interaction across ferromagnetic-ferroelectric interfaces. The magneto-electric is a response from an electric polarization (P) upon magnetic field (H) application (i.e. the ME direct effect is denoted as ME_H effect: $P=\alpha\ H$) and/or the appearance of a magnetization M upon applying an electric field E (i.e., the converse ME effect, or ME_E: $M=\alpha E$). The product properties of the composites can be mathematically represented as follows:

$$ME_H\ effect = \left(\frac{magnetic}{mechanical}\right)_{magnetostriction} \times \left(\frac{mechanical}{electrical}\right)_{piezoelectric} \tag{1.1}$$

$$ME_E\ effect = \left(\frac{electrical}{mechanical}\right)_{piezoelectric} \times \left(\frac{mechanical}{magnetic}\right)_{piezomagnetic} \tag{1.2}$$

The ME effect is a product of the magnetostrictive effect in the magnetic field i.e. magnetic/mechanical effect and the piezoelectric effect in the ferroelectric type i.e. electrical/mechanical effect. Thus, the ME outcome is a product property referring to unique effects which initiate the interaction within ME nanocomposites. The magnetostrictive effect on the magnetic phase and piezoelectric effects in the ferroelectric phase are included [20]. This trend is related to the concentration of individual phases present in the ME nanocomposites. The conceptual points to enhance the ME effect in composites are:

i. Two different phases should be in equilibrium.
ii. Mismatching between grains ought to be minimum.
iii. The magnitude of the magnetostriction coefficient of magnetic phase ought to be as high as possible.
iv. The magnitude of the piezoelectric coefficient of the ferroelectric phase should be high.
v. The accumulated charges must not leak through the magnetic phase, i.e. resistivity of both phases must be comparable and sufficiently large.
vi. The ferroelectric/magnetic transition temperatures need to be higher than the room temperature, near achieving electric and magnetic ordering respectively at room temperature.
vii. Proper electrical poling of the magnetoelectric (ME) nanocomposites is required in order to improve piezoelectricity in ferroelectric phase.

1.3 Statement of Problem

The nanocomposites materials of ferrite and ferroelectric phase show interesting properties that are superior to conventional ferrite and ferroelectric materials. The magnetoelectric (ME) effect observed in such composites is absent in its constituent phases and magnetoelectric (ME) output is small as compared to single phase material.

It has been seen from previous discussion, selection of a suitable ferrite and ferroelectric materials with high piezomagnetic coefficient and piezoelectric coefficient can enhance the magnetoelectric effect in nanocomposites. Where μ is the permeability of magnetic phase and $\acute{\varepsilon}$ is the dielectric permittivity of ferroelectric phase. Thus, the primary criterion for selection of individual phases in the nanocomposite is to identify materials having similar crystallographic symmetry and possesses large magnetic permeability and dielectric permittivity.

In nanocomposites the individual phases are mixed, milled, shaped and sintered in order to obtain very dense samples. The reaction between the individual phases limits the high value of the sintering temperature for nanocomposites. The problem of high leakage current for such systems arises due to the magnetic phases. It has been found that, both the perovskite $A^{2+}B^{4+}O_3$ and spinel $M^{2+}Fe_2O_4$ are appropriate to enhance the magnetoelectric effect in nanocomposite form. Hence it is necessary to select suitable nanocomposite system which will fulfill all the necessary requirements.

Terfenol-D, an alloy of terbium, dysprosium, and iron, is known to exhibit highest magnetostriction. However, Terfenol has many limitations such as its poor mechanical properties, a single crystal is required for many applications, the high costs of Tb and Dy, and the presence of eddy currents when high frequencies are involved. In order to overcome these problems, current research has been focused to obtain an oxide based magnetostrictive material that will exhibit higher magnetostrictive strains at lower magnetic field strengths. The advantages of an oxide based magnetic material are that it would be much cheaper than the commercial alloys and can prevent the generation of eddy currents. Naturally ferrites have very large resistivity (MΩ cm) than the *R*-Fe compounds (μΩ cm) and thus compatible with the ferroelectric materials (GΩ cm) in high frequency applications. They are also known to have saturation in their magnetostriction at low bias magnetic fields due to small magnetic anisotropy. It is known that, $CoFe_2O_4$ exhibits highest magnetostriction among all the known ferrites. Cobalt ferrite in single crystalline form exhibits high anisotropic magnetostrictive strain depending on the composition. Similarly, Nickel ferrite has attracted considerable attention because of its large permeability at high frequency, remarkable high electrical resistivity, mechanical hardness, chemical stability and cost effectiveness. The appropriate choice of substituents in nickel ferrite has made it possible to tailor the materials properties for a variety of diverse requirements of electronic and magnetic devices. [Ni–Co(Mn)] mixed ferrites are highly resistive and magnetostrictive. Thus in thisstudy, nanocomposition for ferrimagnetic phase was varied to change the magnetic properties such as magnetization, permeability, and resistivity. $Ni_{(1-x)}(Co/Mn)_xFe_2O_4$ ferrite provides high resistivity and magnetostriction coefficient which favors the magnetoelectric effect, and is suitable as one of the phases used for the magnetoelectric biphasic composite. Hence $Ni_{(1-x)}(Co/Mn)_xFe_2O_4$ with $x = 0.0$, 0.2, 0.4, 0.6, 0.8, 1.0 is suitable as piezomagnetic phase used for the magnetoelectric biphasic composite. Large piezoelectric responses have been observed in these perovskites near structural phase boundary, the so-called morphotropic phase boundary (MPB). The Pb based ferroelectrics such as Lead-Magnesium-Niobium–Lead Titanium (PMN-PT) is selected to be a good choice as ferroelectric phase. Hence in this case PMN-PT is selected as the ferroelectric phase. The selection of PMN-PT as a ferroelectric phase in magnetoelectric nanocomposites shows enhancement in magnetoelectric voltage coefficient compared to other magnetoelectric nanocomposites. Hence PMN-PT in morphotropic phase boundary (MPB) region is selected as a ferroelectric phase due to its strongest piezoelectric property among various piezoelectric materials.

It is expected that, the selected individual phases will fulfill all the necessary requirements. Microscopic studies of composites also shows leakage of relevance structure of composites, which may be due to mismatching between grains of ferrite and ferroelectric phase in the composites materials. Since physical properties of multiphase nanocomposites depend critically on nanostructure, it is desirable to know the distribution of the constituent phases. A more precise way of morphological analysis required for understanding of nanostructural dependent properties of magnetoelectric nanocomposites.

1.4 Research Objectives

The objectives of this study are to:

1. synthesize magneto-electric composite of [$Co_{(1-x)}Mn_{(x)}Fe_2O_4$, $Ni_{(1-x)}Co_{(x)}Fe_2O_4$] nanoparticles ferrite and Lead-Magnesium-Niobium–Lead Titanium (PMN-PT) ferroelectric materials by using chemical co-precipitation technique.
2. determine the structure of the ferrite, ferroelectric and nanocomposites studies using X-ray diffraction technique.
3. determine lattice strain of nanocomposites using X-ray diffraction technique data.
4. determine morphology of magneto-electrical nanocomposites by scanning electron microscopy (SEM).
5. determine the electrical properties of $Co_{(1-x)}Mn_{(x)}Fe_2O_4$, $Ni_{(1-x)}Co_{(x)}Fe_2O_4$ and PMN-PT.
6. determine the magnetoelectric characteristics[$(dE/dH)_H$] as a function of magnetic field at room temperature for nanocomposites samples [(f) $Ni_{(1-x)}Co_{(x)}Fe_2O_4$ + (1-f) PMN-PT] and [(f) $Co_{(1-x)}Mn_{(x)}Fe_2O_4$+ (1-f)PMN-PT].

1.5 Scope of Research

In this work, ferrites nanoparticles phase of $Ni_{(1-x)}Co_{(x)}Fe_2O_4$,$Co_{(1-x)}Mn_{(x)}Fe_2O_4$ and ferroelectric nanoparticles phase of PMN-PT were synthesized using chemical method. The two phases were mixed together to obtain the nanocomposites. Morphology and structure of nanocomposites were studied by field emission scanning electron microscopy (FE-SEM) and X-ray diffraction. The dielectric properties were determined using impedance analyzer at room temperature and well above room temperature in frequency range of 100 Hz to 5 MHz.

Temperature dependence of electrical resistivity (ρ_{dc}) and the effect of ferrite phase addition on the conductivity were also determined. Magnetic and ferroelectric hysteresis loop were determined using vibrating sample magnetometer (VSM) and polarization versus electric field (P-E) loop tracer system. Chemically synthesized samples will be used to prepare nanocomposites and subjected at a different sintering temperature of 600 °C, 1100 °C, 1200 °C, 1250 °C in order to study the effect of sintering on various properties of ME nanocomposites.

1.6 Significance of Study

The nanocomposite materials (magneticoelectric nanocomposition) were synthesized in particulate form using hydroxide co-precipitation method. All of these samples are carefully processed further in order to obtain a high purity product. Since the physical properties of multiphase (nanocomposites) strongly dependent on the structural, it is desirable to know the distribution of the constituent phases. A more precise way of morphological analysis is required for understanding of nanostructural dependent properties of ME nanocomposites. Since there are tremendous technical demands for large dielectric constant materials, it is significant to study systematically the dielectric properties of the magnetoelectric nanocomposites, which certainly will cast light on the origin of the high dielectric constant in multiphase materials. The detailed analysis of temperature dependent dielectric constant behaviour, especially in the transition temperature region, is useful to understand the diffuse phase transition behaviour. In the present work, we have highlighted the nature of the dielectric peak in the vicinity of phase transition temperature region. For this a modified Curie-Weiss law used which seems to be more suitable than the standard Curie-Weiss law. The Curie–Weiss law describes the magnetic susceptibility χ of a ferromagnet in the paramagnetic region above the Curie point: [$\chi = C / T\text{-}T_C$], where C is a material-specific Curie constant, T is absolute temperature, measured in kelvins, and T_c is the Curie temperature, measured in kelvin. The law predicts a singularity in the susceptibility at $T=T_c$. Below this temperature the ferromagnet has a spontaneous magnetization. It is also necessary to analyze the temperature dependent dielectric constant behaviour of magnetoelectric composites for different contents of individual phases at room temperature and at temperatures well above the transition temperature of both phases. The electrical and magnetic properties of this material as a unique substance in the electrical, electronics and magnetic applications are important in microwave communication, data processing devices, electrical device, circulators and magnetic recording. Additionally, tremendous technical demand for large dielectric material constant is important.

A broad literature reviews on magnetoelectric nanocomposites show wide variation in magnetoelectric (ME) voltage coefficient in bulk nanocomposites. It can be observed that the magnetoelectric (ME) voltage coefficient is affected by the number of factors such as the method of synthesis, grain size of individual phases, nanostructure of the samples, selection of individual phases, porosity, resistivity, dielectric and magnetoelectric properties of the nanocomposites.

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