Girls College for Arts
Science and Education
Ain Shams University

Optimization of the physical properties of Lithium multiferroic to be used for high frequency applications

Thesis Submitted for

M.Sc. Degree in Science (Materials Science)

By

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(2013)

Girls College for Arts
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Dedication

To my great God

Then

To Every One Who Helped Me:

To

My Supervisors……,

My Friends ……,

And

My Family……..

كلية البنات للأداب والعلوم والتربيه
قسم الطبيعة

تحسين الخواص الفيزيائية لليثيوم ملتيفرويك لكى تستخدم فى تطبيقات الترددات العاليه

رسالة ماجيستير

مقدمه من

منة الله محمد مسعد أبوحصوة

بكالوريوس العلوم قسم الفيزياء والحاسب الآلى(2009)

للحصول على درجة الماجيستير فى فلسفة العلوم تخصص فيزياء الجوامد (علوم المواد)

اشراف

ا.د./ محمد علي احمدا.د./ نجوي عكاشة مراد

أستاذ النانوتكنولجي وعلوم المواد – قسم الفيزيا ء أستاذ فيزياء الجوامد علوم المواد

كلية العلوم-جامعة القاهرة *كلية البنات جامعة عين شمس*

Acknowledgement

I would like to thank **Prof.Dr. D.Sc. Mohamed Ali Ahmed** to his helpfulness and his permanent courage in succeeding this work as he is a great scientist and I have learned much information in both physics and nanotechnology fieldsin his lab and under his supervision.

Every day I come to material science lab(1) I learned newimportantthings as everyone in the lab has learned the cooperation from the great scientist **Dr. Mohamed.**

He has the challenge to any obstacle against our work and I learned this thing from him, he also has a strong personality and thewisdom.

I would like to express my respect and deep gratitudeto**Prof.Dr.NagwaOkasha Mourad**in recognition of her great efforts in succeeding this thesis such as her permanent advices and she always try to make everything as easy as possible to me, she helped me morally and I have learned great things in the physics and in the practical life as wellall the period of research, she has high and strong personality.

Special thanks to my father, my mother and my brothers Mosaad and Mohamed, to my sisters Manar and Mona for their great support during this work.

Chapter one
Introduction

1-A: Literature Survey

H. He et al. (2012) [1] studied the internal parameter and state of charge of battery using $LiFePO_4$ which greatly depend on Proper model and high efficiency, high accuracy algorithm which presented in the applications of electric vehicles by using an adaptive extended kalman filter (AEKF) algorithm the thevinin equivalent circuit model is selected to model the $LiFePO_4$ battery and its mathematics equations.

A.E. Abdel - Ghany et al. (2012) [2] studied the Physico-Chemistry and electrochemistry of lithium ferrite synthesized by the solid state reaction characterization included X-ray diffraction (XRD) , scanning electron microscope (SEM), Raman's scattering (RS), fourier transform infrared spectroscopy (FT- IR) and SQUID magnetometry. The results indicated that, XRD peaks gradually sharpen with increasing the temperature and all the diffraction peaks can be indexed to the cubic $\alpha LiFeO_2$ (Fm3m space group) with the unit cell parameter a=4.155 A˚. While, the electrochemical properties of $Li/LiFeO_2$, crystallochemistry of Lithium iron oxide show that this compound crystallizes in cubic structure with well formed crystallites size of 0.55µm a combination of FTIR and RS measurement. The cubic structure of $LiFeO_2$ magnetic measurements have evidence that $\alpha LiFeO_2$ exhibit deviation from the Curie-Weiss law with $\mu_{eff} << 5.9\mu_B$ at room temperature where the cationic disorder affects the magnetic properties, this compound is expected to belong to the class of Fe- diluted magnetic semiconductor, electrochemical texts show severe structural changes occurred during the first charge-discharge process of the cell structural transformation from $\alpha LiFeO_2$ to $LiFe_5O_8$ spinel phase has been evidenced from X-ray diffraction and Raman spectroscopy.

B. Ramesh et al. (2012) [3] studied the AC impedance on $LiFe_{5-x}Mn_xO_8$ ferrite where (x = 0.0, 0.2, 0.4, and 0.6) prepared using Sol-gel method. X-ray analysis confirms that, all the samples were prepared in a single phase with no detectable impurity and crystallize in spinel structure. Complex impedance data measured in the

frequency range 200Hz to 1MHz at different temperatures reveals that, at low temperature low values of conductivity and high impedance value and the results shows that, multiple relaxations process where the relaxation time of the spins is found to decrease with increasing the temperature in the material.

S.E. Shirsath et al. (2011) [4] studied the sintering temperature and particle size dependent structural and magnetic properties of lithium ferrite ($Li_{0.5}Fe_{2.5}O_4$) were synthesized by the sol-gel auto ignition technique and sintered at four different temperatures ranging from 875 K to1475 K in step of 200K. The sample sintered at 875K was also treated for four different sintering times ranging from 4 to 16 hours. The results reveals that, samples sintered at 1475K have cubic spinel structure with small amount of α-Fe_2O_3 (hematite) and γ- Fe_2O_3(maghemite), the samples sintered at ≤ 1275K don't show the hematite and maghemite phases and forms the single phase spinel structure with the cation ordering on the octahedral sites. The particle size of Lithium ferrite is in the range of 13-45nm depending on the sintering temperature and the sintering time. Moreover, the saturation magnetization increased from 45 to76 emu/gm and coercivity decreases from 151 to 139 Oe with an increase in particle size. Besides, blocking temperature effects observed below 244K the dielectric constant increases with an increase in sintering temperature and the particle size were measured by both XRD and TEM are in very good agreement with each other.

S.K. Rakshit et al. (2011) [5] studies the thermodynamics properties of Li-Fe-O system using differential scanning calorimetry and solid state electrochemical technique based on fluoride electrolyte. The heat capacities of $LiFe_5O_8$ (s) and $LiFeO_2$(s) were determined in the temperature range 127-861K using differential scanning calorimetry Gibbs energies of formation $LiFe_5O_8$ (s) and $LiFeO_2$(s) were determined using Knudsen diffusion mass spectrometry. Thermodynamic analysis shows that $LiFe_5O_8$(s) is more stable than $LiFeO_2$(s). These data are valuable for predicting thermodynamic stabilities of these compounds in different Physico-Chemical conditions.

D. Bochenek et al. (2011) [6] investigated the multiferroic ceramics $Pb(Fe_{1/2}Nb_{1/2})O_3$ (PFN) and PFN doped by various amount of Lithium (PFN:Li) in amount of 0.5%, 1% ,1.5%, and 2.0%. X-ray diffraction patterns, microstructure, DC

electric conductivity, temperature dependencies on dielectric losses, electromechanical properties, and hysteresis loops have been investigated. The results shows that, the influences of lithium on the grain size, shape, and the physical properties indicated that, the best microstructure is for 2.0%, and XRD Pattern shows that the samples are single phase Perovskite structure. Lithium addition decrease the diffusion of phase transition and increases the maximum value of dielectric permittivity shifts towards lower temperature, besides, influence the shape of polarization-Electric field (PE) and Strain-Electric field (SE) hysteresis loops will become more typical for ferroelectric materials by increasing Li amount.

M.R. Joung et al. (2011) [7] synthesized $BaTiO_3$ (BT) using the alkoxide-hydroxide route Formation of (BT) commenced at 60°C and their amount increased by increasing temperature. The results shows that, the presence of TiO_2 developed at temperature greater than 100°C due to the insufficient amount of H_2O caused by its evaporation to avoid the TiO_2 phase. By adding water, it's difficult to control the shape and size of BT nanopowder formed by water addition so; two step processes is presented to obtain homogeneous, highly tetragonal (BT) nanopowder without any second phase of TiO_2.

S.Z. Liu et al. (2011) [8] synthesized $BaTiO_3$ by using $Ba(NO_3)_2$ and a high reactive activity of TiO_2 nanocrystals at 600°C. The results reveal that, X-ray diffraction and X-ray photoelectron spectroscopy shows that, the as prepared products were cubic phase $BaTiO_3$ with a surface composition of $Ba_{0.97}TiO_{2.92}$ and TEM image shows that the size of $BaTiO_3$ is of range 38-158 nm.

M. Cerne et al. (2011) [9] synthesized $BaTi_{0.87}Sn_{0.13}O_3$ (BTS_{13}) by low temperature aqueous synthesis (LTAS) method. The evolution of the structure and microstructure of the samples heated at temperatures up to 1000°C was studied by TGA, FT-IR, SEM and XRD techniques. The microstructure analysis confirmed nanosized grains of about 40nm with a great tendency to agglomeration $BaTi_{0.87}Sn_{0.13}O_3$, while $Ba(Ti,Sn)TiO_3$solid solution is obtained as a single phase with perovskite structure of cubic $BaTiO_3$ single phase at 800°C. Moreover, the ceramics prepared

from as obtained (BTS_{13}) powders (60-70nm) shows good dielectric and ferroelectric characteristics, the dielectric was about 4800, and the dielectric loss was 0.229 at 1KHz and the Curie temperature of about (31°C), the remanent polarization P_r were $13\mu C/Cm^2$ and coercive field(E_c) = 0.89KV/cm. The ferroelectric parameters P_r , E_c decrease with increasing frequency in the domain 100Hz to 10KHz, the dielectric and ferroelectric properties of BTS_{13} prove that, the (LTAS) method is able to prepare nanopowders and ceramics with application in electronic field.

C.Shi et al. (2011) [10] studies the bulk ceramics with general formula (1-Y) $BiFe_{1-x}$ Sc_xO_3-Y $BaTiO_3$ (x=0.1- 0.3, y = 0.1- 0.3 mol %) were prepared by the traditional solid state method. X-ray diffraction analysis indicated that, all the samples crystallized in pure perovskite structure, the structure phase transition from R3c to Pm-3m occurred when the amount of $BaTiO_3$ exceed 20 mol%. The room temperature M-H curves shows that, the replacement of Fe by proper amount of scandium could decrease simultaneously the coercive field so the soft magnetic property could be improved and the spontaneous magnetization, the addition of appropriate amount of scandium could reduce the dielectric loss, The results show the formation of solid solutions with BTO increase the dielectric constant of BFO.

M. S. Al-Assiri et al. (2011) [11] transform glasses in the system $BaTiO_3$-V_2O_3-Bi_2O_3 to glass ceramic nanocomposites by annealing at crystallization temperature determined from DSC thermogram. The data shows that after annealing, consist of small crystallites embedded in glassy matrix and the crystallization temperature increases by increasing $BaTiO_3$ content where the average size of the crystallites was 25nm. However, the resultant material has much higher conductivity than the initial glasses due to the interfacial regions between the amorphous phase and the crystalline phase in which the concentration of the V^{4+} - V^{5+} pairs responsible for electron hopping has higher values than inside the glassy matrix, small polaron hopping parameters were obtained. Besides, the conduction was attributed to the non adiabatic hopping of small polaron.

A.Aydi et al. (2011) [12] elaborated new compound of the following composition Ba_{1-x} $Na_x(Ti_{1-y}$ $Sn_y)$ Nb_x O_3 (BTSnNxy) by using solid state technique. The study presents some continuous solid solutions between the next three phases

$NaNbO_3$ antiferroelectric phase that become easily ferroelectric at low rate substitutions, $BaTiO_3$ ferroelectric phase, and the paraelectric stannate phase ($BaSnO_3$). Two different dielectric behaviors can be observed once some substitutions are made either in A or B sites of ABO_3 perovskite these substitutions modify the dielectric properties of the material. The introduction of Sn^{4+} and Ti^{4+} cations in the B site favors respectively a decrease in the transition temperature and an increase in the value of the real dielectric permittivity. The transition temperature should be modulated by varying the rate of cationic substitutions. Some relaxer materials can be obtained at a temperature around room temperature due to some modification of the chemical bonding that results in the substitution of Ti for Sn, the screen effect due to internal d electrons of Sn atoms decreases polarization coupling in ferroelectric lattice comparing with the pure (BTSnNxy) compound. (BTSnNxy) ceramics exhibits an increase in $\acute{\varepsilon}_{r\,max}$ and decrease in T_m or T_c by addition of Ti^{4+} and Sn^{4+} respectively.

Y.Bai et al. (2011) [13] demonstrated the electrocaloric effect (ECE) of $Na_{0.5}Bi_{0.5}TiO_3$-$BaTiO_3$ (NBT-BT) lead free ferroelectric ceramics was fabricated by solid state reaction method. The results reveal that, the polarization of (NBT) increases monotonically within the temperature range of 25-145°C during the application of the electric field as well as the entropy increases and the material absorbs heat, while during withdrawal of electric field, the entropy decreases and the material releases heat that for 0.92(NBT)-0,08(BT). The high field polarization increases and low field polarization decreases with rise of temperature where the temperature and/or entropy change has opposite sign under high and low applied field. Besides, the abnormal (EC) effect on (NBT-BT) is due to the relaxer character between ferroelectric and antiferroelctric phases.

A.Baji et al. (2011) [14] study two step electrospinning process to fabricate $BaTiO_3$/Polyvinylidene fluoride (PVDF) composite fibers. Microstructure examination showed that, $BaTiO_3$ fibers were well dispersed within the (PVDF) fiber matrix and aligned along the fiber axis. $BaTiO_3$ fibers syntheses were cubic tetragonal structure. XRD displayed all the peaks corresponding to the individual (PVDF) and $BaTiO_3$ phases. The piezoresponse force microscopy (PFM) of the composite fibers showed characteristic polarization-voltage and amplitude-voltage hysteresis loops confirming their piezoelectric hysteresis and ferroelectric switching behaviors.

P.Ctibor et al. (2011) [15] confirm the phase composition, optical properties, and photocatalytic activity of $BaTiO_3$ coating prepared by atmospheric plasma spraying. The spraying was carried out by a direct current gas–stabilized plasma gun. $BaTiO_3$ prepared by a reactive sintering of micrometer-sized powder of $BaCo_3$ and TiO_2. XRD and SAD mode of HR-TEM indicated certain content of amorphous fraction in the coating due to the excitation energy of the secondary absorption edge. In the sintered sample case, a secondary absorption edge associated with structural disorder of Ba and Ti is responsible for photocatalytic behavior while, in the case of both coating types, the hydrogen induced donor levels in the band gap are the dominant feature. In the case of long spray distance (SD) coating the structural disorder associated with high amorphous content decreases the photocatalytic ability. In the case of short (SD) coating, Ba loss from the structure during the sprayed process seems to be high and affect the photocatalytic efficiency strongly.

P.Firek et al. (2011) [16] shows investigation of La_2O_3 containing $BaTiO_3$ thin films deposited on Si substrates by radio frequency plasma sputtering(RF-PS) of sintered $BaTiO_3$+ La_2O_3(2wt%) target (Al) electrodes which evaporated on top of the deposited layers. dielectric constant value of the obtained layers was reached 20
where the results shows that, the thin films can be considered as a dielectric for number of microelectronic applications, the relatively low dielectric constant comparing to BT films obtained with other deposition methods is related to their amorphous nature, a better control of the deposition process. Purity may improve the properties. This study confirms the (RF-PS) method is applicable for deposition of BT layers which have very interesting electronic properties.

A. Ianculescu et al. (2011) [17] study the La-doped $BaTiO_3$ ceramics with different stoichiometric with La content of (x = 0.001; 0.0025; 0.005; 0.01and 0.025 respectively, were prepared by solid state reaction method and sintered at 1300°C for 6 hrs. The results indicated that, all the compositions are single phase perovskites with tendency of transition from tetragonal to cubic with increasing La content. The DC-non-linear behavior was analyzed using a multipolar mechanism model which able to

explain the higher tunability values in terms of other additional polarization mechanisms by considering Langevin contribution to the ε(E) dependence.

H.Gao et al. (2011) [18] studied the structure and electronic properties of the cubic barium titanate ($BaTiO_3$) using the different density functional theories(DFT), the calculation results shows that, GGA/PW91 and ultra soft pseudo potential method are good for predicting the electronic structure and properties of $BaTiO_3$,the results also indicates that, $BaTiO_3$ is indirect semiconductor, ionic bond between Ba atom and TiO_3 group and covalent bond between Ti and O atoms in the unit cell, it is also found that the Ti 3d orbitals interact strongly with the orbitals of the octahedral oxygen to split into bonding and antibonding states, electronic maps along (110), (100), and (001) proved that the covalent bond exist between Ti and O atoms in $BaTiO_3$.

Y.Pu et al. (2011) [19] studied the effects of Kaolinite addition on the densification and dielectric properties of $BaTiO_3$ ceramics which prepared through the conventional solid-state method, Kaolinite had a strong effect on the densification, microstructure and dielectric properties of $BaTiO_3$ ceramics. The density characterization shows that, the addition of Kaolinite lowered the sintering temperature of $BaTiO_3$ ceramics to about 1200°C , $BaO\text{-}TiO_2\text{-}Al_2O_3\text{-}SiO_2$ glass phase
was formed in accordance with the SEM-EDX analysis leading to the reduction in grain size and improvement of average breakdown strength of ceramics, $BaTiO_3$ with 4.0%wt Kaolinite addition possessed well temperature stability of dielectric constant, these results provided useful information for the application of Kaolinite in electronic ceramics.

J.Q.Qi et al. (2011) [20] developed a size-modulable and large scale synthesis route to obtain perovskite structured nanoparticles of $BaTiO_3$ at room temperature. The results indicated that, the synthesis efficiency is improved greatly and batch processing scaled up easily because large quantity of solvents is not necessary for this method. The grain size are modulated by water content and the alkanol chain length of the dispersant efficiently where both the content of water and alkanol chain length of

the dispersant are dominant in the grain coarsening during the synthesis of the $BaTiO_3$ nanoparticles.

Y.R.gang et al. (2011) [21] prepared $BaTiO_3$ powders by sol-gel method and the carbonyl iron powder is prepared via thermal decomposition of iron pentacarbonyl, then $BaTiO_3$-carbonyl iron composite with different mixture ratios prepared using the as prepared material. The results shows that, a possible microwave absorbing mechanism of $BaTiO_3$-carbonyl iron composite can find applications in suppression of electromagnetic interference and reduction of radar signature.

S.Sharma et al. (2011) [22] studied the Lanthanum substituted Barium titanate (BT), $Ba_{(1-x)}La_x\ Ti_{(1-x/4)}O_3$ (where x= 0.02,0.04,0.06 and 0.08) singed as BLT_1, BLT_2, BLT_3, and BLT_4 respectively were synthesized in single perovskite phase by microwave (MW) processing technique. MW sintering was carried out at 1100°C for 1h.The date reveal that, with the increase in La^{3+} ions substitution concentration in BT ceramic samples, the structure transformed from tetragonal to cubic. Besides, capacitance stability increased for $BLT_1$and BLT_2 ceramic samples with the increase in La^{3+} ions concentration. Moreover, BLT system transformed from ferroelectric to paraelectric with higher density, lower processing time and temperature with good dielectric properties of MW sintered La^{3+} ions modified BT ceramics than conventional processing technique signifies the importance of MW processing technique.

X.Tan et al. (2011) [23] studied the nitrogen-doped $BaTiO_3$ with a tetragonal phase which synthesized in the NH_3(67%)/Ar atmosphere by the standard solid state reaction method. It can be found that, a weak ferromagnetic behavior is observed at 300K and demonstrated that nitrogen-doped $BaTiO_3$ is ferromagnetic at room temperature.

S. Brivio et al. (2011) [24] studied the epitaxial heterostructures involving ferroelectric (FE) and ferromagnetic (FM) to obtain the magnetoelectric effects. It can be found that, the epitaxial growth of the ferromagnetic material Fe with bcc structure on ferroelectric material $BaTiO_3$ (001) with perovskite structure while Fe growth on $BaTiO_3$ with 45° rotation of its cubic lattice with respect to that of the substrate in

order to reduce the lattice mismatch. Besides, no cation (Ti,Ba) and Fe interdiffusion has been detected and ferroelectric characterization indicates remnant out of plane dielectric polarization of BTO while the magnetic behavior of Fe films is high quality single crystal thin films.

L.Dong et al. (2011) [25] studied the extreme inelastic responses due to the negative stiffness phase have been observed in $Zn_{80}Al_{20}$- $BaTiO_3$ composites not only near the tetragonal-orthorhombic transformation temperature of $BaTiO_3$ but far below the Curie point of $BaTiO_3$ which can be manipulated via specific aging processes that operate by an oxygen vacancy mechanism, this idea proposed to fabricate novel composites for potential engineering applications.

Y.H Kim et al. (2011) [26] studied the using of aerosol deposition(AD) as a fabrication method of $BaTiO_3$ polytetrafluoroethylene (PTFE) composite, the $BaTiO_3$ and $BaTiO_3$-PTFE composite thick film were successfully fabricated by (AD) at room temperature. The results reveal that, the decrement in the $BaTiO_3$ particle size was reduced and distortion of the crystal structure was relieved in the $BaTiO_3$-PTFE

composite film due to the elasticity of the PTFE material. Besides, the permittivity of the composite film was relatively low compared with that of the $BaTiO_3$ films.

A.Reichmann et al. (2011) [27] studied the evolution of ferroelectric domain pattern in donor doped barium calcium titanate during heating and cooling across the curie transition temperature was imaged by orientation contrast imaging using environmental scanning electron microscope (ESEM), domains become blurred and disappear usually when approaching T_C cooling from above T_C causes a recurrence of the domains in a very similar pattern as observed before heating. Predetermine the domain structure and that random orientation of the polar axes during spontaneous polarization can be excluded, only a few grains showed a change in domain pattern during heating and cooling indicating a local change in elastic energy conditions.

S.JShih et al. (2011) [28] mixed small amount of 70%Ag/30%Pd particles with $BaTiO_3$ powder and subsequently sintered at elevated temperature in nitrogen, air and oxygen. The solubility of Ag in the BaTiO3 was determined using an electron

probe microanalysis technique and found that Both Ag and Pd act as acceptors for $BaTiO_3$ where the solution of one acceptor can reduce the solution of another acceptor. Moreover, the lower solubility of Ag in $BaTiO_3$ also reduces the diffusion distance of Ag in $BaTiO_3$.

M.H.M.Ara et al. (2011) [29] investigated the parametric the parametric scattering of a single extraordinary polarized beam of laser in $BaTiO_3$ photorefractive crystal experimentally and theoretically. The resulting pattern consists of beam fanning, isotropic ring, and anisotropic one the ring observed on the screen is not due to the diffraction of new incident beam, as some previous reports believed; because the Bragg condition is not fulfilled in this configuration, but it may be due to the scattered light from impurities inside the crystal that may be Bragg scattered from the gratings that have remained inside the crystal from previous step. The theoretical interpretation is based on the standard theory of parametric scattering in photorefractive crystals. It has been shown that this configuration corresponds to the so called parametric B-process.

W.H. Zhang et al. (2011) [30] studied Barium titanate ($BaTiO_3$) crystal samples with different distribution of oxygen vacancies were prepared through different thermal treatment processes. The results shows that, oxygen vacancies can affect the Raman spectra of $BaTiO_3$ crystal and attributed this phenomenon to the change of interaction between constituent atoms and the breaking of translation symmetry in the surface phase of $BaTiO_3$. X-ray photoelectron spectroscopy (XPS) measurements demonstrate that there are two components in Ba 3d line.

N.Singh et al. (2011) [31] studied the magnetic, structural and dielectric properties of $Ni_{0.5}$ $Li_{1.0-2x}Cu_xFe_2O_4$ (x=0.0, 0.1, 0.2, 0.3, 0.4, 0.5) were prepared by sol–gel auto-combustion method. The experimental results were showed that, X-ray diffraction data and IR bands reveal that, Cu^{2+} ions have stronger A-site preference than the Li^+ ions. With increase in concentration of Cu^{2+} ions, the lattice constant increases because of the larger size of the Cu^{2+} ion than the Li^+ ion. Besides, increasing the annealing temperature from 400 to 800^0C there exist grain growth in the samples. Dielectric constants of the annealed samples decrease with the frequency as observed for most of the ferrites while the conductivity and the dielectric constants increase with temperature increasing. Moreover, the dielectric constants, tan δ, and ac

conductivity increase with increase in grain size of the crystal .Incorporation of Cu^{2+} ions in the Ni–Li ferrites increases the dielectric constant and dielectric loss. It is observed that the saturation magnetization first increases with the addition of Cu^{2+} ions (up to x=0.2) and with further increase in x, the saturation magnetization decreases. On the other hand, the coercivity increases with the increase in the concentration of Cu^{2+} ions.

N.Singh et al. (2011) [32] studied the dielectric, conductivity, and magnetic properties of $Zn_{0.5}Li_{1-2x}Mg_xFe_2O_4$ where (x = 0.1, 0.2, 0.3, 0.4, 0.5) Prepared via sol-gel auto-combustion method. The dielectric properties of these ferrites have been studied using impedance measurements in the frequency range 10 Hz - 10 MHz and in the temperature range 310-473 K. They found that, Dielectric constant (έ) and dielectric loss (tan δ) decreases with the frequency and increases with temperature.
The presence of a peak in the dielectric loss spectra is due to the matching of electrons hopping frequency (Fe^{2+} / Fe^{3+}) with that of applied field frequency. Also, the conductivity is constant in the low frequency region and shows dispersion in the high-frequency region. The linear variation of conductivity with temperature confirms the semiconducting nature of the samples. Moreover, the dc and ac conductivity of these ferrites increases with the increase of Mg^{2+} ion composition due to the increased hopping of electrons between (Fe^{2+} / Fe^{3+}) ions. the hysteresis loop in high magnetic field shows a superparamagnetic phase for all the samples The increase in Ms with increase in Mg^{2+} ion composition is due to redistribution of cations on A and B-sites.

M.Triki et al. (2011) [33] prepared composite material composed of ferroelectric and ferromagnetic compounds having the formula (1-x)($Pr_{0.6}$ $Sr_{0.4}MnO_3$) / (x) ($BaTiO_3$); (x=0.0,0.03,0.05,0.10 and 0.30) using conventional ceramic double sintering process. The data showed that, the temperature dependence of magnetization reveals that the composite sample show paramagnetic to ferromagnetic phase transition (PM-FM), the presence of BTO has no great effect on the physical properties of PSMO compound, the Curie temperature remains unchanged (T_C about 273K), the maximum of magnetic entropy and relative cooling power values are not very affected for low content of (BTO) and decrease for x>0.1.