Low Temperature Preparation and Effect of Pr^{3+}, La^{3+}, Sm^{3+} and Gd^{3+} Substitution on Structural, Magnetic and Dielectric, Studies of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ Ferrite Nanoparticles

Rakesh Kumar Singh, Amarendra Narayan and Dolly Sinha

Abstract- ***$Ni_{0.5}Zn_{0.5}Fe_{1.97}R_{0.03}O_4$ Nanoparticles; R= Pr, Sm, La and Gd, were synthesized using Chemical based Citrate Precursor method, annealed at low temperature 450°C for 2hr. X-ray diffraction (XRD) tool was used for estimation of average particle size and phase analysis. The average particle size was found to be 25nm, 33nm, 31nm, 22nm and 13 nm respectively. Room temperature magnetic measurement was done by vibrating sample magnetometer (VSM). The magnetization values observed are 50.692 emu/g, 43.781 emu/g, 47.875 emu/g, 43.335 emu/g and 43.518 emu/g respectively. The dielectric properties for all the samples were investigated at room temperature as a function of frequency while impedance was measured as a function of temperature. $Ni_{0.5}Zn_{0.5}Fe_{1.97}Sm_{0.03}O_4$ nanoparticles show a dielectric behavior appreciably different from $Ni_{0.5}Zn_{0.5}Fe_{1.97}Gd_{0.03}O_4$, $Ni_{0.5}Zn_{0.5}Fe_{1.97}Pr_{0.07}O_4$, and $Ni_{0.5}Zn_{0.5}Fe_{1.97}La_{0.03}O_4$ nanoparticles.***

Keywords:-$Ni_{0.5}Zn_{0.5}Fe_{1.97}R_{0.03}O_4$(R=Pr,La,Gd,Sm) Nanoparticles, Citrate Method, Magnetic and Dielectric properties,

I. INTRODUCTION

Ferrites are a magnetic materials with remarkable magnetic properties that have been investigated and applied during the last about 5 decade. Their uses encompass an impressive range extending from electronics science to power handling, simple permanent magnets, magnetic recording, Medical science, Agriculture science and Biotechnology sensing applications and water purification [1-5]. These applications are based upon the very fundamental properties of ferrites: a significant saturation magnetization, a high electrical resistivity, low electrical losses, and very good chemical stability and low eddy current loss [1-6].Ferrite materials behave as semiconductor materials with low mobility (μ) of electric charge carriers and an exponential dependence of electrical conductivity and synthesis temperature. The relation between activation energy (E_a), mobility (μ), Boltzmann constant (K) and temperature (T) is $\mu = -E_a/KT$. The structural, electrical, optical and magnetic properties such as resistivity, initial permeability, magnetization, etc., can be controlled by substitution of trivalent rare earth ions (R) in the host spinel lattice [6-10]. As rare earth oxides are good electrical insulators and have high resistivity.

In this work, substitution effects on microstructural, magnetic and dielectric properties of nanoferrites $Zn_{0.5}Ni_{0.5}R_{0.03}Fe_{1.97}O_4$ with R: La, Gd, Pr and Sm studied ,which are synthesized by the chemical based citrate precursor method.

Our objective is to carry out magnetic and dielectric study on the rare earth substituted Ni-Zn ferrite, synthesized using precursor method at low annealing temperature 450°C.

II. EXPERIMENTAL

2.1. Material Preparation

$Ni_{0.5}Zn_{0.5}R_{0.03}Fe_{0.97}O_4$ with R = Pr, Sm, Gd& La (named as NiZnPr, NiZnSm, NiZnGd and NiZnLa) respectively were synthesized by chemical based the citrate precursor method. The starting materials such as Nitrates of Iron, Nickel, Zinc and Rare earth metals-R (R= Pr, Sm, Gd and La) were carefully weighed in molar proportion. Solution was prepared using double distilled deionized water. The ingredients were mixed together and stirred continuously in a magnetic stirrer until a brown slurry solution was obtained. The mixed solution was dried up at 60°C in an air oven for 33 hours until brown powder were obtained. The resulting material was annealed at 450°C for 2 hours in muffle furnace.

2.2. Crystalline size measurement

The crystallographic phase analysis of all prepared nanomaterials samples was carried out using a Rigaku Miniflex – II X-Ray diffracometer with CuKα radiation source (λ = 1.5418 Å) operating in the Bragg-Brentano geometry in a 2θ range of 10° - 80° at a scan step of 0.02° with a scan rate of 2°/min. Particle size was calculated from the width of the prominent peak position (311) line using the Scherrer's formula corrected for instrumental broadening.

2.3. Magnetic and Dielectric measurements

Magnetization curves were obtained at room temperature by using Vibrating Sample Magnetometer(7304, Lakeshore) over a field range of ± 5000G. The parallel plane surfaces of pellets were coated with the silver paste for dielectric study. The impedance study were performed using the LCR meter (N4L PSM 1735) in the frequency range of 100 Hz – 1.5 MHz and temperature range (40°C – 400°C)

Revised Version Manuscript Received on October 20, 2015.

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III. Results and Discussion

3.1. Structural-Properties

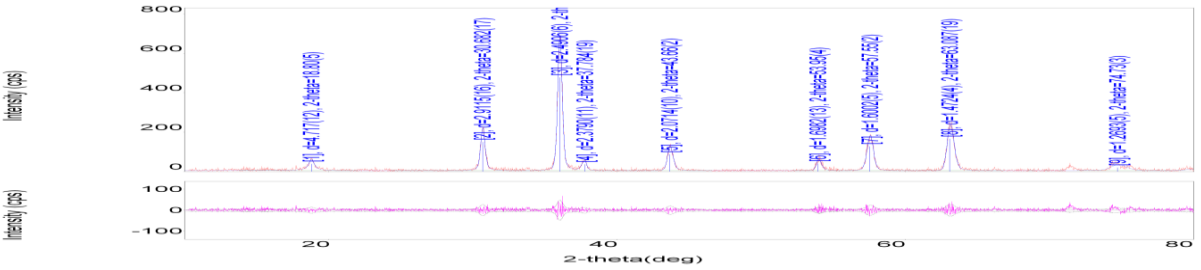


Fig.1(a)- $Ni_{0.5}Zn_{0.5}Fe_2O_4$, 2theta=36.031(degree)

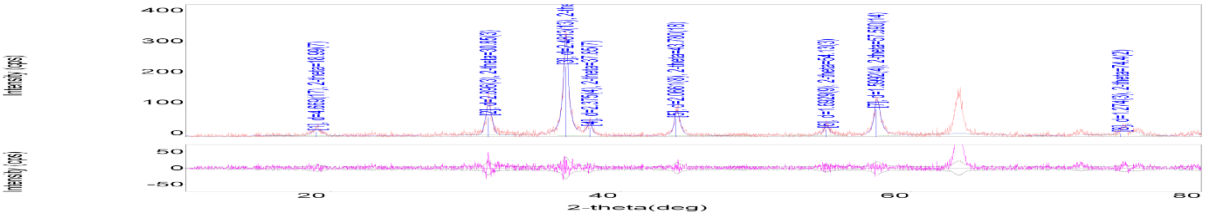


Fig.1(b) - $Ni_{0.5}Zn_{0.5}Fe_2O_4$ $Fe_{1.97}Pr_{0.03}O_4$,2theta=35.586(degree)

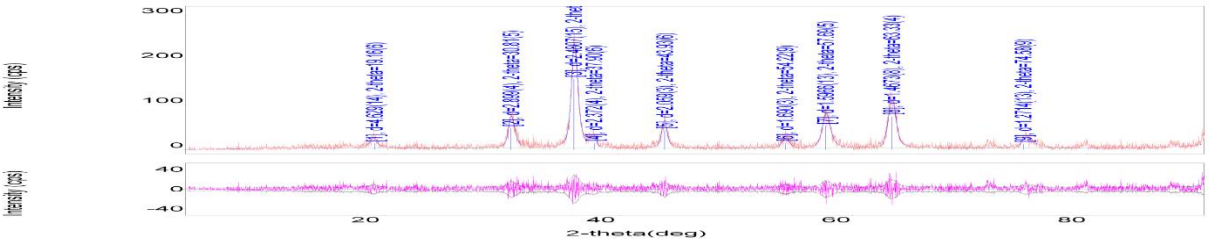


Fig.1(c) - $Ni_{0.5}Zn_{0.5}Fe_2O_4$ $Fe_{1.97}$ $Sm_{0.03}O_4$, 2theta=36.181(degree)

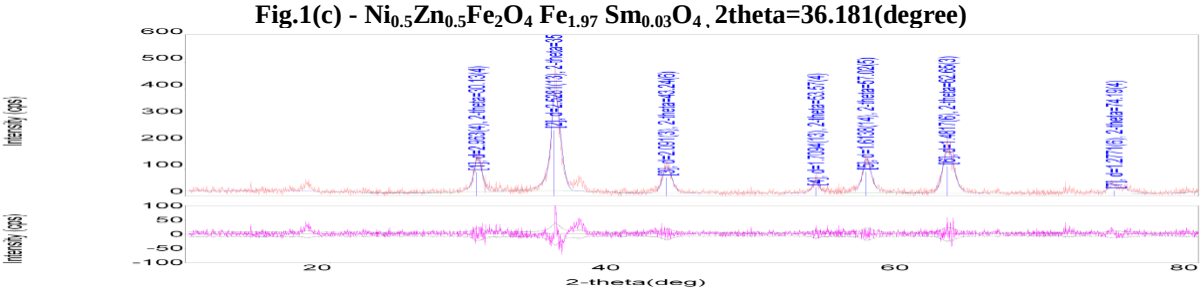


Fig.1(d) - $Ni_{0.5}Zn_{0.5}Fe_2O_4$ $Fe_{1.97}$ $La_{0.03}O_4$, 2theta=35.824(degree)

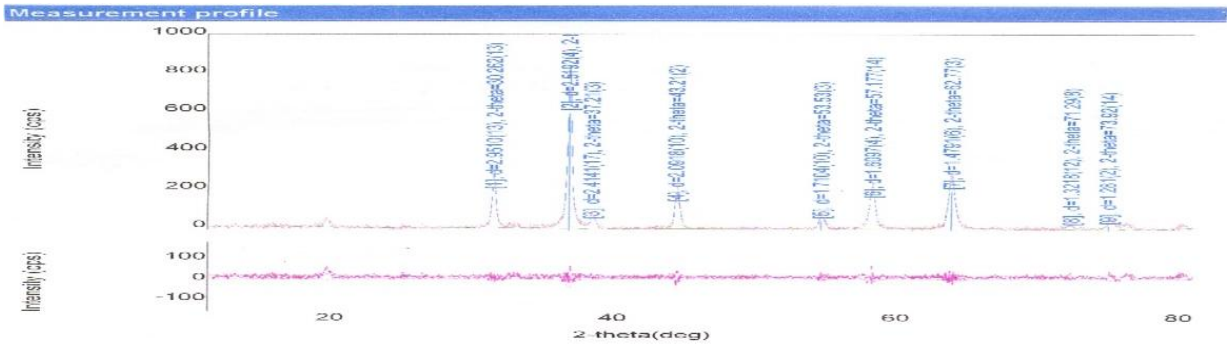


Fig.1 (e) - $Ni_{0.5}Zn_{0.5}Fe_2O_4$ $Fe_{1.97}$ $Gd_{0.03}O_4$, 2theta=35.609(degree)

Figure 1: X-ray Diffraction pattern of synthesized samples, with angular position (2 theta) of prominent peak position

Figure 1(a-e) shows the profile analysis of XRD pattern of all prepared materials. It is important to note that all samples $Ni_{0.5}Zn_{0.5}Fe_{1.97}R_{0.03}O_4$ nanoparticles (R= Pr, Sm, Gd and La) are single pure phase having spinel structure. It has been reported quite extensively that when spinel ferrite is substituted with rare earth elements, it is very difficult to get a single pure phase at low synthesis annealing temperature 450^{o}C for 2hr time. Whereas in the present work citrate precursor method leads the formation of single phase rare earth-substituted spinel ferrite giving this method an advantage over some of the others [11, 12, 13, 14, 15, 16].

The XRD spectrum also shows a broadening of diffraction peaks, indicating nano-size particle formation. Table 1 gives the crystalllite sizes of the substituted spinel ferrites having size 25nm for unsubstituted pure NiZn, 33nm for NiZnPr, 31nm for NiZnGd, 22nm for NiZnLa, 13nm for NiZnSm, calculated by Scherrer's formula [17] and corresponding lattice constants are 8.260Å, 8.360 Å, 8.355Å, 8.306 Å, and 8.227 Å are respectively. Substituation of Pr and Gd increases size of NiZn ferrite particles while La and Sm lowers the size. The peak intensities in XRD pattern follow the following order: NiZPr > NiZnGd > NiZnLa > NiZnSm. There is also a slight shifting in peak position. This gradual shift in XRD patterns as shown in Fig.1. The variation in lattice constant and very small distortion in the materials with rare earth ions substitution can be explained on the basis of the ionic radii of rare earth ions Gd^{3+}(94pm), Sm^{3+}(96pm) and Pr^{3+}(101pm).

Table 1: Detail of Structural and Magnetic measurement, Annealed at 450°C for 2hr

Sample name	Particle Size(peak Intensity Height), Lattice constant, 2theta position in degree	Magnetization
$Ni_{0.5}Zn_{0.5}Fe_2O_4$	25nm(478) , 8.260 Å, 36.031	50.692 emu/g
$Ni_{0.5}Zn_{0.5}Pr_{0.03}Fe_{1.97}O_4$	33 nm(793), 8.360 Å, 35.586	43.781 emu/g
$Ni_{0.5}Zn_{0.5}Gd_{0.03}Fe_{1.97}O_4$	31 nm (606), 8.355 Å, 36.609	47.875 emu/g
$Ni_{0.5}Zn_{0.5}La_{0.03}Fe_{1.97}O_4$	22 nm(374), 8.306 Å , 35.824	43.335 emu/g
$Ni_{0.5}Zn_{0.5}Sm_{0.03}Fe_{1.97}O_4$	13 nm(158), 8.227, Å 36.181	43.518 emu/g

Out of the samples given in Table 1, the sample, $Ni_{0.5}Zn_{0.5}Fe_2O_4$ which has been earlier reported [7] has been used for baseline data for comparison of XRD and Magnetization.

3.2. Magnetic Properties

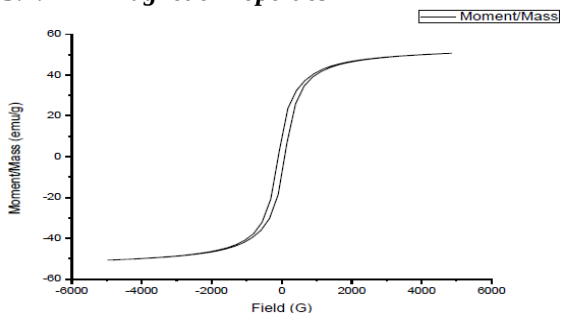


Fig.2(a) - $Ni_{0.5}Zn_{0.5}Fe_2O_4$

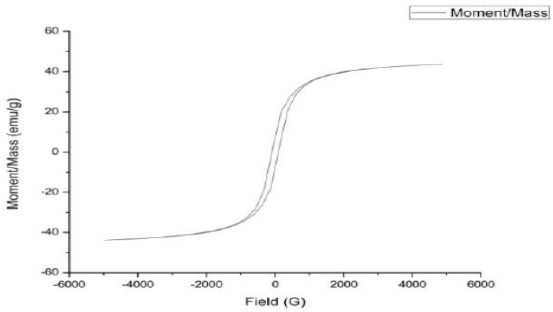


Fig.2(c)- $Ni_{0.5}Zn_{0.5}Fe_{1.97}Pr_{0.03}O_4$

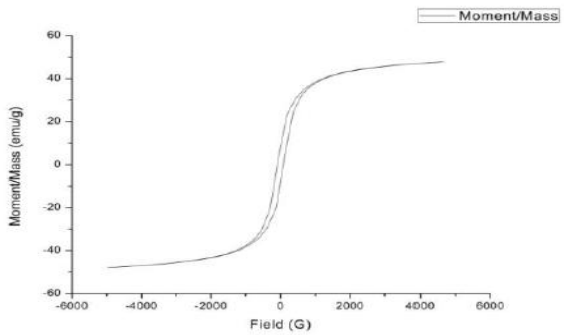


Fig.2(b)- $Ni_{0.5}Zn_{0.5}Fe_{1.97}Gd_{0.03}O_4$

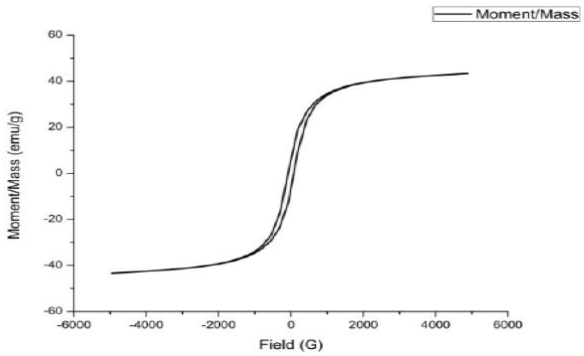


Fig.2(d) - $Ni_{0.5}Zn_{0.5}Fe_{1.97}La_{0.03}O_4$

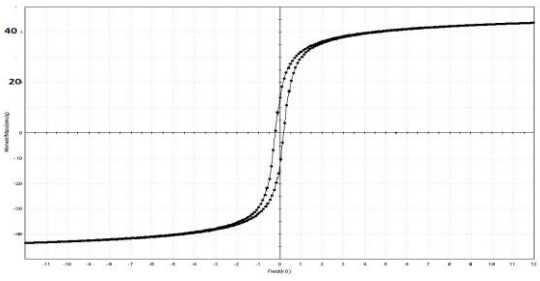


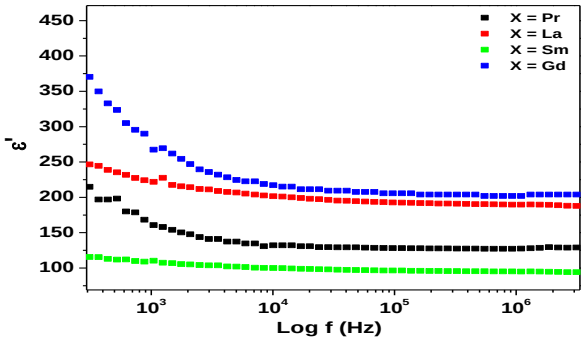
Fig.2(e)- $Ni_{0.5}Zn_{0.5}Fe_{1.97}Sm_{0.03}O_4$

Figure 2(a-e): Magnetization curves of prepared Magnetic nanomaterials

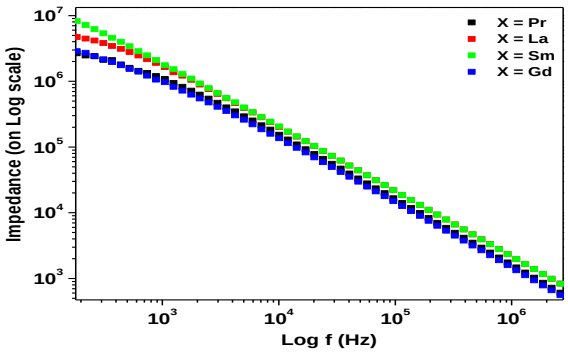
Hysteresis loops at room temperature, was measured for all the rare earth substituted Ni-Zn nano ferrite samples, are shown in Fig.2(a-e). The adding of much larger ionic radii Rare Earth ions, results in local distortion that induces a strong softening of the network and cation distribution took place.

Room temperature magnetization values for Pr, Gd, La, Sm substituted ferrites are 43.781, 47.875, 43.335 and 43.518 emu/g respectively as against 50.69 emu/g for pure(unsubstituted) $Ni_{0.5}Zn_{0.5}Fe_2O_4$ .The magnetic properties is mostly dependent upon the crystallite size, method of synthesis, cation substitution, anisotropy, tetrahedral-octahedral exchange interaction(A-B), domain wall movement, potential energy constant due perturbation occurring in iron-oxygen ion bond by introducing rare earth ions [3-10]. Substitution of rare earth ions of different ionic radius causes some distortion in the lattice of the sample causing change in the magnetization. Due to larger radius of Rare earth ions compared with that of Fe^{3+} ions, the lattice will be distorted and generate internal stress, which changes the lattice constant and crystalline size. For our samples, however, distortion is relatively small and it is worth noting that the material formed has single phase. Also due to large bond energy of R^{3+}- O^{2-} as compared to that of Fe^{3+}- O^{2-} more energy is required to force R^{3+}(R: Pr, Gd, La, and Sm) ions into the lattice. Hence rare earth substituted nanoferrites have higher thermal stability compared to pure Ni-Zn ferrites and more energy is needed for the substituted nano samples to complete crystalline and growth. But in our

Dielectric Properties:

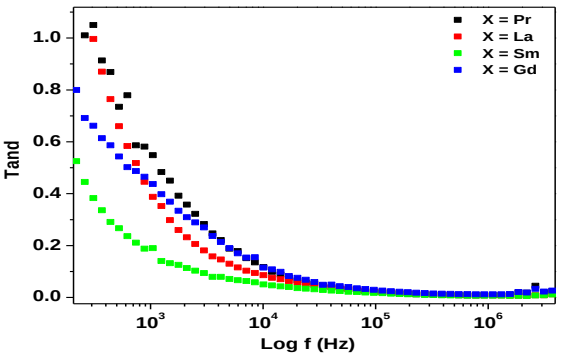


3(a). Room temperature dielectric constant versus frequency

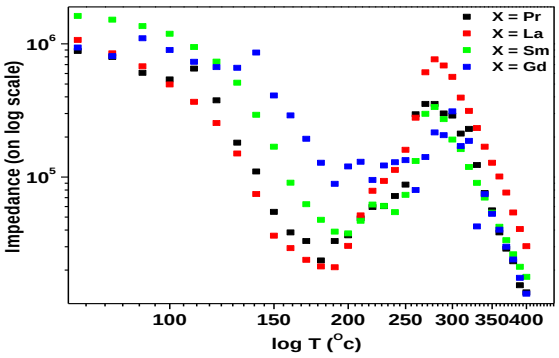


3(c). Room temperature impedance versus frequency

materials, annealed at temperature 450^0C, almost complete crystalline and grain growth formed without any amorphous phase. The slight changes in the magnetization and particle size is due to the doping concentration which is relatively large (0.03 mole of rare earth ions) compared to that of the other research groups [9, 13, 18]. At low concentration of rare earth ions, Gd, Pr, La and Sm causes a major change in centro-symmetric FCC structure of parent crystal that causes cation distribution and hopping of elctron and resultant polarization effect (It is stipulated that part of rare earth ions somehow enter in the octahedral sites that creates slight distortion and change in overall magnetization. The magnetization or demagnetization caused by domain wall movement requires less energy than that required by domain rotation and as a results domain wall movement is influenced by the particle size. Magnetization changes may be attributed due to domain wall movement in the direction of the applied field. Further, reduction of particle size and change in super exchange interactions can causes spin canting at the surface of the nanomaterials thus influencing magnetism of these nano samples. Thus from the magnetization data, it can be concluded that the presence of rare earth ions disrupts the normal preferences of cation distribution in spinel ferrites giving us an extra tool to control its magnetic properties. The spin orbit coupling (L-S coupling) is generally much stronger in rare earth ions compared to others. This is also one of the reason of changes in magnetic behavior (Table-1).



3(b). Room temperature dielectric loss versus frequency



3(d). Impedance versus temperature

3.3. Dielectric Properties: The dielectric constant of any material is due to electronic, ionic and interfacial polarizations. The variation of the dielectric constant and dielectric loss of $Ni_{0.5}Zn_{0.5}R_{0.03}Fe_{0.97}O_4$ (with R = Pr, Sm, Gd& La) as a function of frequency is shown in Fig.3 (a) and (b). It can be seen from the figure that the dielectric constant decreases with increasing frequency with a negative exponential type of dependence on log of frequency. The decrease of dielectric constant with increasing frequency as observed in the case of nickel–zinc ferrites is a normal dielectric behavior of spinel ferrites.

At lower frequency the dielectric constant decreases in the following order of R: Gd, La, Pr and Sm. In the frequency range 10^2-10^6 Hz highest dielectric loss was observed for Gd substituted ferrite while Sm, Pr and La substituted samples show very small loss. The high value of the dielectric constant in the present Gd-substituted ferrite may be attributed to either space charge polarization or large crystallite size comparative to the others samples. Electronic and ionic polarizations dominate at higher frequencies and their temperature dependence is generally insignificant. The dielectric loss of ferrimagnetic materials depends on other factors such as stoichiometry of Fe^{3+} and R^{3+}(R = La, Gd, Pr and Sm) and on structural homogeneity which in turn depend on the composition, particle size, cation distribution and annealing temperature. Dielectric peak loss occurs when the hopping frequency of electrons become equal to the frequency of the applied electric field. Hopping of electrons is facilitated by the increase in temperature and therefore the hopping frequency is found to increase with temperature. This results in an increase in space charge polarization and thus dielectric constant increases with temperature. The dielectric loss in ferrimagnetic material usually follows the DC electrical resistivity. This means low loss will occur in highly resistive materials. Such materials are important application-wise because of their better energy efficiency. At low frequencies, dipolar and interfacial contributions are dominant and both of these polarizations are temperature dependent. This explains the rapid increase in dielectric constant with temperature at low frequencies. Fig3(c) shows the variation of impedence with frequency for all rare earth nickel–zinc ferrites. An interesting observation is that Sm substituted ferrite possesses the smallest particle size, large magnetization compared to Gd substituted and unsubstituted Ni-Zn ferrite and minimum dielectric constant cum dielectric loss variation as compared to others samples. Similar behavior was also observed by some other research groups (19-24).Due to difference in ionic radii of rare earth ions with respect to Fe^{3+} in octahedral site the motion of charge carriers gets impeded. Therefore the resistivity verses temperature curve in the case of multiphase-phase system is a zigzag curve as shown in fig.3(d).This indicates that the change in the dielectric behavior with temperature may be due to a magnetic transition. At this low annealing temperature 450^{o}C, magnetic nanomaterials were prepared with remarkable magnetic properties [6, 25,26].

IV. CONCLUSION

In the present work, we have used Citrate precursor method to synthesize nanoferrites $Ni_{0.5}Zn_{0.5}Fe_{1.97}R_{0.03}O_4$; R= Sm, Gd, La and Pr by annealing for 2 hours at a single low annealing temperature of 450^{o}C. The change in lattice constant, prominent peak intensity height with rare earth ion substitution is due to different ionic radii of rare earth ions and perturbation in Fe^{3+}-O^{2-} bond due to substitution of rare earth ions. Similarly change in the crystallite size, saturation magnetization and dielectric behavior was observed on substitution by rare earth ions. Therefore such rare earth substituted nanoferrites can be treated as energy efficient magnetic materials which find applications Electronics, Environmental science, Medical science areas due to low dielectric losses in the high frequency range, purity of materials, and observed magnetic properties.

ACKNOWLEDGEMENTS

Author Rakesh Kumar Singh is thankful for financial support as a minor research project to UGC, approval No. F.PSB-43/11-12(ERO) at Dept. of Physics, Patna Women's College, Patna University. Authors are thankful to Dr. Manoranjan Kar, Dept. of Physics, IIT Patna for characterization help in dielectric study and Dr. R. K. Kotnala, Chief Scientist, CSIR-National Physical Laboratory (NPL) Delhi, for Magnetic measurements.

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AUTHORS PROFILE

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