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Structural, Magnetic & Dielectric behavior of $Ni_{0.5}Zn_{0.5}Fe_{1.99}R_{0.01}O_4$ Nanoparticles; R= Pr, Sm and Gd, synthesized using Citrate Precursor method, annealed at low temperature 450C.

Rakesh Kr Singh, Amarendra Narayan

Abstract-$Ni_{0.5}Zn_{0.5}Fe_{1.99}R_{0.01}O_4$ Nanoparticles**; R= Pr, Sm and Gd,** were synthesized using Citrate Precursor method, annealed at low temperature 450°C. X-ray diffraction (XRD) tool was used for estimation of average particle size and phase analysis. The mean particle size was found to be 25nm, 20nm, 29nm and 30 nm respectively with spinel structure. The variation in lattice constant with rare earth ion substitution was found to depend on the ionic radii of Gd^{3+} (94pm), Sm^{3+}(96pm), Pr^{3+}(101pm). Room temperature magnetic measurement was done by vibrating sample magnetometer (VSM). The magnetization values observed are 50.692 emu/g, 39.243 emu/g, 52.742 emu/g and 49.318 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. $Ni_{0.5}Zn_{0.5}Fe_{1.99}Sm_{0.01}O_4$ nanoparticles show a dielectric behavior appreciably different from $Ni_{0.5}Zn_{0.5}Fe_{1.99}Gd_{0.01}O_4$ and $Ni_{0.5}Zn_{0.5}Fe_{1.99}Pr_{0.01}O_4$ nanoparticles.

Index Terms **-** Rare-earth nanoferrites, Citrate Precursor method, magnetic and dielectric studies**.**

I. INTRODUCTION

Ferrite Magnetic nanomaterials are a special kind of functional engineering materials that have various applications in medical science, Electronics, Agriculture, Defence, Water purification, environmental science and so on[1,2,3,4,5]. In terms of magnetic behavior, Ferrite nanoparticles have special advantage over common materials. As for example Nano-iron oxide has excellent working performance in high density magnetic recording, its recording density is nearly 10 times more than normal iron oxides. Because of its special characteristics such as small size, better magnetic orientation, biocompatibility, biological degradability and active functional groups, biological affinity or reactivity, it can be combined with a variety of functional molecules such as enzymes, antibodies, cell, DNA or RNA, for drug delivery, cell separation, immobilization, immunoassay [6, 7, 8]. Magnetic, electrical, dielectric and optical properties of ferrite materials depend on method of synthesis, composition, substitution of metals ions, annealing temperature and purity of chemicals [9, 10, 11]. Preparation of nanoferrites by citrate precursor based chemical method has various advantages and these materials often show improved performance in given conditions [12, 13, 14].

Ni-Zn ferrites are well known spinel magnetic materials with a large number of technological applications emerging because of low eddy current losses, high resistivity and high frequency behavior [15]. Various new uses have emerged for Rare earth ions substituted ferrites in the last one decade such as in wastewater treatment, etc.[16,17].

In this work, we have used Citrate precursor method to synthesize Rare-earth element-substituted Nickel-Zinc nanoferrites $Ni_{0.5}Zn_{0.5}Fe_{1.99}R_{0.01}O_4$; R= Sm, Gd, and Pr, annealed for 2hr at a single **low annealing temperature 450 C.**

II. EXPERIMENTAL

Nitrates of Iron, Nickel, Zinc and Rare earth element-R, (R = Sm, Gd and Pr) were obtained from Sigma Aldrich and used without any further purification. Stock solutions were prepared using double distilled deionized water. The solutions were mixed together in stoichiometric proportion and stirred constantly for two hours until a brown solution was obtained. The mixed solution was dried up at 60 C in an air oven for 24 hours from which a brown fluffy mass was obtained. No precipitation was observed in the solution before drying in the oven. The dried brown solid material was annealed in muffle furnace at temperature 450C for two hour to obtain the Nanoparticles. X-ray diffraction pattern has been obtained using a Rigaku Miniflex - II. Magnetic characterization has been done using a Vibrating sample magnetometer at room temperature and magnetic field up to 5000G. Dielectric characterization has been done by making pellets of the respective samples using a LCR meter.

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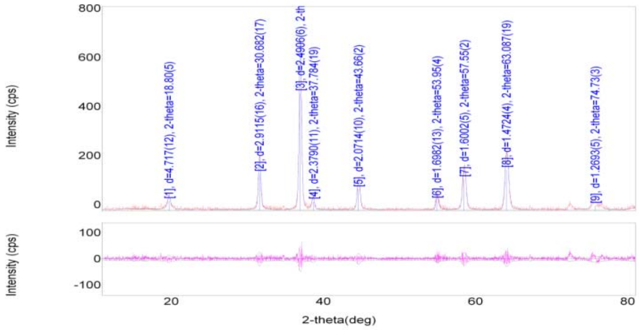
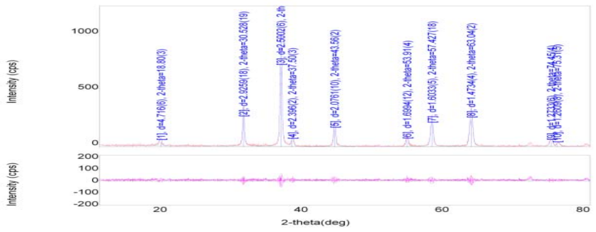
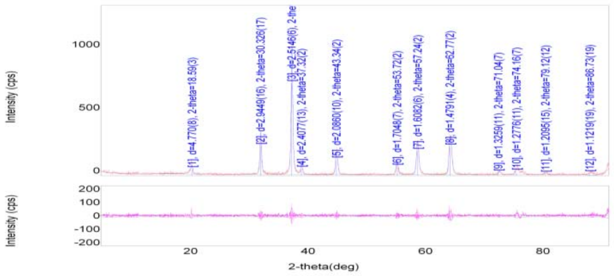
III. RESULTS AND DISCUSSION



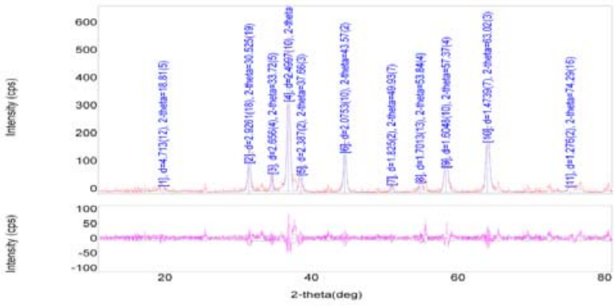
$Ni_{0.5}Zn_{0.5}Fe_2O_4$



$Ni_{0.5}Zn_{0.5}$ $Fe_{1.99}Pr_{0.01}O_4$



$Ni_{0.5}Zn_{0.5}Fe_2O_4$ $Fe_{1.99}$ $Sm_{0.01}O_4$



$Ni_{0.5}Zn_{0.5}Fe_2O_4$ $Fe_{1.99}$ $Gd_{0.01}O_4$

Figure.1: X-ray Diffraction pattern

X-ray diffraction (XRD) results shown in Figure-1 indicate that substitution with small concentration (0.01mole) of rare earth ions, Sm, Gd, and Pr allows their entrance into the spinel lattice**.** Peak intensity (height) was largest for Sm and Pr substituted samples with particle size 31nm and 29nm while Gd sample with particle size 20nm has smaller peak intensity.

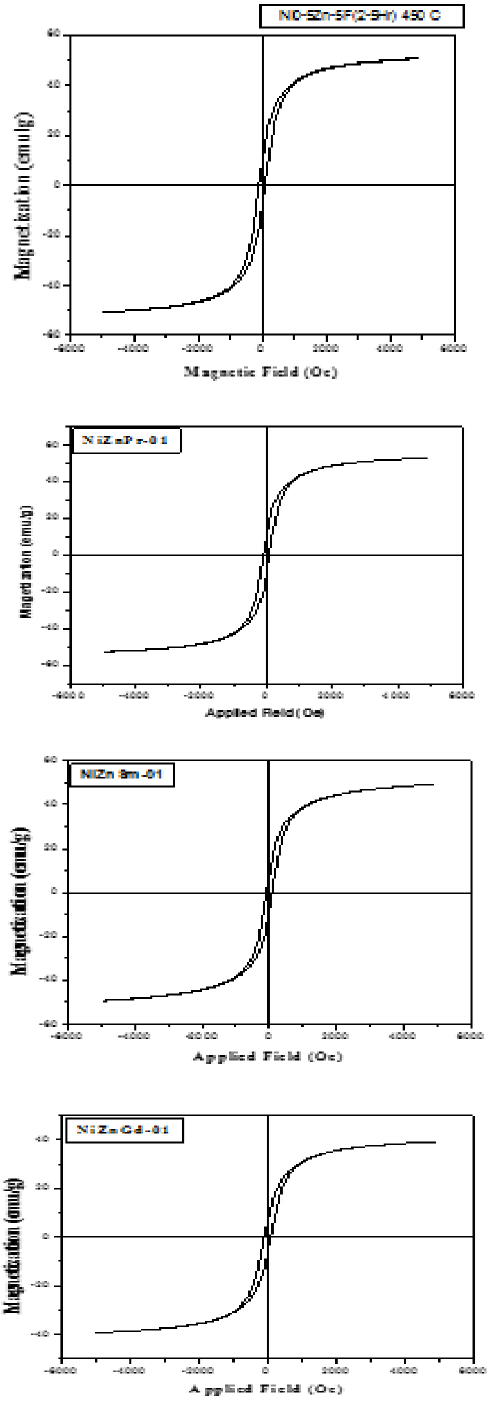


Fig.2: Magnetization Curve

Average particle size was calculated using Scherrer formula [18] and found to be 25nm, 20nm, 29nm and 30 nm respectively as shown in Table 1. The particles have spinel structure [ICDD file number 03-065-3107]. The small change in structural behavior can be easily explained as due to substitution by rare earth ions having large ionic radii, viz. Sm, Gd and Pr in place of smaller Fe^{3+}(0.67Å) ions. Rare earth ions present at the grain boundaries generally cause hindrance in the grain growth to large size. However additional very small intensity phases appear to be present in the samples. Such findings have also been observed by other research groups [19, 20, 21]. In the present work the increase in crystallite size of $Ni_{0.5}Zn_{0.5}Fe_{1.99}Sm_{0.01}O_4$ and $Ni_{0.5}Zn_{0.5}Fe_{1.99}Pr_{0.01}O_4$ as indicated by the prominent intensity peaks can be considered as a good indicator of improved crystallinity and better chemical homogeneity. The variation in lattice constant in the samples with rare earth ion substitution can be explained on the basis of the ionic radii of Gd^{3+}(94pm), Sm^{3+}(96pm), Pr^{3+}(101pm). The lattice constant, crystallite size and height of the prominent peak position are shown in Table1.

Magnetic measurements done at room temperature using Vibrating sample magnetometer **(VSM**) are shown in Figure 2. Magnetization value was found to be maximum (52.742 emu/g) for Pr substituted ferrites while in Sm and Gd substituted ferrites reduced magnetization values i.e. 49.318 emu/g and 39.243 emu/g were observed. Magnetic properties of Ferrite nanomaterials mainly depend on the Grain size, Cation distribution and Superexchange interaction characteristics. Substitution by rare earth ions of different size and changes in A-B interactions causes Canting of spin at the surface of nanoparticles that changes the magnetization of the samples (22, 23, 24, 25).

A comparative study of pure $CoFe_2O_4$ nanoparticles and La-doped $CoFe_2O_4$ nanoparticles, prepared by microemulsion route has been reported by Simona Burianova et al. [26]. They observed that the doping of small amount of La^{3+}ions (up to 3 molar %) causes significant reduction in the particle size compared to undoped samples prepared using identical route. Non-negligible canting angles up to 40° were observed in the La-doped samples. The presence of Spin-Surface effect was also supported by magnetic measurement as the magnetization did not saturate even in the presence of considerably high magnetic fields (7 Tesla). Moreover, significantly reduced values of the saturation magnetization were obtained. The observed features originated by the surface spin disorder in nano-sized particles were explained in the framework of the Core-shell model.

M-type strontium ferrites, $Sr_{0.8}La_{0.2}Fe_{12}O_{19}$ have been prepared by conventional ceramic process by Wandee onreabroy et al. [27]. Microstructural analysis of the $SrFe_{12}O_{19}$ and $Sr_{0.8}La_{0.2}Fe_{12}O_{19}$ specimens, sintered at different temperatures shows that mean grain sizes of $SrFe_{12}O_{19}$ ferrites were larger than that of $Sr_{0.8}La_{0.2}Fe_{12}O_{19}$ ferrite and increased with increasing sintering temperature. Maximum saturation magnetization value of 102 emu/g were obtained for the $SrFe_{12}O_{19}$ ferrite sintered at 1150C and for the $SrFe_{12}O_{19}$ and $Sr_{0.8}La_{0.2}Fe_{12}O_{19}$ ferrites sintered at 1300C, respectively.

The insertion of small amounts of different R (III) cations (R=Ruthenium, Yttrium and rare-earth cations) into a nickel zinc ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) has been also investigated by Elsa E Sileo et al. [28]. XRD studies have been carried out in order to determine if the R (III) cations enter the spinel structure. Samples with several $Ni_{0.5}Zn_{0.5}Fe_{2-y}R_yO_4$ compositions were prepared by the auto-combustion method. In all cases, XRD measurements show distortions in the spinel cell and, in some cases, the formation of various rare earth iron oxides.

Sample name	Particle Size(peak Intensity Height in cps)/ Lattice Constant	Magnetization
$Ni_{0.5}Zn_{0.5}Fe_2O_4$	25nm(478)/8.2603Å	50.692 emu/g
$Ni_{0.5}Zn_{0.5}Gd_{0.01}Fe_{1.99}O_4$	20nm(324)/8.2905 Å	39.243 emu/g
$Ni_{0.5}Zn_{0.5}Pr_{0.01}Fe_{1.99}O_4$	29nm(716)/ 8.3399 Å	52.742 emu/g
$Ni_{0.5}Zn_{0.5}Sm_{0.01}Fe_{1.99}O_4$	31nm(731)/ 8.2921 Å	49.318 emu/g

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

Dielectric behavior: Dielectric constant is a measure of the degree to which a medium gets polarized in applied electric fields. Figure 3. shows the variation of dielectric constant with frequency. The dielectric constants in all cases except $Ni_{0.5}Zn_{0.5}Sm_{0.01}Fe_{1.99}O_4$ show a trend of dielectric constant decreasing with frequency in frequency range 100 to 10^4 Hz. Ferrimagnetic materials normally display decrease of dielectric constant with increasing frequency. Such behavior may be explained as due to the interfacial polarization predicted according to Maxwell-Wagner principle [29]. According to this principle, the ferrite materials is assumed to be made of two layers in which one layer is a conducting layer consisting of large ferrite grain interiors and the other being the grain boundaries that are poor conductor. The polarization in ferrites is also explained through a mechanism similar to that given by Rabinkin and Novikova [30]. In this mechanism electron exchange between Fe^{2+} and Fe^{3+} causes the local displacement of electrons in the direction of the applied field which determines the polarization.

A similar trend was also observed by other groups [31-34]. When the hopping frequency is nearly equal to that of external applied electric field a maximum loss tangent may be observed [34]. A detailed explanation can be given for the occurrence of the maxima in tan δ versus frequency curves in some Ferrites as explained by Iwauchi [35].

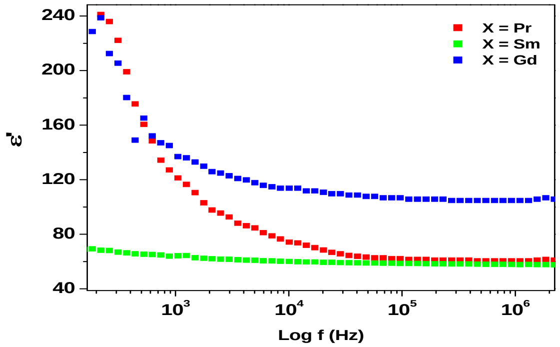


Fig.3. Room temperature dielectric constant versus frequency

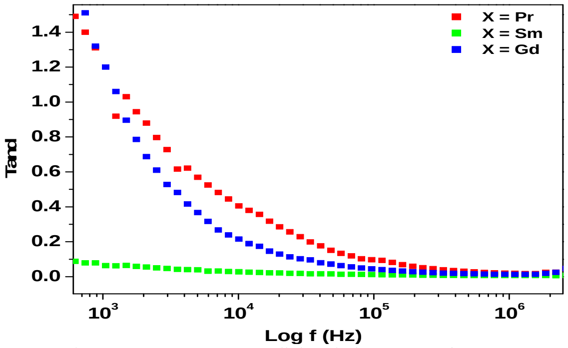


Fig. 4. Room temperature dielectric loss versus frequency

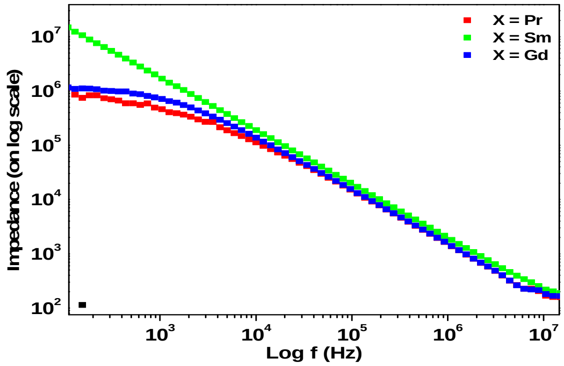


Fig.5: Room temperature impedance versus frequey

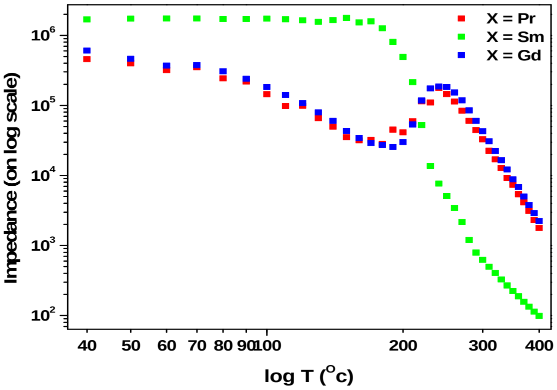


Fig.6.Impedence versus temperature

Figure 3- 6. . Room temperature Dielectric Study (Plotted at log scale) of $Ni_{0.5}Zn_{0.5}X_{0.01}Fe_{1.99}O_4$ for X = Pr, Sm and Gd annealed at 450C

Figure 4 shows curves of the dielectric loss at room temperature as a function of the applied frequency. The results shows decrease in dielectric loss with applied frequency which reaches a constant value at high frequencies, but in the case of $Ni_{0.5}Zn_{0.5}Sm_{0.01}Fe_{1.99}O_4$ nanoferrites, we get almost constant dielectric constant and dielectric loss with increasing frequency. Generally decrease in dielectric loss is attributed to the decrease in the polarization of the sample as the dipoles cannot follow-up the field variation [28, 29, 36]. Impedance versus frequency and Impedance versus temperature curves are shown in Figure 5 and Figure 6. Also the room temperature impedance was highest for $Ni_{0.5}Zn_{0.5}Sm_{0.01}Fe_{1.99}O_4$ substituted Ferrite at low frequencies and it decreased steadily with increasing frequency while the decrease of impedance with temperature showed a sharp decrease around 250C. For the other samples, the impedance decreased slowly at low frequencies and afterward it decreased at a faster rate similar to other work [31, 32]. Impedance versus temperature curves for other samples peak around 230C in an overall decreasing trend as shown in Figure 6. A detailed study of dielectric behavior of nano ferrites has been reported by Iftikhar Gul [37].

Conclusion: In this work, we have used Citrate precursor method to synthesize nanoferrites $Ni_{0.5}Zn_{0.5}Fe_{1.99}R_{0.01}O_4$; R= Sm, Gd, and Pr by annealing for 2 hours at a single low annealing temperature of 450C. The increase in crystallite size of $Ni_{0.5}Zn_{0.5}Fe_{1.99}Sm_{0.01}O_4$ and $Ni_{0.5}Zn_{0.5}Fe_{1.99}Pr_{0.01}O_4$ with prominent intensity peaks can be considered as a good indicator of improved crystallinity and better chemical homogeneity. Magnetization value was found maximum (52.742 emu/g) for Pr substituted ferrites while in Sm and Gd substituted ferrites smaller magnetization values of 49.318 emu/g and 39.243 emu/g were respectively observed. The increase in lattice constant with rare earth ion substitution can be explained on the basis of the progressively increasing ionic radii of Gd^{3+}(94pm), Sm^{3+}(96pm), Pr^{3+} (101pm). A smooth change in the crystallite size and saturation magnetization was observed on substitution by rare earth ions. Dielectric constant and dielectric loss decrease with increasing frequency except for $Ni_{0.5}Zn_{0.5}Sm_{0.01}Fe_{1.99}O_4$, which shows almost constant dielectric constant and dielectric loss with increasing frequency.

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