"Effect Of Composition On Structural And Magnetic Properties Of Nano-Crystalline Mg_{0.6-X}ni_xzn_{0.4}Fe₂O₄ Spinal Ferrite"

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Abstract:

In the present work nanoparticles of magnesium nickel zinc Ferrite ($Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$) for x = 0.3 was synthesized by sol-gel auto combustion method. Prepared sample were sintered at different temperature viz $150^{\circ}C$, $300^{\circ}C$ and $450^{\circ}C$. Formation of single phase cubic spinel structure without addition of other peaks was confirmed from XRD data. Infrared spectra was recorded at room temperature for different wave numbers ranging from $250 - 500 \text{ cm}^{-1}$. Surface morphology of sintered sample was studied using SEM. The magnetic properties of prepared sample at different sintering temperature were studied using VSM. Chemical composition for prepared sample was confirmed by using EDAX.

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I. Introduction:

Nowadays spinal ferrite nanocrystal is been widely investigated due to their remarkable electrical, magnetic properties and wide practical applications in memory storage system, medical diagnosis, magnetocaloric refrigeration and ferrofluid technology [1]. To see the demand of high performance device, an important step is to synthesize ferrite crystals at nanoscale. At this scale, crystals exhibit single domain state and therefore the domain wall resonance is avoided and material can work at higher frequencies [2]. A number of techniques are used in the formation of nano-sized ferrites such as organic precursors [3], cathodic electrophoretic deposition (EDP) [4], mechanochemical synthesis [5], reverse micelle [6], electrochemical deposition [7] and utilizing egg-white [8]. More recently, cost effective sol-gel auto combustion method is being widely used.

II. Experimental

Magnesium nickel zinc nanoparticles ferrite was synthesized by sol-gel auto combustion method by using precursor such as zinc nitrate, ferric nitrate, magnesium nitrate and citric acid. All reagents are analytical reagent (AR) grade. The stoichiometry formula used for the preparation of nanoparticles is $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = 0.3). Analytical grade reagent precursors were dissolved in distilled water separately for sol-gel method. The solution was stirred in a magnetic stirrer for 4 hours with constant heating. The sol then gets converted into gel. Further stirring converts the gel to ash. The ash was pulverised using agate mortar for the formation of nanoparticles. Lastly the prepared powder was sintered at different temperature viz 150°C, 300°C and 450°C for an hour.

III. Result and Discussion

Prepared sample was characterized by using X-ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive analyzing X-ray (EDAX), Fourier transformation infrared spectroscopy (FTIR) and Vibrating sample magnetometer (VSM) and results are discussed below.

XRD

The graph of diffraction intensity for different diffraction angle (2θ) for the composition of Mg_{0.6-x}Ni_xZn_{0.4}Fe₂O₄ nanoparticles for x = 0.3 for different sintering temperatures such as 150^oC, 300^oC and 450^oC is as show in the figures (Fig.1). At this temperature, the single phase spinel Mg_{0.6-x}Ni_xZn_{0.4}Fe₂O₄ was formed. The most abundant crystalline phase was observed for (311) which correspond to Fe₂O₄ and the peaks for other miller indices (220), (400), (422), (333) and (440) also matched well with standard data of the reflections of the

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magnesium-nickel-zinc ferrite crystals. The 2θ position are in agreement with standard JCPDS. The value of these peak positions are used to find the value of interplaner spacing (d) among the Bragg planes. By using Scherer formula average crystalline size (D) of all powder samples was calculated and it gradually decreases with increasing sintering temperature. The lattice parameter (a) was calculated using miller indices (hkl) and the interplaner spacing (d). These values increase with decrease sintering temperature.



Fig 1. XRD of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ for x = 0.3 at temperature 150°C, 300°C and 450°C

Different parameters calculated from XRD:

Temperature	150°C	300°C	450°C
X-Ray density (gm/cc)	5.245	5.156	5.128
Lattice Constant (Å)	8.311	8.359	8.374
Ionic Bond Length (A-O) (Å)	1.799	1.803	1.81
Ionic Bond Length (A-O) (Å)	2.07	2.089	2.093
Ionic radii R _A (Å)	0.399	0.403	0.413
Ionic radii R _B (Å)	0.677	0.689	0.6936
Particle size (nm)	38	37	34

SEM

Scanning electron microscopy (SEM) of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ nanoparticles for x = 0.3 for different sintering temperature such as 150°C, 300°C and 450°C is show in fig. 2. Average grain size calculated was found to be 200nm – 300nm.

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Fig 2. SEM of Mg_{0.6-x}Ni_x $\overline{Zn_{0.4}Fe_2O_4}$ for x = 0.3 at temperature 150^oC, 300^oC and 450^oC

EDAX

Energy dispersive analyzing X-ray (EDAX) as shown in fig.3 analysis of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ nanoparticles for x = 0.3 for different sintering temperature such as 150°C, 300°C and 450°C was carried out for the conformation of elements present in the prepared sample with those which are used in precursor. The presence of elements Fe, Mg, O, Ni and Zn are clearly seen throughout the sample.



Fig 3. EDAX of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ for x = 0.3 at temperature 150°C, 300°C and 450°C

FTIR

FTIR spectrum analysis can be used for identification of molecular bond attachment as a residual functional group of the chemical which are used in the synthesis of the sample. The formation of Mg_{0.6-x}Ni_xZn_{0.4}Fe₂O₄ nanoparticles for x = 0.3 for different sintering temperature such as 150^oC, 300^oC and 450^oC for grain size between 250–500 cm⁻¹ was investigated using Fourier transform infrared spectroscopy (FTIR) and results are presented in fig. 4. It can be seen that for high wave number $v_1 = 350 \text{ cm}^{-1}$ and low wave number $v_2 = 300 \text{ cm}^{-1}$ the coordination complex is tetrahedron complex (M_{tetra}-O) and octahedral complex (M_{octa}-O) respectively. The vibrational modes for tetrahedral cluster are generally higher than the vibrational modes of octahedral cluster which was observed due to the bond length of each cluster.



Fig. 4 FTIR spectra of Mg_{0.6-x}Ni_xZn_{0.4}Fe₂O₄ for x = 0.3 at temperature 150^oC, 300^oC and 450^oC

VSM:

The magnetic characteristic of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ nanoparticles for x = 0.3 for different sintering temperature such as $150^{\circ}C$, $300^{\circ}C$ and $450^{\circ}C$ were carried by using Vibrating Sample Magnetometer (VSM) at room temperature. VSM of the sample is shown in fig. 5. The saturation magnetization is quite high of 32emu/gm. From graph there is a formation of loop which shows the prepared material is ferromagnetic in nature at room temperature.



Fig. 5 M-H Hysteresis loop of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ for x = 0.3 at temperature 150°C, 300°C and 450°C

IV. Conclusion:

 $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ for x = 0.3 nanoparticle have been successfully synthesized by using metal nitrate and citrate sol-gel auto combustion route with enhanced magnetic properties. X-ray diffraction confirms the formation of single phase cubic spinal structure of sample. SEM analysis revealed average grain size of 200nm

to 300nm.The magnetic properties of prepared sample for different sintering temperature are carried out by using VSM. FT-IR Spectra confirmed the formation of ferrite.

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