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Study of structural properties of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ ferrite Nanoparticles prepared by Sol Gel auto combustion method

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Abstract

In the present study, nanoparticles of Mg-Ni-Zn ferrite with general formula $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.5) have been prepared by using sol gel auto combustion method. The sintering process of synthesized compound was carried at temperatures 150^0C, 300^0C and 450^0C for an hour duration. For the study of structural and morphological properties, various physical and chemical methods were used. For the confirmation of single-phase cubic spinel structure; the X-ray diffraction method was used. The lattice parameter, bond length (A – O and B - O), ionic radii (R_A and R_B), particle size x-ray density were determined from X-ray diffraction pattern. From calculations, it is found that crystalline size decreases from 37 nm to 35 nm and the lattice constant (a) increases from 8.3320 A.U to 8.3853 A.U as temperature increases from 150^0C to 450^0C. The grain size of prepared sample for various sintering temperatures were investigated by using Scanning Electron Microscope (SEM) image. It is found that the average grain size varies from 320nm to 210nm for increasing sintering temperature. EDAX analysis of X- ray diffraction shows the presence of used metals in the appropriate composition.

Keywords: Sol gel, auto combustion, x-ray density, XRD, SEM, EDAX, grain size, bond length, ionic radii, lattice constant.

Introduction

In the recent years; ferrite nanoparticles are found wide applications in various fields due to their attractive structural, electrical and magnetic properties. It finds applications in biological separations, enzyme and protein immobilization, targeted drug delivery, RNA and DNA purification cell sorting, MRI, and biosensors [1,2]. In the present study, we report the synthesis of Mg-Ni-Zn nanoparticles by sol gel auto combustion method. Ferrites are the ferromagnetic materials and possess the combined properties of electrical insulator and magnetic conductor. They have been extensively investigated and being a subject of great interest because of their importance in many technological applications such as antenna rods, transformer cores, magnetic data storage etc. [3]. The ferrites are commonly prepared by ceramic technique, precipitation method, sol-gel method, spray pyrolysis, chemical bath deposition method etc. In the recent years, nanoscale spinel ferrite particles received a considerable attention because of their interesting magnetic properties [4]. Spinel ferrites are compounds of iron oxides and some transition metal oxides. They show important electrical and magnetic properties, which made them extensively useful in technological and industrial applications such as magnetic storage in microwave devices [5,6]. Various substituents of magnetic and nonmagnetic metals like Ni, Zn, Al, etc have been incorporated in ferrite to modify their structural, electrical and magnetic properties.

Ferrites mostly exhibits spinel structure with general formula AB_2O_4 (7) where A cations are present in tetrahedral sites and B cations are present in Octahedral site. Ferrites have wide applications in microwave devices and magnetic recordings. The study of structural and morphological properties of ferrites reveals its physical and chemical behavior. For making high density information storage devices, nanoferrite materials are most useful. Because of their high electric resistivity and low eddy current loss, ferrite nanoparticles are most important. The ferrites are structure sensitive material [8]. Because of its various applications and easiness in preparation, in the present work, sol-gel auto combustion method is used for the preparation.

Methodology

In the present study, the Magnesium Nickel Zinc ferrite powder was prepared by sol-gel auto combustion method. The general formula used for preparation is $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = 0.5). The A.R grade nitrates such as magnesium nitrate, nickel nitrate, zinc nitrate and ferric nitrate metals used for the preparation of the compound. For the formation of homogeneous mixture of metal cations, the citric acid ($C_6H_8O_7$) plays an important role as a fuel. The metal nitrate and fuel ratio are used in the proportion 1:3. For the formation of sol, all metal nitrates and fuel citric acid were dissolved in distilled water. The pH of the solution was maintained to 7 by slowly adding ammonia solution. The magnetic stirrer was used for continuously stirring. The stirring of solution was continued till the gel is formed at constant temperature 100^0C. The gel gets converted into ash form after heating at about 4 to 4.5 hours. The prepared powder was crushed using agate mortar to obtain nanoferrite. The prepared powder was sintered at temperatures 150^0C, 300^0C and 450^0C for 1 hour.

The general chemical reaction:

$0.1Mg(NO_3)_2.6H_2O + 0.5Ni(NO_3)_2.6H_2O + 0.4Zn(NO_3)_2.6H_2O + 2Fe(NO_3)_3.9H_2O + 3C_6H_8O_7 \rightarrow (Mg_{0.1}Ni_{0.5}Zn_{0.4}Fe_2O_4) + 4N_2\uparrow + 18CO_2\uparrow + 36H_2O$

Result and Discussion

The synthesized sample was characterized by using EDAX, XRD, and SEM. The results obtained are presented below;

1 XRD studies

Figure-1 shows the XRD patterns of composition $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.5). The study of XRD patterns shows that all the prepared samples are single

phase spinel cubic structure [9]. It is found that the Bragg's angles of corresponding peaks in the XRD pattern approximately matches with the characteristics of the reflection peaks of Mg- Ni- Zn ferrites which is reported in JCPDS by Barakat, m et al., J. Therm Anal 37.241 (1991) [10].

Fig. 2 shows variation of lattice constants (a) with temperature. From graph it is seen that lattice constant (a) increases with increases in sintering temperature (T). The metal oxygen bond length (A-O) on tetrahedral site is calculated by using formula

$$A–O = (u – 0.25\ ).a.\sqrt{3} \text{ ---------- (1)}$$

The metal oxygen bond length (B-O) on octahedral site is calculated by using formula

$$B–O = (0.625 – u)\ a \text{ ---------- (2)}$$

Where, u is known as oxygen ion parameter.
From the calculation, it is found that the bond length (B–O) of octahedral site is greater than the bond length of tetrahedral site (A–O) for all the composition. The bond length (A–O) and the bond length (B–O) founds to be increases with increases of sintering temperatures.

The determination of ionic radii on A site (tetrahedral site) and B site (octahedral site) have been obtained by using equations (3) and (4)

$$R_A = (u – 0.25).\ a.\sqrt{3}\ – r(O_2)^- \text{ --------- (3)}$$

$$R_B = (0.625 – u)\ a – r(O_2)^- \text{ ----------(4)}$$

Where, $r(O_2)^-$ is known as the ionic radii of the oxygen.
From the calculations; it is found that the ionic radii R_A and R_B increases with increase in sintering temperature

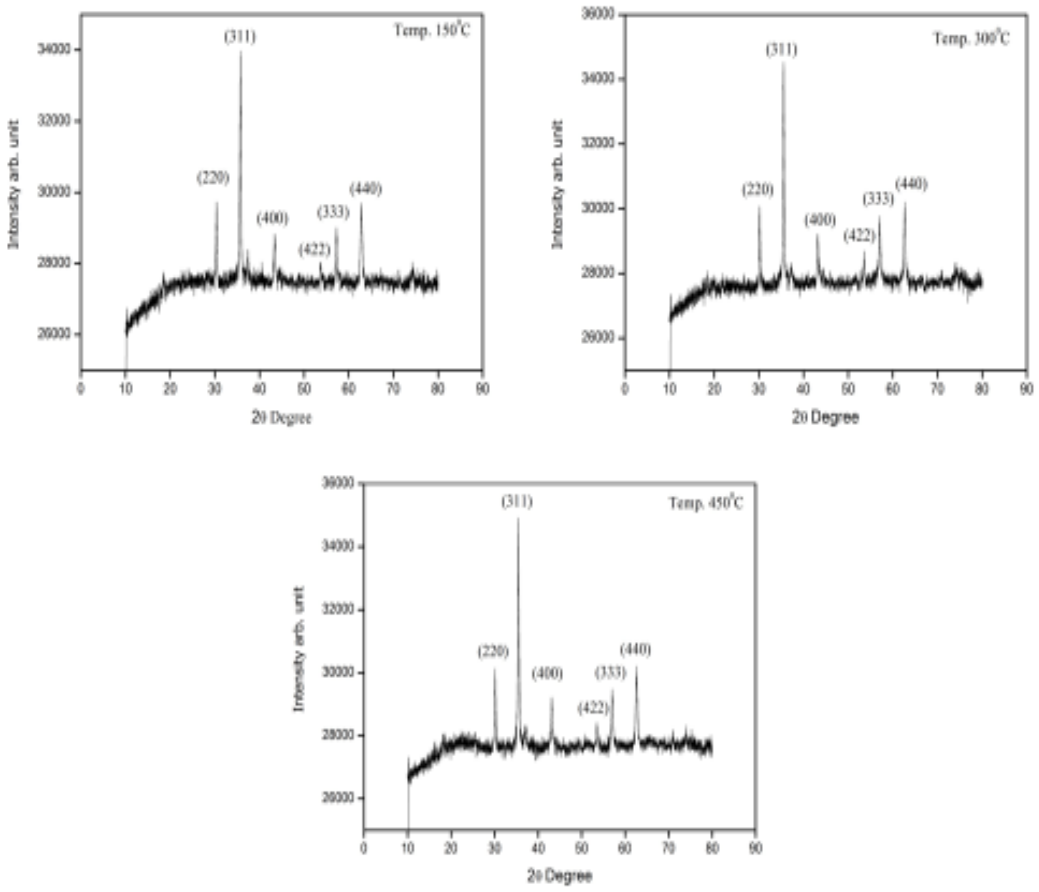

Fig.1: XRD of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.5) for temperatures 150^{0}C, 300^{0}C and 450^{0}C.

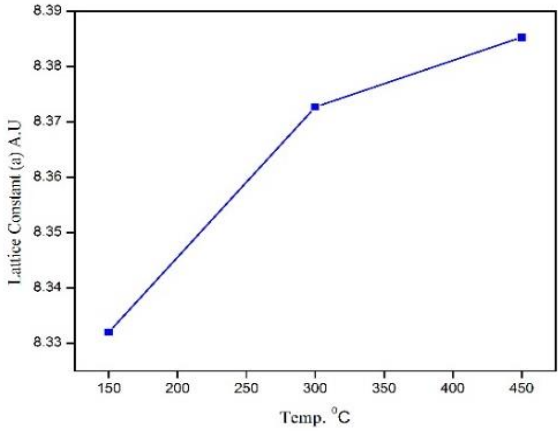

Fig. 2: Lattice Constant (a) Vs Temperature (T)

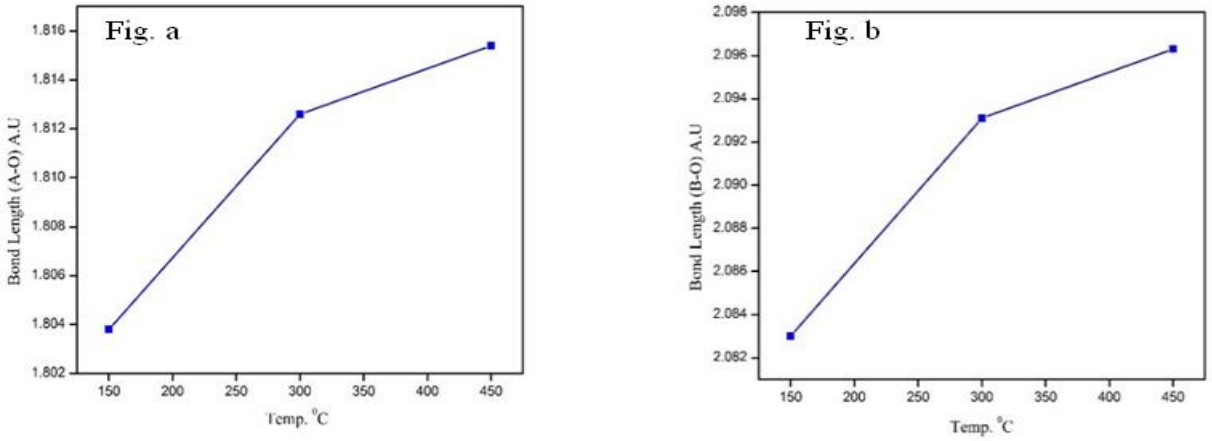

Fig.3: (a) Bond Length (A-O) A.U Vs Temperature (b) Fig. Bond Length (B-O) A.U Vs Temperature

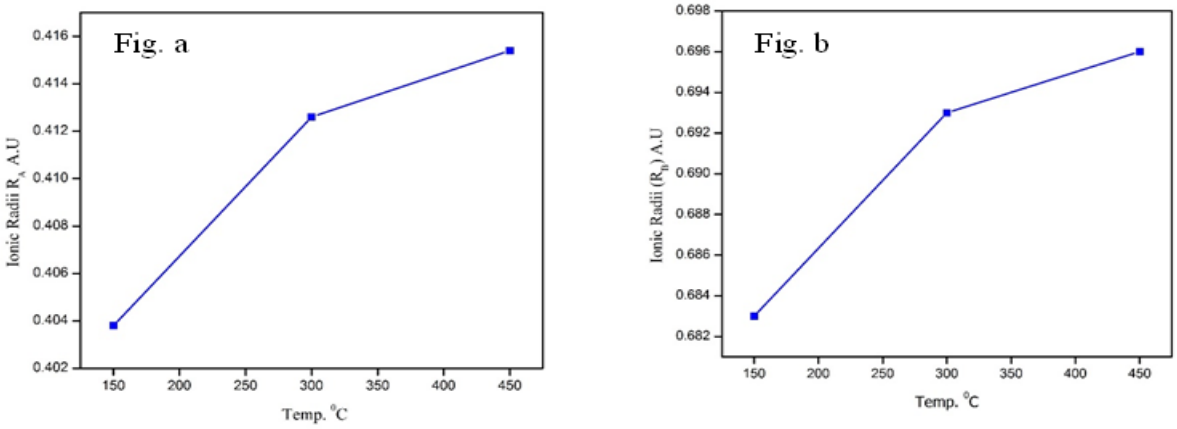

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Fig.4: (a) Ionic Radii (R_A) A.U Vs Temperature (b) Ionic Radii (R_B) A.U Vs Temperature

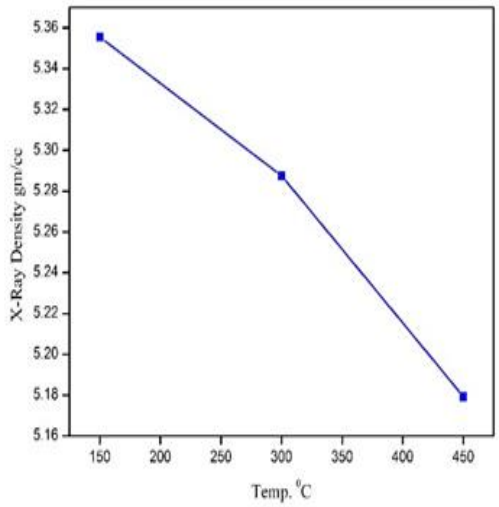

Fig.5: X-Ray Density (D_x) Vs Temperature

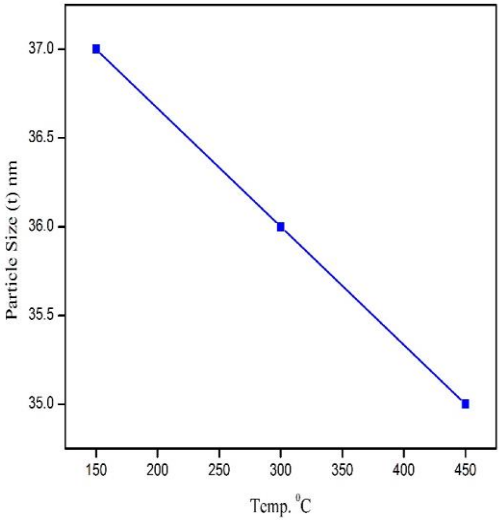

Fig.6: Particle Size (t) nm Vs Temperature

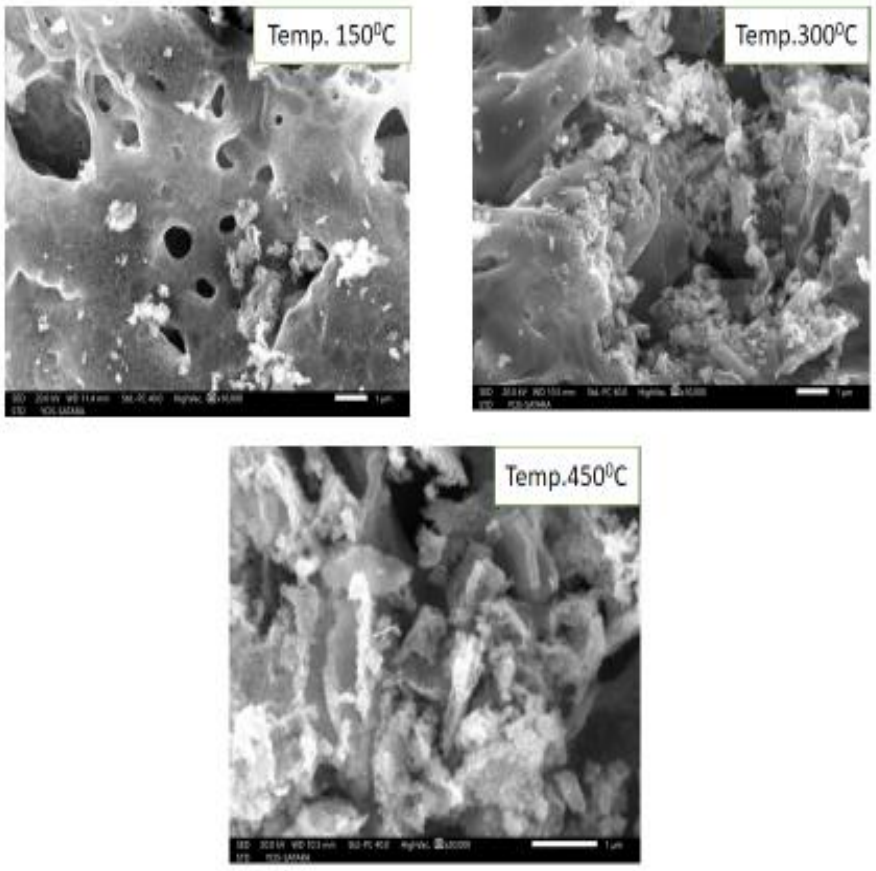

Fig:7 SEM Micrograph of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.5)

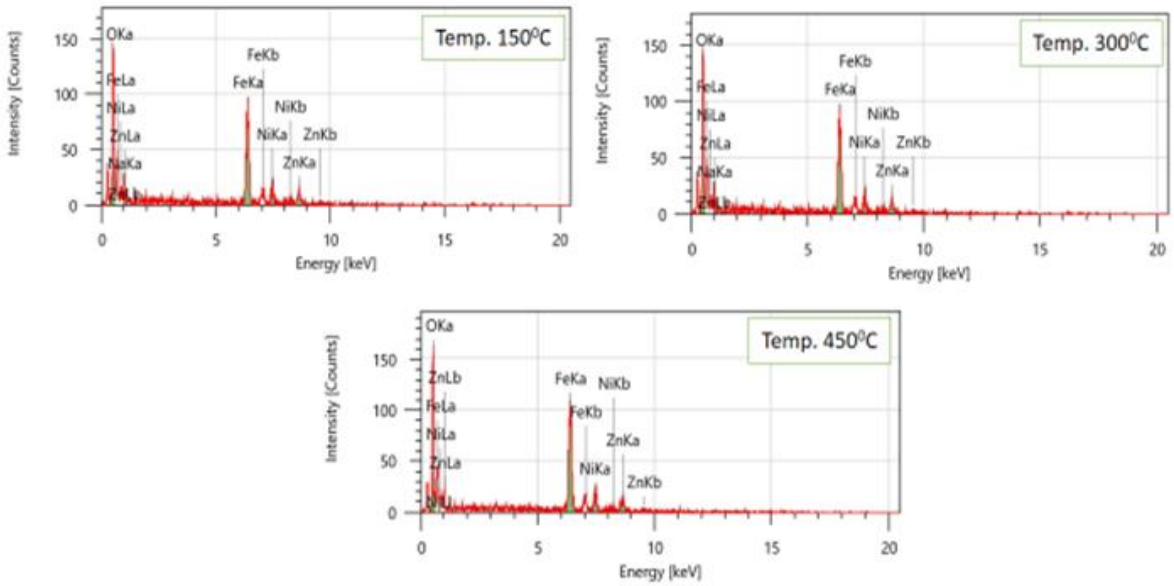

Fig.8: EDAX Pattern of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.5)

Table:1 Different parameters from X-ray spectra.

X	Temp. ^{0}C	Lattice Constant (a) A.U	X-Ray Density D_x gm/cc	Ionic Bond Length (A-O) A. U	Ionic Bond Length (B-O) A. U	Ionic Radii (R_A) A. U	Ionic Radii (R_B) A. U	Particle Size (t) nm
0.5	150	8.3320	5.3555	1.8038	2.083	0.4038	0.683	37
	300	8.3727	5.2876	1.8126	2.0931	0.4126	0.693	36
	450	8.3853	5.1793	1.8154	2.0963	0.4154	0.696	35

Fig. 5 (a and b) shows the variation of X-Ray density (Dx) with sintering temperature (T). From the graph it is seen that X-Ray density decreases with increases in sintering temperature (T).

Fig. 6 shows the variation of particle size (t) with sintering temperature (T). From the graph it is seen that particle size decreases with increases in sintering temperature (T).

2 Morphological studies

Figure 7 shows the SEM image of prepared ferrite sample for sintering temperatures 150^{0}C, 300^{0}C and 450^{0}C.

It is found that the prepared samples have large clusters of ferrites formed by assembling of small particles of nearly consistent in size with good spherical nature.

The surface morphology containing full of small grains. It proves that for the used sintering temperatures the microstructure is completely formed. On drawing various intersecting lines, the grain size is determined. With increase in sintering temperature; the average grain size is found to decrease. The grain growth mechanism such as diffusion coefficient, sintering temperature and ion concentration, the changes in average grain size depends upon.

3 EDAX studies

Figure 8 shows the EDAX spectra of Mg-Ni-Zn for x = 0.5 ferrite for the sintering temperatures 150^{0}C, 300^{0}C and 450^{0}C. The study of EDAX is done for the confirmation of metals used for the preparation of the sample.

By using EDAX; compositional study of Mg-Ni-Zn ferrite was investigated. According to EDAX data, the precursors used for preparation of sample shows in appropriate amount. No any foreign element was found in the present compound. The elements detected in EDAX data are in good agreement with the elements used for the preparation of the sample. The presence of Mg, Ni, Zn, O and Fe was determined by their corresponding peaks.

Conclusions

By using sol-gel auto combustion method, the $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.5) ferrite sample was successfully prepared. The X-Ray study reveals that the particle size decreases from 37nm to 35nm with increase in sintering temperature from 150^{0}C to 450^{0}C. The lattice constant, ionic radii and bond length increases whereas the x-ray density decreases with increase in sintering temperature, From microstructural study (SEM); it is found that the average grain size decreases from 320 to 210nm with increase in sintering temperature. The EDAX analysis reveals that the presence of metals are in good agreement with the metals used for the preparation of ferrite sample.

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Conflicts of interest: The authors stated that no conflicts of interest.

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