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Synthesis of Mg-Ni-Zn ferrite Nanoparticles by Sol Gel auto combustion route and study of its structural and morphological characteristics

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Abstract

Nanoparticles of Magnesium Nickel Zinc ferrite with general formula $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.3) have been synthesized by using sol gel auto combustion route. The prepared powder was sintered at 150^{0}C, 300^{0}C and 450^{0}C for 1 hour duration. By using different physical and chemical methods, structural and morphological characteristics of prepared sample were studied. The X-ray diffraction method was used for the confirmation of single-phase cubic spinel structure. Also, this method was used to calculate lattice parameter, x-ray density, bond length (A – O and B - O), ionic radii (R_A and R_B) and particle size. As temperature increases from 150^{0}C to 450^{0}C, the lattice constant (a) increases from 8.3117 to 8.3744 A. U and also crystalline size decreases from 38 nm to 34 nm. By using Scanning Electron Microscope (SEM) image, grain size of prepared compound for different sintering temperatures were determined. The average grain size varies from 300nm to 200nm for increasing sintering temperature. Energy Dispersive Analysis of X- ray diffraction reveals the presence of all the metals in the appropriate composition as used in compound preparation.

Keywords: Nanoparticles, x-ray diffraction, SEM, EDAX, crystalline size, lattice constant, bond length, ionic radii, x-ray density.

Introduction

Many researchers have been studied structural, electrical and magnetic behavior of ferrites in the view of their applications within the range of frequency up to few GHz. Ferrites mostly exhibits spinel structure with general formula AB_2O_4 [1] where A cations are present in tetrahedral sites and B cations are present in Octahedral site. Mainly, Ferrites are the mixed metal oxide with iron oxide as main component [2]. 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 [3]. At present, nano-ferrites are most useful material for technological applications and drug delivery [4]. Because of their high electric resistivity and low eddy current loss, ferrite nanoparticles are most important. The ferrites are structure sensitive material [5]. The ferrites containing magnesium and zinc have higher resistivity and it is most useful in high frequency devices [6]. At present, there are several methods which are useful for the preparation of ferrite nanoparticles. Because of its various benefits and easiness in preparation, in the present work, sol-gel auto combustion method is used for the preparation.

Methodology

In the present work, the Mg-Ni-Zn 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 = o.3). The required metals used for the preparation were analytical reagent (A.R) grade nitrates such as magnesium nitrate, nickel nitrate, zinc nitrate and ferric nitrate. The citric acid $C_6H_8O_7$ plays an important role as a fuel in the formation of homogeneous mixture of metal cations. The metal nitrate and fuel ratio is 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. During this process, the solution was kept continuously stirring by using magnetic stirrer. 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 temperature 150^0C, 300^0C and 450^0C for 1 hour.

The general chemical reaction:

$(0.6\text{-}x)\ Mg(NO_3)_2.6H_2O + (x)Ni(NO_3)_2.6H_2O + (0.4)\ Zn(NO_3)_2.6H_2O + 2Fe(NO_3)_3.9H_2O + 3C_6H_8O_7 \rightarrow (Mg_{0.6-x}Ni_x\ Zn_{0.4}\ Fe_2O_4) + 4N_2\uparrow + 18CO_2\uparrow + 36H_2O$

Result and Discussion

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

XRD studies

Figure-1 shows the XRD patterns of composition $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.3). The study of XRD patterns shows that all the prepared samples are single phase spinel cubic structure [7]. 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) [8].

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

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

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

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

Where, u is known as oxygen ion parameter.

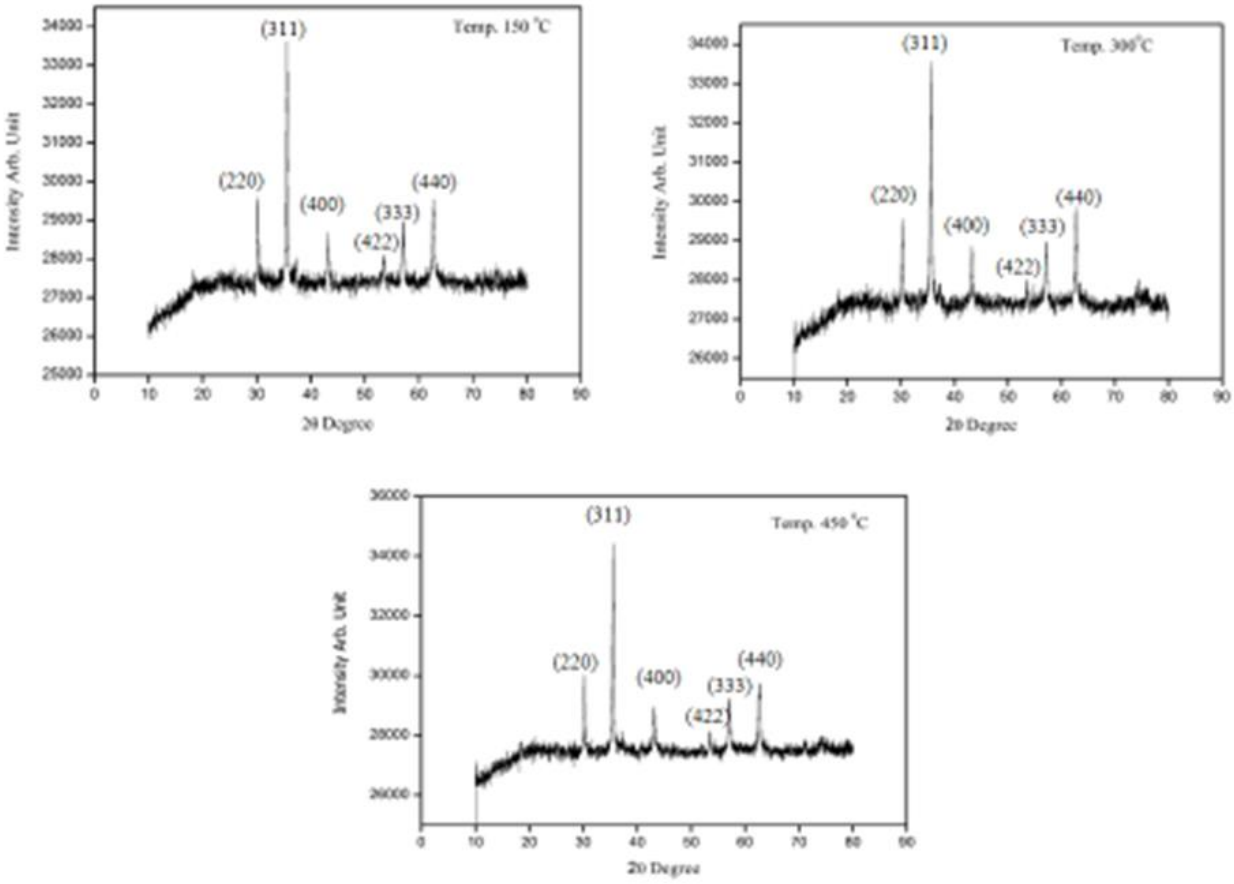

Fig.1: XRD of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.3) for temperatures 150^{0}C, 300^{0}C and 450^{0}C.

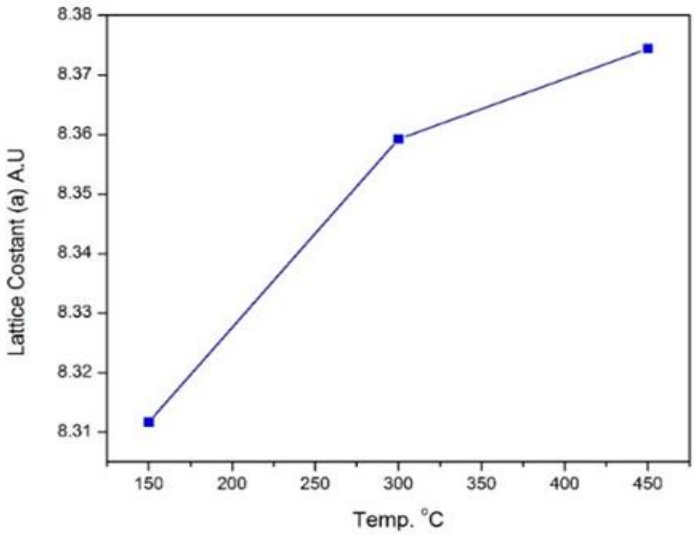

Fig. 2: Lattice Constant (a) Vs Temperature (T)

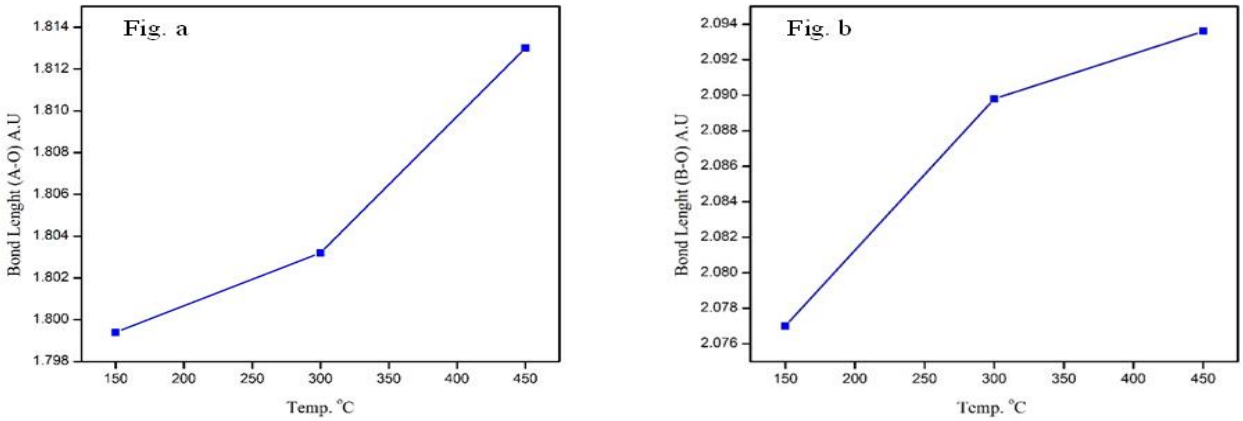

Fig.3: (a) Bond Length (A-O) A.U Vs Temperature (b) Fig. Bond Length (B-O) A.U Vs Temperature

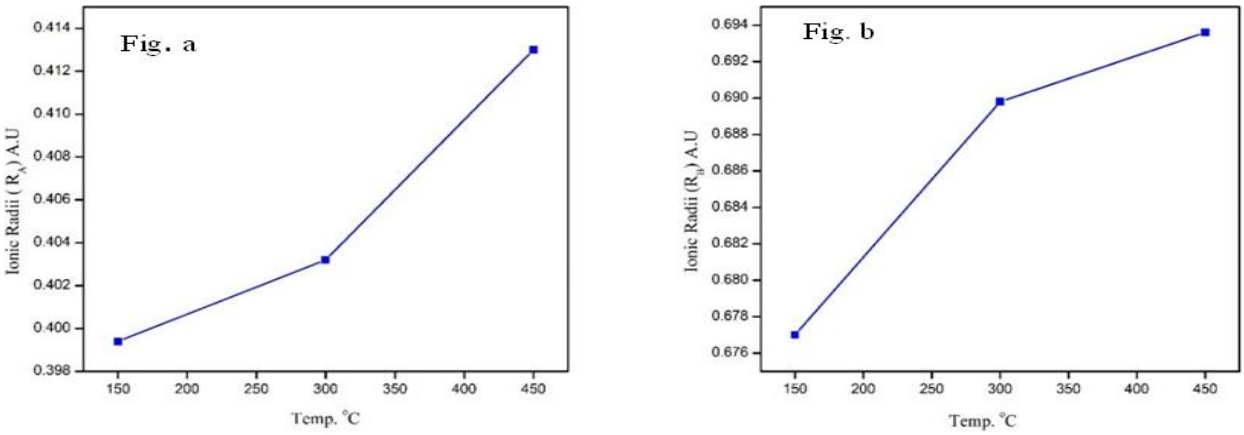

Fig.4: (a) Ionic Radii (R_A) A.U Vs Temperature (b) Ionic Radii (R_B) A.U Vs Temperature

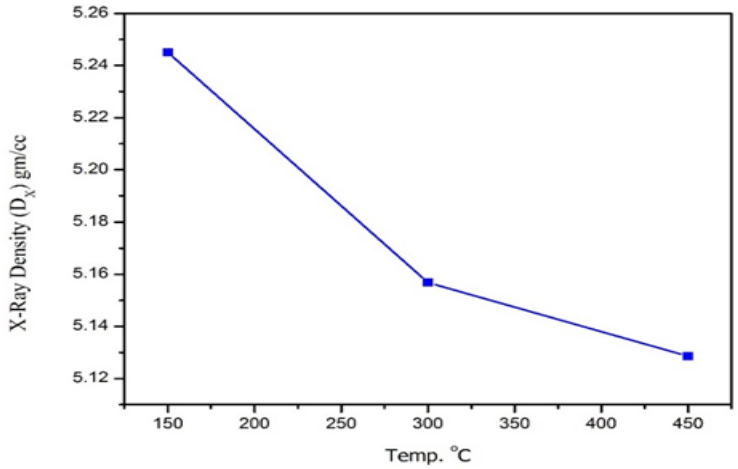

Fig.5: X-Ray Density (D_x) Vs Temperature

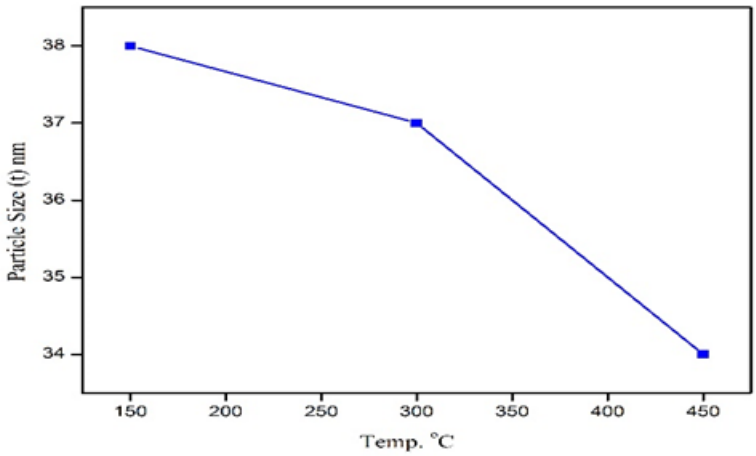

Fig.6: Particle Size (t) nm Vs Temperature

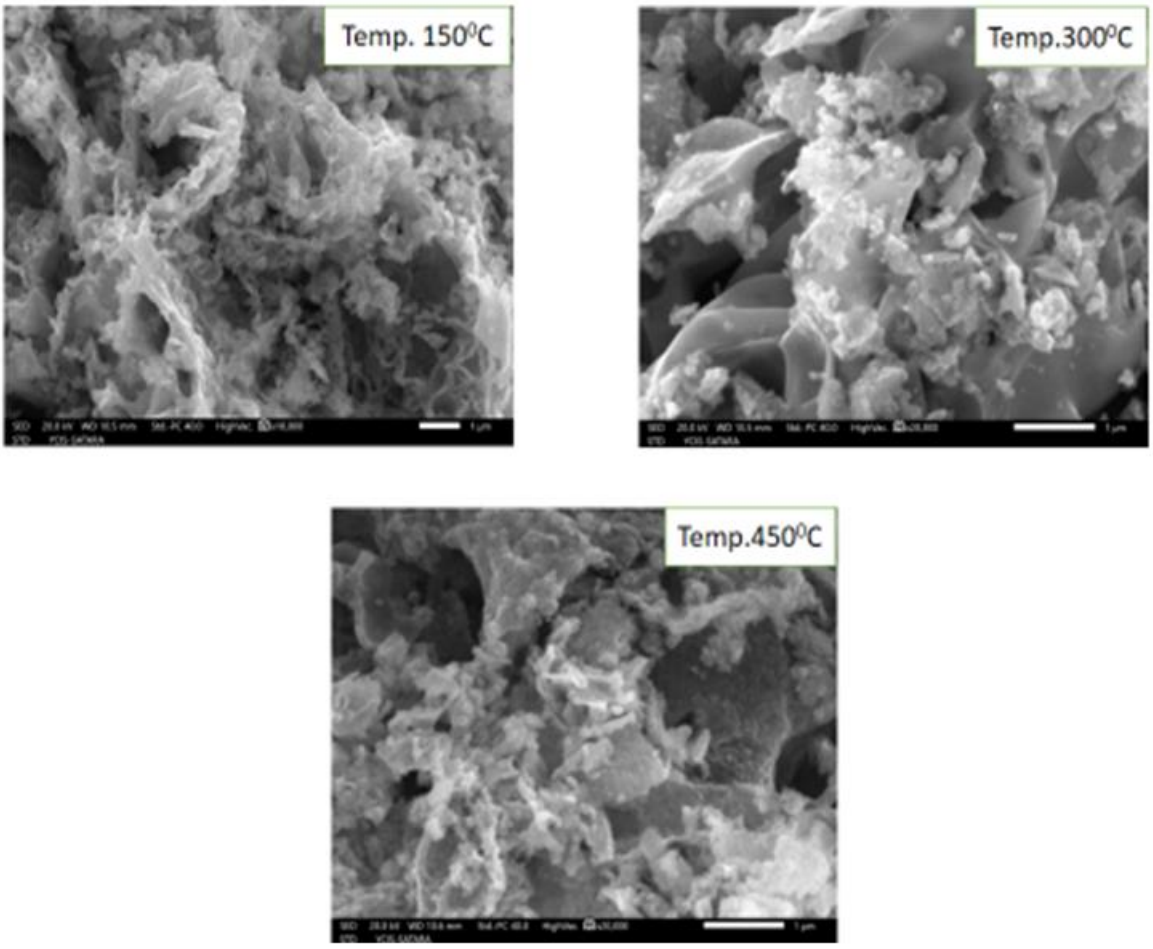

Fig:7 SEM Micrograph of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = 0.3)

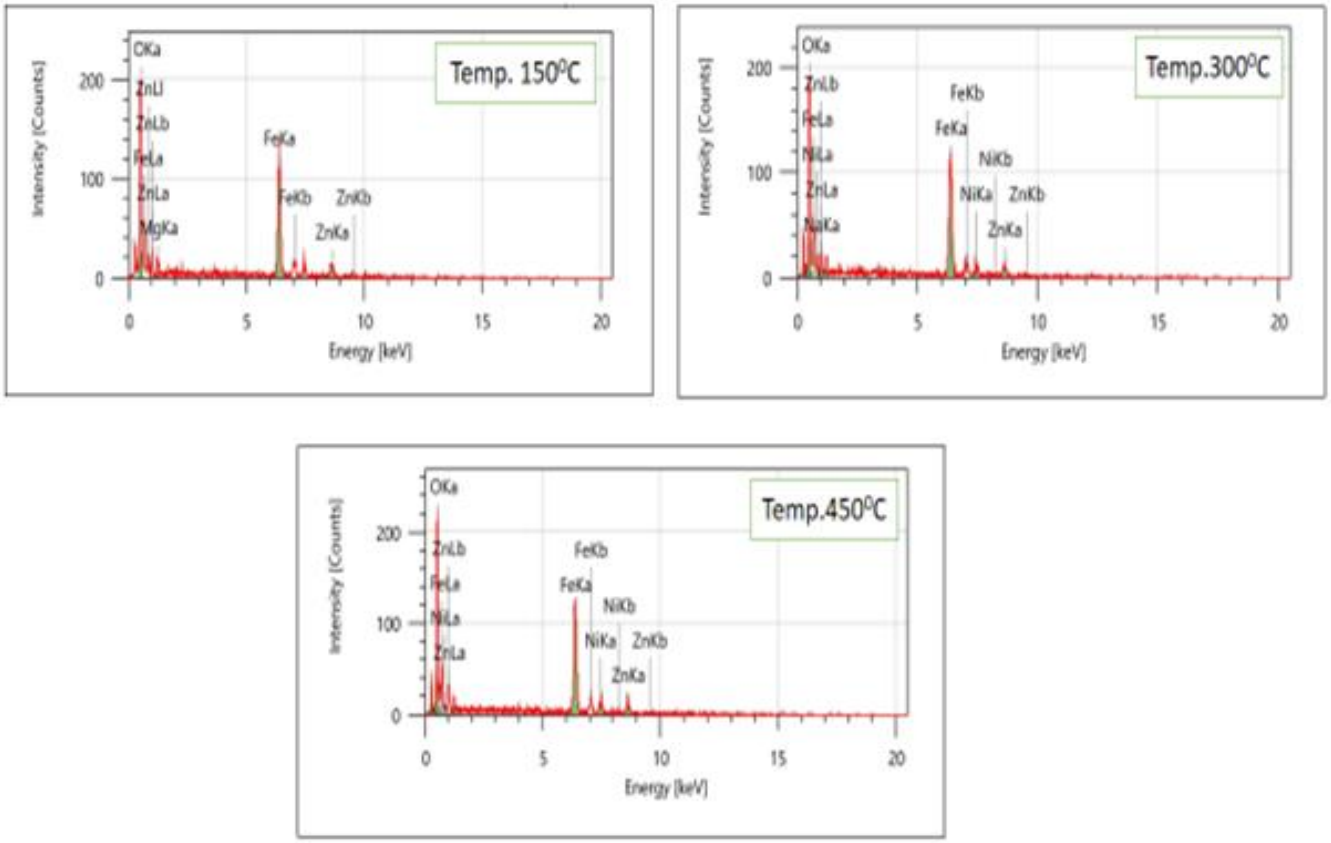

Fig.8: EDAX Pattern of $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.3)

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
0.3	150	8.3117	5.2451	1.7994	2.077	0.3994	0.6770	38
	300	8.3592	5.1568	1.8032	2.0898	0.4032	0.6898	37
	450	8.3744	5.1286	1.8130	2.0936	0.4130	0.6936	34

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)

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

$R_B = (0.625 - u)\ a - r(O_2)^-$ ----------(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. 5 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).

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.

The micrograph of the prepared sample shows that the surface morphology containing full of small grains. It proves that the microstructure is completely formed for the used sintering temperatures. The grain size is determined by drawing various intersecting lines [9]. The average grain size is found to decrease with increase in sintering temperature. The changes in average grain size depends upon the grain growth mechanism such as diffusion coefficient, sintering temperature and ion concentration [10].

EDAX studies

Figure 8 shows the EDAX spectra of Mg-Ni-Zn 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.

Conclusion

The $Mg_{0.6-x}Ni_xZn_{0.4}Fe_2O_4$ (where x = o.3) ferrite sample was successfully prepared by using sol-gel auto combustion method. The X-Ray study reveals that the particle size decreases from 38nm to 34nm with increase in sintering temperature from 150^{0}C to 450^{0}C. Also with increase in sintering temperature, lattice constant, ionic radii and bond length increases whereas the x-ray density decreases. From microstructural study (SEM); it is found that the average grain size decreases from 300nm to 200nm with increase in sintering temperature. The EDAX analysis reveals that the presence of metals is in good agreement with the metals used for the preparation of ferrite sample.

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