



## Size Controlled Synthesis and Magnetic Behaviour of Mg-Zn Nano Ferrites by Using Aloe vera Extract Solution

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Accepted on: 10-11-2014; Finalized on: 30-11-2014.

### ABSTRACT

Mg<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>O<sub>4</sub> (x = 0.65, 0.85) ferrite nanoparticles were prepared by a modified sol-gel method using high purity metal nitrates and aloe vera plant extracted solution. Using of aloe vera extract simplifies the process; provide an alternative process for a simple and economical synthesis of nanocrystalline ferrite. The structural characteristics of calcinated sample of Mg<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>O<sub>4</sub> (x = 0.65, 0.85) ferrite nanoparticles were determined by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) and transmission electron microscopy (TEM). The prepared samples have spinel structure. From XRD we observed that particle size decreases with increasing Mg content. All the prepared samples have spinel structure with particle size of 13 nm-14.7 nm. Nano size of the particles was confirmed by TEM measurement. FTIR spectral analysis helps to confirm the formation of spinel structure in ferrite samples. Magnetization measurements were obtained at room temperature by using vibrating sample magnetometer (VSM), which showed that the calcinated samples exhibited magnetic behavior.

**Keywords:** Aloe-vera, Electron microscopy, Magnetic properties, Sol-gel, Synthesis, Vibrating sample magnetometer.

### INTRODUCTION

Mg-Zn ferrites are soft magnetic material is mostly used as various inductance components, such as magnetic cores of filters, transformers, deflection, antenna, video magnetic heads and magnetic heads of multiple path communication and so on. Furthermore, the material has also brought potential applications in magnetic liquid absorbing materials.<sup>1-7</sup> With rapid development of electronic information industries such as communications and computer networks, the size of electronic apparatus and equipments is miniaturized.<sup>8-9</sup> Demand for electronic components with high density, light weight, thin type and fine performance is greatly increasing, which accelerate the demand for soft magnetic ferrites with high performance and thus contributes to the development of soft magnetic ferrites on the direction of higher frequency and lower power consumption.<sup>10-15</sup> Ferrite particles in nano scales can be produced by soft chemical methods, such as co-precipitation, sol-gel and hydrothermal synthesis.<sup>16-20</sup> Among other established synthesis methods, simple and cost effective routes to synthesize nanocrystalline Mg-Zn ferrite by utilization of cheap, non-toxic and environmentally benign precursors are still the key issue.

In this present work, we report for the synthesis of nanoparticles of Mg-Zn ferrite by simple method using metal nitrates and aloe vera extract solution as a precursors. The samples were characterized by, XRD, FT-IR and TEM. The magnetic properties of prepared nanoparticles were investigated by vibrating sample magnetometer (VSM).

### MATERIALS AND METHODS

#### Materials

All materials were of analytical grade and were used without further purification. Distilled water was used in all experiments.

#### Synthesis of Mg<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>O<sub>4</sub> ferrite nanoparticles

In this study, the Mg<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>O<sub>4</sub> ferrite nanoparticles were synthesized by the modified sol-gel method. In this study either Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O or Mg(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O mixed with Fe(NO<sub>3</sub>)<sub>2</sub>·9H<sub>2</sub>O were used as the starting materials. In a typical procedure, 60 ml of aloe vera plant extract, instead of toxic organic polymers, was mixed with 40 ml distilled water under vigorous stir until homogenous solution was obtained. According to this formula Mg<sub>x</sub>Zn<sub>1-x</sub>Fe<sub>2</sub>O<sub>4</sub> (x = 0.65, 0.85), each metal nitrate was added slowly to the aloe vera solution under vigorous stirring for 2 h to obtain a well – dissolved solution. No pH adjustment was made. Then the mixed solution was evaporated by heating on the hot plate at 100°C under vigorous stirring for several hours until a dried precursor was obtained. The dried precursor was crushed into powder using mortar and pestle. The dried precursor then was calcinated in a muffle – furnace at 700°C for 2 hours.

#### Particle characterization

The X-ray diffraction (XRD) patterns of the samples were recorded on a PANalytical X'Pert PRO X-ray diffractometer using Cu Kα radiation (λ = 0.15406 Å). The crystallite size of nanocrystalline samples was measured from the line broadening analyses using Debey-Scherrer



formula after accounting for instrumental broadening (Equation 1):

$$D_{XRD} = 0.89 \lambda / \beta \cos \theta \dots\dots\dots(1)$$

Where  $\lambda$  – wavelength of X-ray radiation used in Å,  $\theta$  is the diffraction angle,  $\beta$  is the full width at half maximum (FWHM) in radians in the  $2\theta$  scale,  $D_{XRD}$  is the crystallite size in nm.<sup>21</sup>

### Particle Morphology

The particle morphology was examined by transmission electron microscopy (HITACHI model, H-7500). For the TEM observations, powders were supported on carbon-coated copper grids which were ultrasonically dispersed in ethanol.

### Magnetic measurements

Room temperature magnetic measurements were carried out using a Lakeshore vibrating sample magnetometer (VSM) and parameters like specific saturation magnetization ( $M_s$ ), coercive force ( $H_c$ ) and remanence ( $M_r$ ) were evaluated.

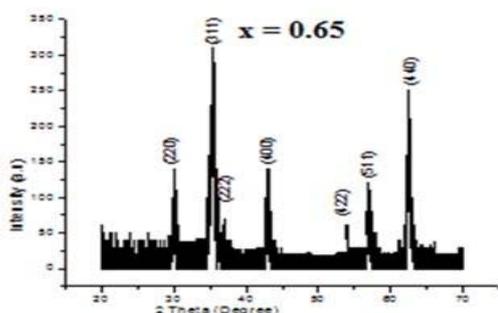
### Spectral measurements

FTIR spectra were recorded for dried samples of  $Mg_xZn_{1-x}Fe_2O_4$  ( $x = 0.65, 0.85$ ) with an Perkin – Elmer FTIR spectrometer. The dried samples were in KBr matrix, and spectra were measured according to transmittance method.

## RESULTS AND DISCUSSION

### XRD Analysis

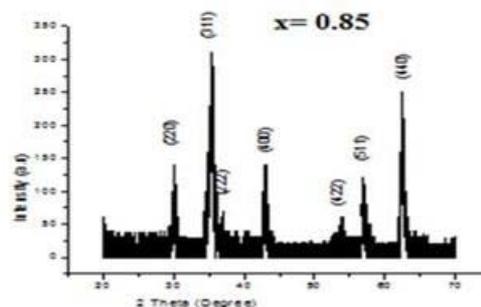
XRD can be used to characterize the crystallinity of nanoparticles and it gives the average diameters of all the nanoparticles. The fine particles were characterized by XRD for structural determination and estimation of crystallite size. XRD pattern were analyzed. All experimental peaks were matched with theoretically generated one and indexed. The XRD patterns of all the samples were shown in Figure 1.



**Figure 1a:** XRD patterns of  $Mg_{0.65}Zn_{0.35}Fe_2O_4$  ferrites nanoparticles calcinated at  $700^\circ C$  for 2 hours.

It shows the formation of spinel ferrite phase in all the samples. The broad XRD line indicates that the ferrite particles are in nano size. The crystallite size for each composition are calculated from XRD line width of the

(311) peak using Scherrer's formula.<sup>22</sup> The average crystallite size decreases from 14.7 nm to 13.0 nm when the partial substitution of Mg increases ( $x = 0.65$  to  $x = 0.85$ ). Such a decrease in the value of the particle size for series is expected because replacement of  $Zn^{2+}$  ion of larger radius, by  $Mg^{2+}$  ions of smaller size. The values of the particle size, lattice constant as deduced from X-ray data are given by Table 1. The lattice constant was found to decreases from 8.369 to 8.260 Å with increase in Mg concentration as shown in Table 1. This is due to the fact that replacement of larger  $Zn^{2+}$  cation of larger radius, by smaller  $Mg^{2+}$  cations, causing shrinkage in the unit cell dimensions of the spinel lattice. Therefore as the concentration of  $Mg^{2+}$  ions increases, the lattice constant decreases.



**Figure 1b:** XRD patterns of  $Mg_{0.85}Zn_{0.15}Fe_2O_4$  ferrites nanoparticles calcinated at  $700^\circ C$  for 2hours

**Table 1:** Crystallite size, Lattice constant and unit cell volume for  $Mg_xZn_{1-x}Fe_2O_4$  ( $x = 0.65, 0.85$ ) nanoferrites calcinated at  $700^\circ C$  for 2h

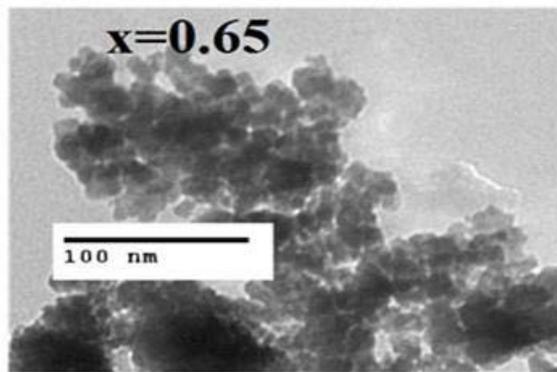
Mg Concentration (x)	Crystallite Size (D) (nm)	Lattice constant (Å)	Unit Cell Volume $a^3$ (Å <sup>3</sup> )
0.65	14.7	8.369	586.166
0.85	13.0	8.260	567.663

The strongest reflection comes from the (311) plane. This denotes the spinel phase. All the compositions had a spinel structure. The peaks indexed to (200), (311), (400), (422), (511) and (440) planes of a cubic unit cell, corresponds to cubic spinel structure. The calculated lattice constant (Å), identified the sample to be cubic spinel.

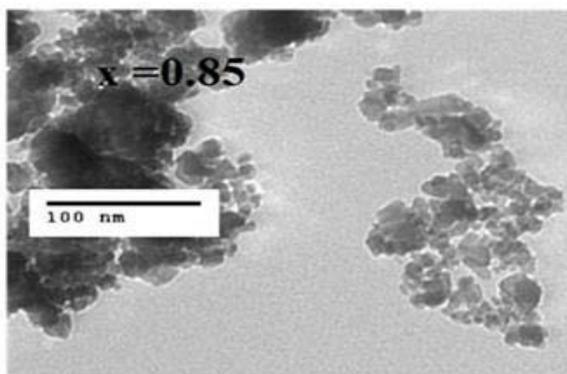
### Transmission electron microscopy

The morphology and structure of the prepared ferrite samples calcinated at  $700^\circ C$  were investigated by TEM techniques as shown in Figure 2. The results indicate that the samples prepared by sol-gel method are almost uniform in both morphology and particle size distribution. A close inspection would reveal the presence of particles showing the spherical in shape. The particle sizes decreased with increasing Mg concentration. Mean particle size from TEM image is in good agreement with the crystallite size measured from X-ray line (311) broadening using scherrer's formula. This is lower than

the particle size of nanoferrites prepared by other chemical method.



**Figure 2a:** TEM images of  $Mg_{0.65}Zn_{0.35}Fe_2O_4$  ferrites nanoparticles calcinated at  $700^\circ C$  for 2 hours.

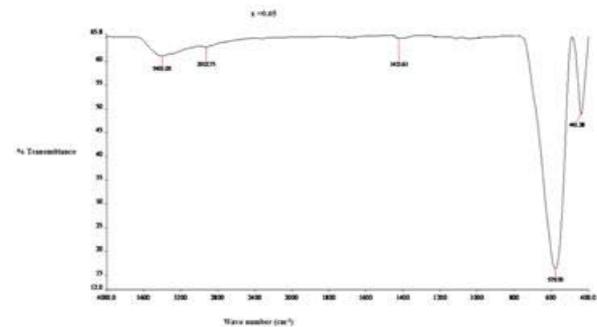


**Figure 2b:** TEM images of  $Mg_{0.85}Zn_{0.15}Fe_2O_4$  ferrites nanoparticles calcinated at  $700^\circ C$  for 2 hours.

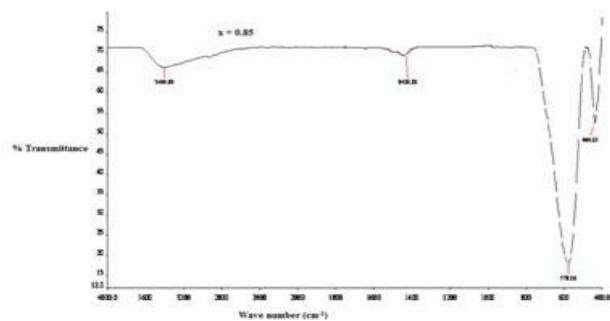
#### Fourier transform infrared analysis (FT-IR) measurements

FTIR spectral analysis helps to confirm the formation of spinel structure in ferrite samples. The FTIR spectra of the investigated  $Mg_xZn_{1-x}Fe_2O_4$  ( $x = 0.65, 0.85$ ) samples are shown in figure 3. In the wave number range of  $1000-300\text{ cm}^{-1}$ , two main broad metal-oxygen bands are seen in the infrared spectra of all spinels, especially ferrites. The higher one ( $\nu_1$ ) generally observed in the range  $600-550\text{ cm}^{-1}$ , is caused by the stretching vibrations of the tetrahedral metal-oxygen bond. The lowest band ( $\nu_2$ ) usually observed in the range  $450-440\text{ cm}^{-1}$ , is caused by the metal-oxygen vibrations in the octahedral sites (Waldron 1955). This difference in the spectral positions is expected because of the difference in the  $Fe^{3+}-O^2-$  distance for the octahedral and tetrahedral compounds. This is confirmed from fourier transform Infrared spectroscopy (FTIR) that the structure remains cubic spinel after magnesium substitution in zinc nano ferrite.<sup>22-24</sup> The vibrational frequencies of IR bands  $\nu_1$  and  $\nu_2$  of samples prepared by sol-gel are shown in figure 3(a) and figure 3(b). The spectra show prominent bands near  $3400$  and  $1600\text{ cm}^{-1}$ , which are attributed to the stretching modes and H-O-H bending vibrations of the free or absorbed water. The peaks are around  $3429.27\text{ cm}^{-1}$ ,  $2921\text{ cm}^{-1}$ , corresponding to the stretching and bending

vibration of O-H, C-H. The band near  $1400\text{ cm}^{-1}$  is due to the antisymmetric NO-stretching vibrations arising from the nitrate group which is present as residue in the samples (Priyadharsini et al 2009). This band is very weak in the spectra of sol-gel derived sample, indicating the purity of ferrite nanoparticles synthesized by this method.



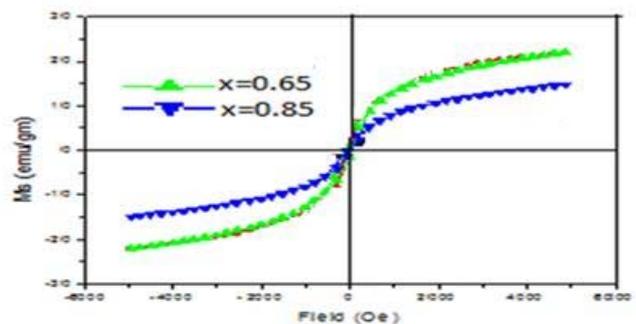
**Figure 3a:** FTIR spectrographs of  $Mg_{0.65}Zn_{0.35}Fe_2O_4$  ferrites nanoparticles calcinated at  $700^\circ C$  for 2 hours



**Figure 3b:** FTIR spectrographs of  $Mg_{0.85}Zn_{0.15}Fe_2O_4$  ferrites nanoparticles calcinated at  $700^\circ C$  for 2 hours

#### Magnetic measurements

From the hysteresis curve one can get information such as saturation magnetization ( $M_s$ ), coercivity ( $H_c$ ), remanance magnetization ( $M_r$ ) and squareness ratio ( $M_r/M_s$ ) for a given sample. The magnetic properties of the  $Mg_xZn_{1-x}Fe_2O_4$  ( $x=0.65, 0.85$ ) nanoferrites powder sample can be determined at room temperature using vibrating sample magnetometer (VSM) with applied field up to  $6000\text{ Oe}$ . Figure 4 shows the variation of magnetization with applied field for all  $Mg_xZn_{1-x}Fe_2O_4$  ( $x=0.65, 0.85$ ) nanoferrites.



**Figure 4:** Variation of magnetization with applied field at room temperature for  $Mg_{0.65}Zn_{0.35}Fe_2O_4$  and  $Mg_{0.85}Zn_{0.15}Fe_2O_4$  ferrites nanoparticles

The hysteresis curve (Figure 4) recorded at room temperature shows very low coercivity, remanence at room temperature. Nanoferrites do not attain saturation in applied field.<sup>25-29</sup> Calculated values of Saturation Magnetization (Ms), Coercivity (Hc), Remanent

Magnetization (Mr) and Squareness ratio for  $Mg_xZn_{1-x}Fe_2O_4$  ( $x=0.65, 0.85$ ) nanoferrites at 700°C for 2 hours as shown in table 2. The reduced values of the Mr/Ms ratio show a powder ferrite behavior, within the magnetization field, closer to the super paramagnetic one.

**Table 2:** Calculated values of Saturation Magnetization (Ms), Coercivity (Hc), Remanent Magnetization (Mr) and Squareness ratio for  $Mg_xZn_{1-x}Fe_2O_4$  ( $x=0.65, 0.85$ ) nanoferrites at 700°C for 2 hours.

Mg concentration (x)	Magnetic Properties			
	Saturation Magnetization (Ms)(emu/gm)	Coercivity(Hc) (Oe)	Remanent Magnetization (Mr)(emu/gm)	Squareness Ratio(R= Mr/Ms)
0.65	22.0	93.29	2.47	0.11
0.85	14.8	61.30	1.44	0.09

## CONCLUSION

Nanocrystalline  $Mg_xZn_{1-x}Fe_2O_4$  ferrites with varying x were synthesized by a simple solution route using high purity nitrates and aloe vera plant extract solution. Form XRD, FT-IR spectra and TEM analysis, it is indicated that the crystalline spinel ferrite can be obtained using calcination temperature at 700°C for 2 hours. XRD pattern confirms the synthesis of fully crystalline single phase Mg-Zn nano ferrites. The particle size size of nanocrystalline spinel ferrite calculated from FWHM of XRD (311) peak and in good agreement with TEM result. The room temperature M-H hysteresis curve shows that the particles are super paramagnetic at room temperature. This work demonstrates the use of a simple synthetic method using cheap precursors of Aloe vera plant extract provides high – yield nanosized ferrites with well crystalline structure and uniform particle sizes, energy saving, high purity, no reaction with containers which increases purity, no pH adjustment, environmental friendly, and acceptable magnetic properties.

**Acknowledgements:** Author is grateful to SAIF, Punjab University, Chandigarh for characterization (XRD, TEM and FTIR) and to National Physical Laboratory for VSM measurement.

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**Source of Support: Nil, Conflict of Interest: None.**

