

## Ionizing Radiation Induced Structural Modification in Nanoferrites

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Metal doped ferrites have wide applications in various areas such as environment, medical, agriculture, electronics, industry and so on. In this study, magnesium doped zinc ferrites ( $Mg_xZn_{1-x}Fe_2O_4$ ) were prepared by sol-gel auto-combustion method. They were characterized by X-ray Diffraction (XRD) and Fourier-Transform Infra-red Spectroscopy (FTIR) techniques respectively. The doped ferrite was synthesized in two different reaction media viz, double distilled water and aloe vera gel.  $Mg_{0.5}Zn_{0.5}Fe_2O_4$  was prepared using standard methods of synthesis. Both the synthesized doped ferrites were gamma irradiated to 50 kGy and 300 kGy doses and were again characterized using XRD and FTIR techniques to study the changes in structural properties of the ferrites. The effect of two different doses of gamma radiation on ferrites is compared using XRD and IR spectra. The post irradiation structural changes were investigated in the doped ferrites.

**Keywords:** FTIR, Gamma irradiation, Magnesium doped zinc ferrite, XRD

### 1 Introduction

Ferrites are magnetic mixed metal oxides in which iron oxide is major unit and spinel ferrites are represented as  $MFe_2O_4$  where M can be divalent metal ions<sup>1,2</sup>.  $Mg_{0.5}Zn_{0.5}Fe_2O_4$  is a combination of magnesium and zinc ferrite. Generally, zinc ferrite possesses a spinel structure in which  $Zn^{2+}$  ions occupy the tetrahedral sites whereas the magnesium ferrite possesses an inverse spinel structure in which  $Mg^{2+}$  ions occupy octahedral sites. It has been reported that Mg-Zn ferrites have applications in biomedical science like hyperthermia, memory devices, magnetic fluid and in electronic appliances<sup>3-5</sup>. Mg-Zn ferrites are inexpensive soft ferrites having low coercive property, electrical resistivity and high curie temperature<sup>6,7</sup>. Pendyala et.al studied the effect of magnesium doping in Zn ferrite synthesized by sol gel auto combustion method and concluded that magnesium doped zinc ferrite is spherical in shape and owns cubic spinel. Due to incorporation of magnesium ions in zinc ferrite, ionic conductivity and magnetization increases while lattice parameters, average crystal size and grain size decreases<sup>8</sup>. Manikandan et. al used aloe vera plant extract for green synthesis of co ferrite and studied its catalytic

property. Aloe vera gel have pH 4.4-4.7 and consists of 99% to 99.5% water, polysaccharides, minerals, enzymes, fat-soluble vitamins, organic acids and phenolic compounds. Aloe vera gel is used for nanomaterial synthesis as it is environment friendly reaction media, good reducing and gelling agent<sup>9,10</sup>. Angadi et. al depicted that gamma radiation affects the structural, electrical and magnetic properties of ferrites because after absorption of high dose gamma radiation such as 50kGy the some crystal defects arises in the structure due to vibrational and electronic excitation, ionization of atoms and scattering of particles<sup>11,12</sup>.

In the present study, magnesium doped zinc ferrite ( $Mg_{0.5}Zn_{0.5}Fe_2O_4$ ) is prepared in two ways i.e in first case sol-gel auto-combustion route is followed using double distilled water as reaction medium and secondly, Aloe vera gel is used as a reaction medium instead of double distilled water. This can be called as green sol-gel route of synthesis. Both the samples were gamma irradiated by the Cobalt-60 source with the two different doses i.e 50 kGy and 300 kGy. Two widely different doses were deliberately chosen to compare the effect of radiation on the ferrites. X-ray diffraction (XRD) pattern and Fourier-Transform Infra-red spectrometry (FTIR) studies of both un-irradiated and irradiated samples were carried out to

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observe the structural differences after irradiation with gamma rays.

## 2 Materials & Method

### 2.1 Synthesis

Iron nitrate [ $\text{Fe}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$ ] was procured from S. D. Fine chemicals, urea [ $\text{CO}(\text{NH}_2)_2$ ], zinc nitrate [ $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ] and magnesium nitrate [ $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ] were procured from Loba Chemie Analytical Grade Reagents and Fine chemicals. Double distilled water and aloe vera gel from freshly cut aloe vera leaves were used as reaction media without any further purification.

$\text{Mg}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  was prepared by using stoichiometric amount of oxidizers i.e iron nitrate, zinc nitrate and magnesium nitrate and fuel as a reducing agent i.e urea (nitrate: urea was maintained as 1:2) by sol gel auto combustion method as reported in literature<sup>8-10</sup>. Aloe vera gel was extracted from freshly cut aloe vera leaves. They were washed to clean, then outer green leaf part was removed and gel was extracted. The gel was ground in mixer grinder to liquify all the solid contents in a gel. Reaction media in both the cases were maintained at 100 mL to dissolve the nitrates and urea (fuel).

It was observed that auto-combustion reaction time decreased in synthesis using aloe vera gel as compared to double distilled water. This is attributed to the presence of phytochemicals present in the aloe vera gel which catalyses the reaction.  $\text{Mg}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  using double distilled water as a reaction media is abbreviated as MgZnF-W and other one using aloe vera gel is named as MgZnF-AG.

### 2.2 Gamma irradiation

Both the samples MgZnF-W and MgZnF-AG were gamma irradiated with the exposure dose of 50 kGy and 300 kGy at room temperature in the Cobalt-60 Gamma Chamber (GC-1200) housed at Department of Chemistry, Rashtrasant Tukadoji Maharaj Nagpur University, Nagpur. The gamma irradiated MgZnF-W and MgZnF-AG samples named as MgZnF-W-50kGy, MgZnF-AG-50kGy, MgZnF-W-300kGy and MgZnF-AG-300kGy for samples with 50 kGy and 300 kGy doses respectively.

### 2.3 Characterization

Characterization was carried out using instruments X-ray diffractometer at RUSA Department and

Brucker FT-IR Spectrometer at Department of Chemistry, Rashtrasant Tukadoji Maharaj Nagpur University, Nagpur. Both the samples MgZnF-W and MgZnF-AG were characterized before and after gamma irradiation. The obtained XRD and FT-IR spectra are given below and structural changes after gamma irradiation are discussed.

## 3 Result and Discussion

XRD spectra of unirradiated and  $\gamma$ -irradiated  $\text{Mg}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  are given in Fig. 1.

### 3.1 XRD analysis

XRD analysis was done by using Cu-K $\alpha$  radiations to calculate the average crystallite size and to observe the phase of the particle. Nadeem et. al. reported that  $\text{Mg}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  has cubic spinel structure and phase free of impurity.<sup>[14]</sup> The spinel structure is evident from the diffraction peaks (220), (311), (400), (422), (511), (440) in the scanning range 20° to 70°. Crystallite size was calculated using Debye Scherrer formula using peak of maximum intensity at XRD

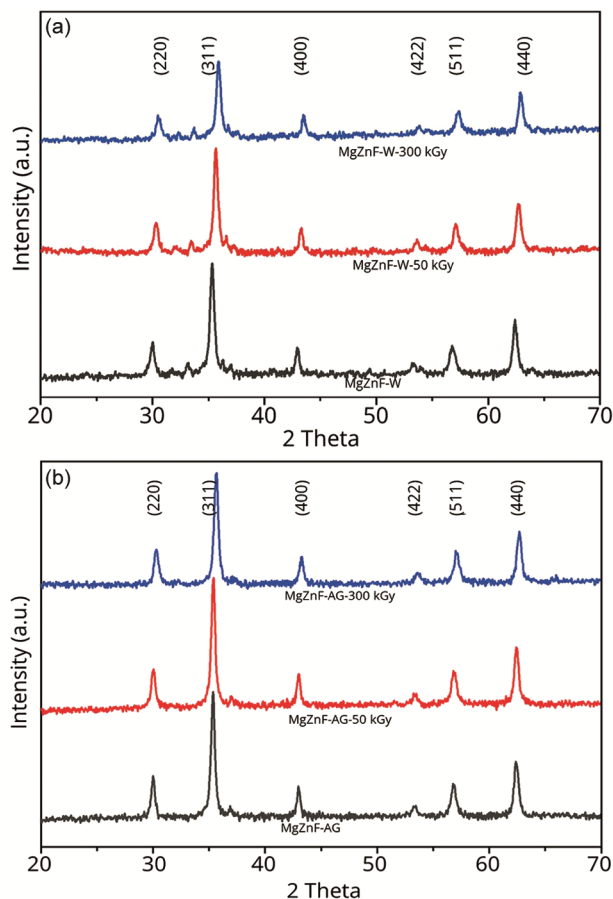


Fig. 1 — XRD spectra of unirradiated and  $\gamma$ -irradiated (a) MgZnF-W, & (b) MgZnF-AG.

Table 1 — Crystallite size determination of all synthesized and  $\gamma$  irradiated  $Mg_{0.5}Zn_{0.5}Fe_2O_4$  ferrites

SN	Ferrites $Mg_{0.5}Zn_{0.5}Fe_2O_4$	FWHM ( $\beta$ ) (in degrees)	FWHM ( $\beta$ ) (in radian)	$2\theta$ (degrees)	$\theta$ (degrees)	$\theta$ (radian)	(D)crystallite size in nm
1	MgZnF-W	0.419	0.007313	35.293	17.6465	0.3079	17.87
2	MgZnF-W-50kGy	0.43	0.007505	35.637	17.8185	0.3109	17.39
3	MgZnF-W-300kGy	0.433	0.007557	35.889	17.9445	0.3132	17.26
4	MgZnF-AG	0.385	0.006719	35.334	17.6670	0.3083	19.44
5	MgZnF-AG-50kGy	0.371	0.006475	35.379	17.6895	0.3087	20.17
6	MgZnF-AG-300kGy	0.435	0.007592	35.632	17.8160	0.3109	17.19

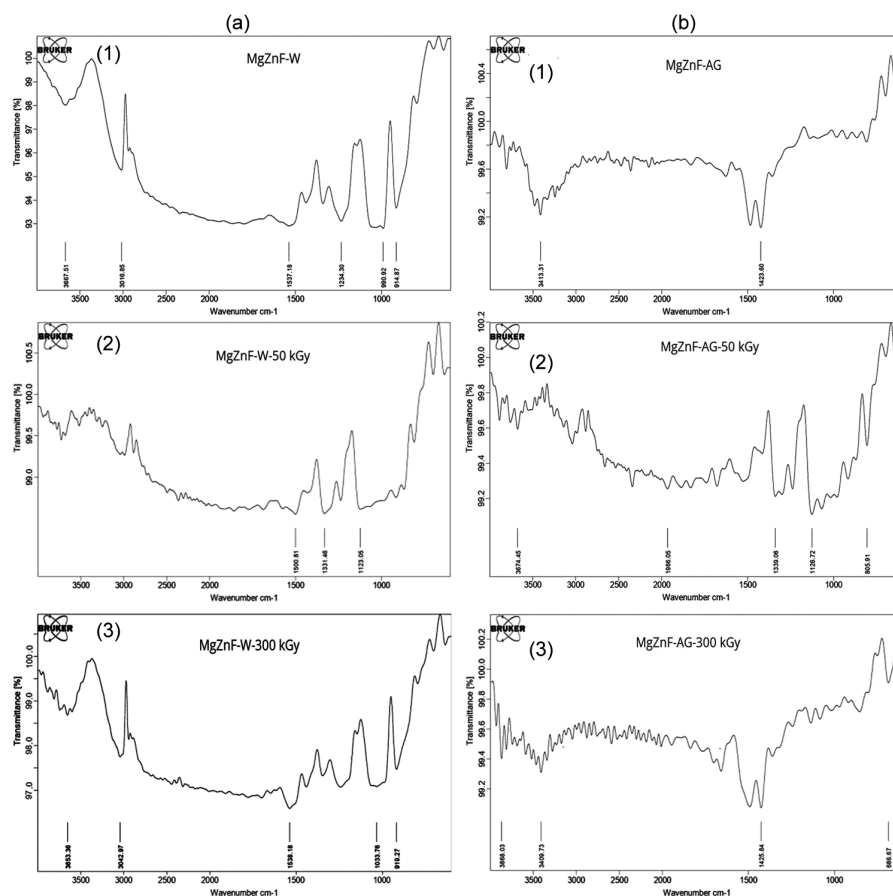


Fig. 2 — FT-IR spectra of unirradiated and  $\gamma$ -irradiated (a) MgZnF-W, & (b) MgZnF-AG.

graph.(Table no. 1)<sup>12</sup>. Formula is given by the equation,  $D = 0.89\lambda/\beta\cos\theta$  where  $\lambda$ =wavelength of X ray,  $\beta$ =FWHM,  $\theta$ =Bragg's diffraction angle and D-crystallite size in nm<sup>15</sup>

The decrease in crystallite size, albeit marginal is observed with increase in gamma dose. This is being investigated further. Moreover, there is broadening of peaks (422) and (511) with increasing dose. It probably is indicative of lattice compression. This might arise due to radiation induced defects. The compression of lattice might lead to mechanical strain within the crystal lattice and thereby affecting the property of the material.

### 3.2 FTIR spectra

FT-IR spectra shows different vibrational bands for ions residing at tetrahedral and octahedral sites<sup>3</sup>. It provides insight into intrinsic vibrations of the metal ions. The bands below  $1000\text{cm}^{-1}$  is indicative of intrinsic vibration at tetrahedral and stretching vibrations at octahedral sites between metal (M) and oxygen (O) ions<sup>3</sup>. The changes are observed in said range of IR spectra, is indicative of changes in M-O bonds, which could be attributed to the radiation induced defect. The same is reflected by the change in the peak position as shown in Fig 2 (a) and (b) for MgZnF-W and MgZnF-AG respectively.

#### 4 Conclusion

The XRD study depicts marginal decrease in crystallite size and broadening of peaks (422) and (511) with increasing dose. The phenomenon was evident in both the samples. The peak broadening is indicative of lattice compression indicating radiation induced defects. This was further corroborated through the changes in vibrational bands (below 1000  $\text{cm}^{-1}$ ) with increasing dose. The changes in vibrational bands for both the tetrahedral and octahedral sites indicates defect arising due to M-O bond. The ferrites irrespective of the modes of synthesis depicted similar behavior on irradiation. However, the ease of synthesis is enhanced using aloe vera gel as reaction medium, which can be ascribed to catalyzing effect of phytochemicals. This would be taken up as a separate study in future.

Thus it can be concluded that the studies indicate chances towards significant changes in property of the ferrites due to gamma radiations. The change in crystallite structure may trigger many other interesting related properties. However, further investigations are required to conclusively ascertain its applicability in various domains.

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