



## Synthesis and characterization of Mn ferrite nano crystalline by sol - gel method

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### Abstract:

**Nanocrystalline** ferrite samples having the chemical formula  $(\text{Mn}_{1-x}\text{Cu}_x\text{Fe}_{1.7}\text{Ga}_{0.3}\text{O}_4)$ , where  $(x = 0, 0.25, 0.5, 0.75 \text{ and } 1)$  was prepared by sol-gel method. The XRD pattern confirmed the formation of cubic spinel structure of ferrite. Only for the composition  $(\text{Cu}_{1-x}\text{Mn}_x\text{Fe}_{1.7}\text{Ga}_{0.3}\text{O}_4)$ , impurity peaks were observed. Also the infra red spectra were recorded at the room temperature using FT-IR for all samples in the range  $(4000 \text{ cm}^{-1} \text{ to } 400 \text{ cm}^{-1})$ . The FTIR spectrum exhibited  $\nu_1$  and  $\nu_2$  fundamental bands, corresponding to octahedral and tetrahedral sites of the ferrite structure.

### 1. Introduction:

Ferrites are a very important group of magnetic materials due to their extensive use in a wide range of applications, such as ferrofluids, radar absorbing coatings, waveguides in the gigahertz region, biomedical and clinical devices, magnetic resonance imaging, oxyfuel technology,

gas sensors, for the fabrication of magnetic cores of read/write heads for high-speed digital tapes or for disc recording [1-5].

Research worker stated that, Spinel ferrites are a large important class of materials with the general formula  $MFe_2O_4$ , where M is a divalent cation. Manganese ferrite,  $MnFe_2O_4$ , is an inverse spinel in which the tetrahedral sites (A) are occupied by  $Fe^{2+}$  and  $Mn^{2+}$  ions [6, 7].

Nano crystalline spinel ferrites with general formula  $MFe_2O_4$  (M = Ni, Co, Cu, Zn, Mn, etc.) have face centered-cubic structure. These materials have a cubical spinel structure where oxygen forms a face centered cubic close packing and  $M^{2+}$  occupy either tetrahedral or octahedral interstitial sites. Ferrites with uniform particle size and narrow size distribution are desirable for variety of applications like gas sensing applications, high density data storage, ferro-fluid technology, magneto caloric refrigeration, magnetic guided drug delivery and electronic devices for high frequency applications [8]. Also the study of FT-IR spectra is very important tool for getting the ions positions in the lattice.

In the present work,  $(Mn_{1-x}Cu_xFe_{1.7}Ga_{0.3}O_4)$  spinel ferrite was prepared by sol-gel method. Consequently the products are characterized by FTIR spectroscopy, and X-ray diffraction (XRD).

## 2. Experimental:

$(Mn_{1-x}Cu_xFe_{1.7}Ga_{0.3}O_4)$ , where ( $x = 0, 0.25, 0.5, 0.75$  and 1) nanocrystalline samples were synthesized using sol gel method [8]. We used Stoichiometric amounts of iron, manganese, gallium, copper nitrates, (purity 99.99%), and citric acid using distilled water as a solvent. The solution was heated under stirring to evaporate the solvent water, then heated at 400 °C for 5 hours and then cooled to room temperature. The synthesized samples were characterized for crystal phase identification by

X-Ray at room temperature using monochromatic  $\text{CuK}\alpha$  radiation with  $\lambda=1.54056 \text{ \AA}$ . The samples were scanned in continuous normal scan mode from  $10$  to  $80^\circ$ . The diffraction peaks were fitted by modified Gaussian functions. Fourier Transform IR spectrum for the powder sample was recorded in the frequency range from ( $400 \text{ cm}^{-1}$  to  $4000 \text{ cm}^{-1}$ ) at room temperature using Bruker IFS 66V FTIR spectrometer.

### 3. Results and discussion:

#### 3.1 XRD measurements:

Fig.1. Shows samples which prepared using sol-gel method (Nano structure). The diffraction peaks are fitted by modified Gaussian functions. The result of XRD patterns shows that the nominal composition structures with different concentration are single phase with no additional lines corresponding to any other phases.

All the samples have the cubic spinel structure. Only for  $\text{Cu}_{1-x}\text{Mn}_x\text{Fe}_{1.7}\text{Ga}_{0.3}\text{O}_4$ , impurity peaks were observed. The results assured that all samples can be crystallized in cubic phase at low temperature in nano structure phase using different preparation methods as sol- gel method.

The substitution of  $\text{Mn}^{3+}$  in  $\text{CuFe}_2\text{O}_4$ , results in cubic structure. Although the Jahn–Teller cations  $\text{Cu}^{2+}$  and  $\text{Mn}^{3+}$  were present in all samples,  $\text{Mn}_{1-x}\text{Cu}_x\text{Fe}_{1.7}\text{Ga}_{0.3}\text{MnO}_4$  was single phase having spinel structure [9 ].

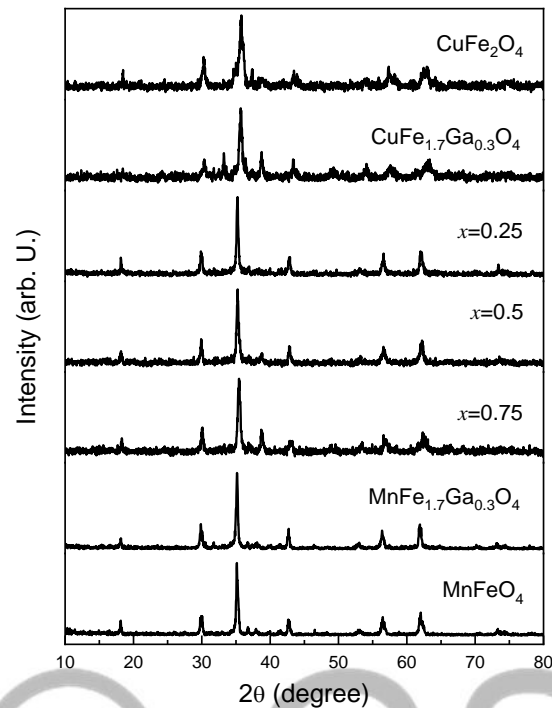


Fig.1. X-ray powder diffraction patterns of Nano  $Mn_{1-x}Cu_xFe_{1.7}Ga_{0.3}O_4$ ,  $CuFe_2O_4$  and  $MnFe_2O_4$ .

### 3.2. Magnetic properties measurements:

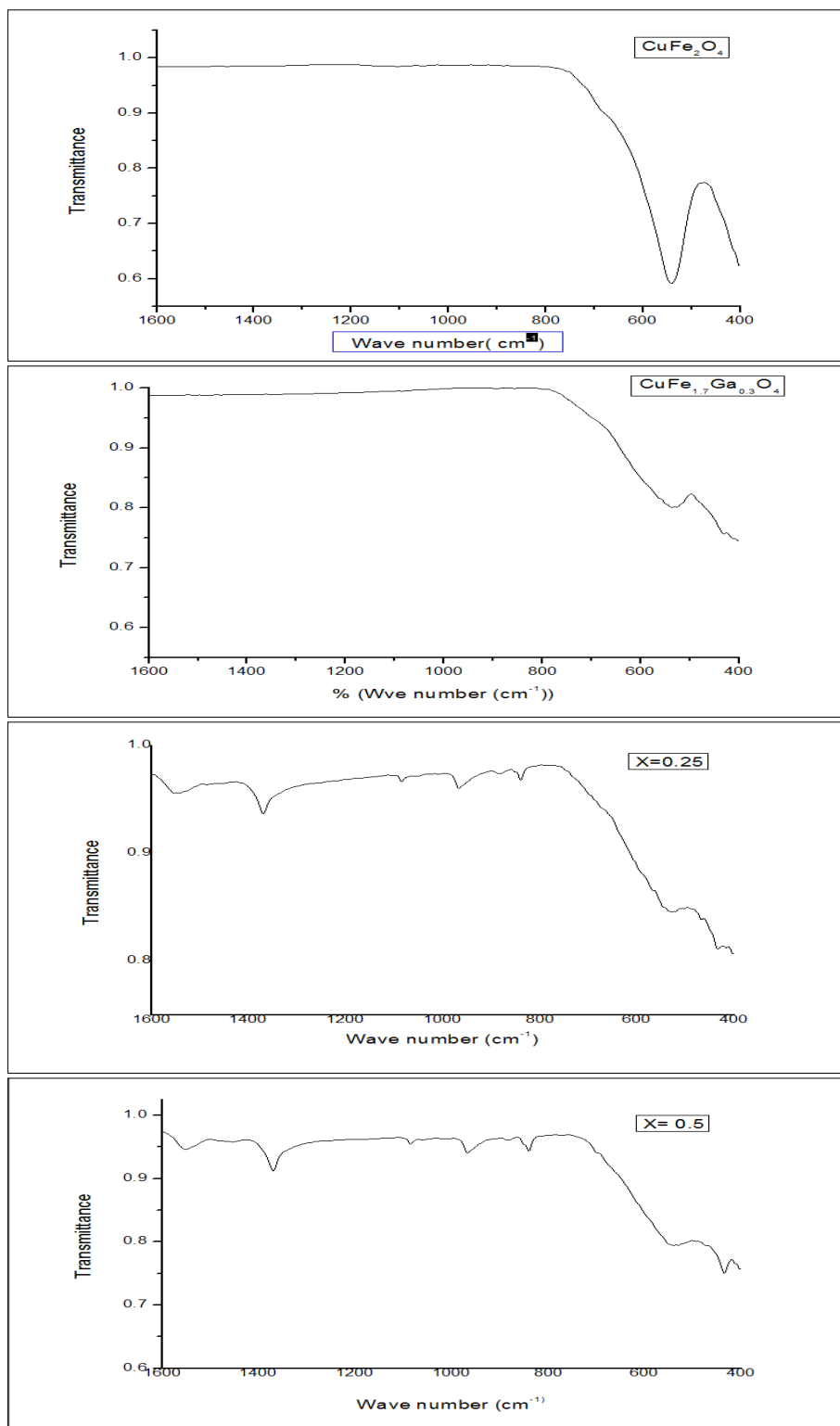
Since the spinel ferrites have four IR active bands that are shown as 4F1u. They are in the spectral region  $\nu_1$  (300-200  $cm^{-1}$ ),  $\nu_2$  (380-335  $cm^{-1}$ ),  $\nu_3$  (525-390  $cm^{-1}$ ), and  $\nu_4$  (630-560  $cm^{-1}$ ). Two of the bands reported and found in the far-IR region have not been observed because the range started from 400  $cm^{-1}$ . [ 10-12].

The recorded FTIR spectra for the synthesized samples are shown in Fig. 2. The FT-IR spectroscopy provides valuable information regarding to the nature of the functional groups present in the Mn ferrite samples. The band (1625 - 3333  $cm^{-1}$ ), are attributed to the stretching modes and H-O-H bending vibrations of the free or absorbed water.

The band near ( $1104\text{ cm}^{-1}$ ) is due to the antisymmetric NO-stretching vibrations arising from the nitrate group which is present as residue in the samples. This band is very weak in the spectra of sol-gel auto combustion derived sample, indicating the purity of Mn ferrite nanoparticles synthesized by this method.

The spectrum showed two main broad metal-oxygen bands are seen in the infrared spectrum of all spinels, especially ferrites. The higher one generally observed in the range ( $745\text{-}599\text{ cm}^{-1}$ ), is caused by the stretching vibrations of the tetrahedral metal-oxygen bond. The lowest band usually observed in the range ( $482\text{-}399\text{ cm}^{-1}$ ), is caused by the metal-oxygen vibrations in the octahedral sites fig. 2.

These bands are assigned to the vibrations of the tetrahedron and octahedron for the trivalent ( $\text{Fe}^{3+}$ ) cation respectively. First mode is attributed to the stretching vibrations of tetrahedrally coordinated  $\text{Fe}^{3+}\text{-O}^{2-}$  bonds and second mode is attributed to the metal oxygen vibrations in the octahedral sites (B).



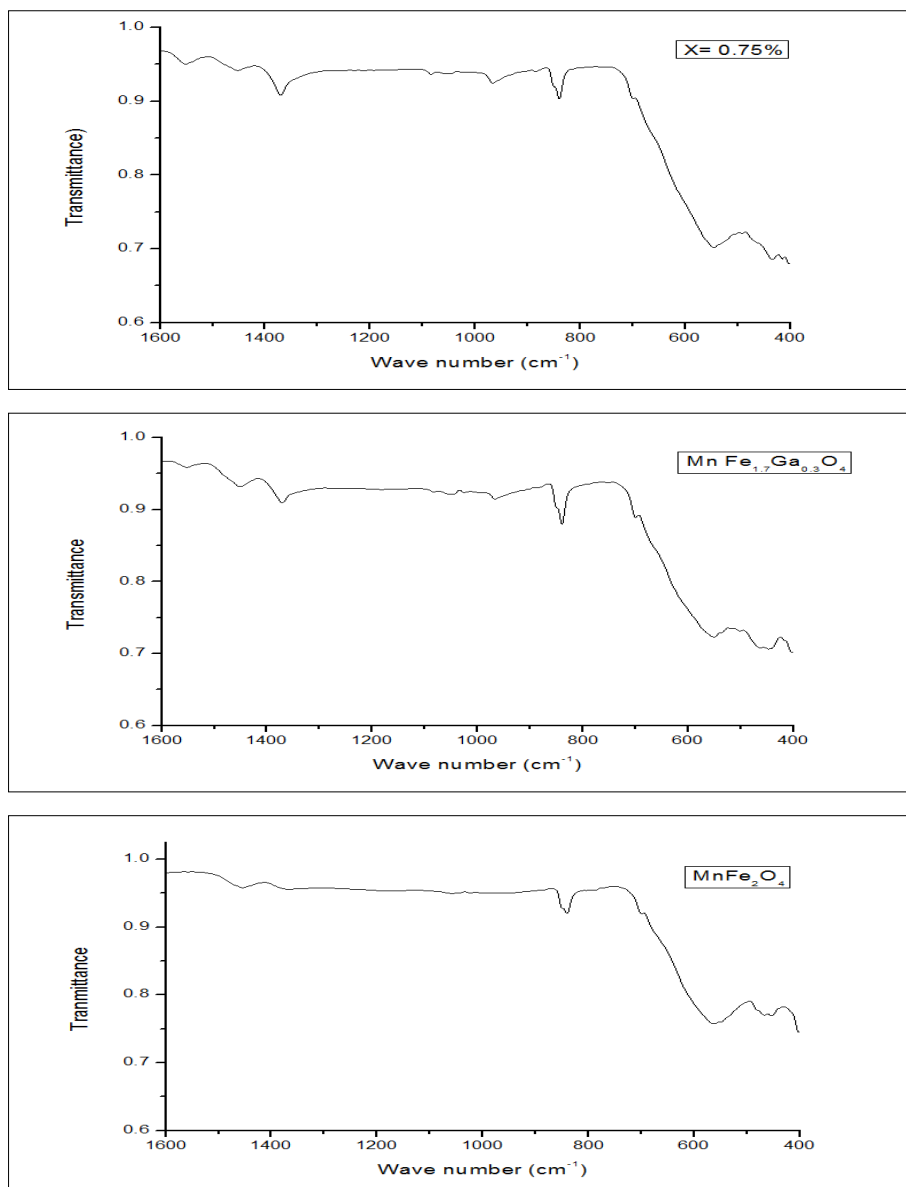


Fig. 2. FTIR spectra for  $Mn_{1-x}Cu_xFe_{1.7}Ga_{0.3}O_4$ ,  $CuFe_2O_4$  and  $MnFe_2O_4$  samples.

<i>Sample</i>	<i>Wave number</i> $\nu_1$ ( $Cm^{-1}$ )	<i>Intensity I</i> <sub>1</sub> (%)	<i>Wave number</i> $\nu_2$ ( $Cm^{-1}$ )	<i>Intensity I</i> <sub>2</sub> (%)
$CuFe_2O_4$	436.474545	31	599.285042	18
$CuFe_{1.7}Ga_{0.3}O_4$	399.660006	21	526.946226	12
$Mn_{0.25}Cu_{0.75}Fe_{1.7}Ga_{0.3}O_4$	423.765808	45	505.248783	77

$Mn_{0.5}Cu_{0.5}Fe_{1.7}Ga_{0.3}O_4$	415.278447	26	688.995517	16
$Mn_{0.75}Cu_{0.25}Fe_{1.7}Ga_{0.3}O_4$	482.221363	26	696.183541	11
$MnFe_{1.7}Ga_{0.3}O_4$	420.983843	31	696.670034	12
$MnFe_2O_4$	421.315418	19	745.738648	20

Table.1 Absorption band positions and their respective intensities

The observed changes in intensities in table.1 at both A and B sites also support the occupancy of copper ions at both A and B sites. But more change in these parameters at octahedral site than that at tetrahedral site suggests that occupancy of copper ions at B site is more than that at A site.

The sample  $CuFe_2O_4$  was in agreement with Waldron et. al. [13] who reported that the higher absorption band  $\sim 595\text{ cm}^{-1}$  corresponds to the intrinsic vibrations of tetrahedral complexes and the lower absorption band at  $\sim 447\text{ cm}^{-1}$  is attributed to the vibrations of octahedral complexes. The different values of absorption peaks for octahedral and tetrahedral complexes of  $CuFe_2O_4$  crystals are due to the different values of  $Fe^{3+}-O^{2-}$  distances for octahedral and tetrahedral sites.

### Conclusion:

Single-phase, well-crystallized octahedral nanocrystals of Mn Cu ferrite were successfully synthesized using sol- gel method. XRD analysis revealed all the samples have the cubic spinel structure. Only for  $Cu_{1-x}Mn_xFe_{1.7}Ga_{0.3}O_4$ , impurity peaks were observed. The FTIR spectrum exhibited  $\nu_1$  and  $\nu_2$  fundamental bands, corresponding to octahedral and



tetrahedral sites in the ferrite structure and was in good agreement with other references.

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