

# FTIR Studies of Ni Substituted Mn-Zn Ferrite Nanoparticles

F. A. Ahmed<sup>1</sup>, L. N. Singh<sup>2</sup>

\*<sup>1,2</sup>Department of Physics, Dr. Babasaheb Ambedkar Technological University, Lonere, Raigad, Maharashtra, India

## ABSTRACT

Nanoparticles of Ni substituted Mn-Zn ferrite were prepared by sol-gel method. The samples were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). The x-ray diffraction confirms the nanocrystalline nature of the materials. The FTIR spectra reveal the two strong absorption bands in the range  $350\text{ cm}^{-1}$  –  $500\text{ cm}^{-1}$  and weak absorption bands in the range  $500\text{ cm}^{-1}$  –  $1500\text{ cm}^{-1}$ . EDX spectra of the samples display the elemental composition and it agree with stoichiometric interactions of the prepared materials.

**Keywords:** Nanoparticles, Sol-gel method, FTIR, EDX spectra

## I. INTRODUCTION

Ferrites are ceramic ferromagnetic materials are extensively used in electronic and magnetic industries [1]. The general chemical formula of ferrites is  $MFe_2O_4$  where M is a divalent metal ions of elements such as Fe, Mn, Ni, Zn, Mg, Cu, Cd etc. A mixture of these ions is also achievable; which is generally known as mixed ferrite [2] Ferrites are electrically insulating materials, so they are widely used as high frequency magnetic materials. Mn-Zn ferrites have relatively high saturation magnetization however; resistivity is relatively low, so that they are not suitable for high frequency applications. On other hand Ni-Zn ferrites possess extremely high resistivity but relatively low permeability [3]. The literature on combination of Mn-Zn and Ni-Zn ferrite is poor [4]. The ferrites with relatively high resistivity and high permeability, for high frequency magnetic applications have been achieved by Ni substitution in Mn-Zn ferrite.

A large number of mixed ferrites like Mn-Zn-Cr [5], Ni-Zn-Al [6], Mn-Zn-Cd [7], Mn-Ni-Zn [8], Mn-Ni-Zn [9], Ni-Cu-Zn [10], Ni-Mg-Zn [11] have been synthesized with various processing methods and their structural, magnetic and electrical properties have been studied. The processing technique plays a very crucial role in the structural, magnetic and electrical properties of the ferrite nanoparticles, as properties of nanoparticles depend on shape and size. A variety of processing methods have been used for the synthesis of ferrite nanoparticles. The sol-gel method is favourable because it is simple, quick and economical. The major benefit of this method is that it is cheap, better mixing of starting materials and too much chemical homogeneity in the prepared material [12]. In the present work we reported the FTIR, SEM and EDX studies of Ni substituted Mn-Zn ferrite nanoparticles prepared by sol-gel method.

## II. METHODS AND MATERIAL

In the present work nanoparticles of  $Mn_{0.5-x}Ni_xZn_{0.5}Fe_2O_4$  ( $x=0.0, 0.1, 0.2, 0.3$ ) have been

synthesized by sol-gel method. The analytical grade materials- manganese chloride, zinc chloride, nickel chloride, iron chloride, citric acid and ethylene glycol were weighed in stoichiometric proportions and dissolved in a minimum amount of distilled water. After complete dissolution of all materials, a proper amount of ammonia solution was added drop wise to this solution with continuous stirring and pH of the solution was adjusted between 6 and 7. The solution was kept on a hot plate with continuous stirring at 90°C. The molar ratio of metal chlorides to citric acid was taken as 1:3 and the citric acid was mixed with metal chlorides solution during continuous heating to remove the insoluble residue and ethylene glycol was also added in the solution for homogeneity. After 2-3 hours of continuous heating due to evaporation, the solution becomes viscous brown gel. The gel was then placed in oven at 250°C for 12 hours and then it was crushed into fine powder form. The obtained powders were annealed at 800°C for 2 hours and then sintered at 1000°C for ½ hour. The X-ray diffraction of the sintered materials was obtained by using Bruker Diffractometer with Cu K $\alpha$  radiation source at room temperature. The Fourier transform infrared (FTIR) spectra has been recorded in the range 350 cm<sup>-1</sup> to 1500 cm<sup>-1</sup> by using a FTIR spectrometer. SEM images of various samples were recorded by using Hitachi E-1010 type Scanning Electron Microscope.

### III. RESULTS AND DISCUSSION

#### A. Structural Analysis

XRD have been used to determine the crystalline structure of metal oxides by equating the diffraction angle ( $2\theta$ ) through that of metal oxides of familiar compositions. Figure 1 exhibits the XRD pattern of  $Mn_{0.5-x}Ni_xZn_{0.5}Fe_2O_4$  ( $x=0.0, 0.1, 0.2, 0.3$ ) ferrite nanoparticles.

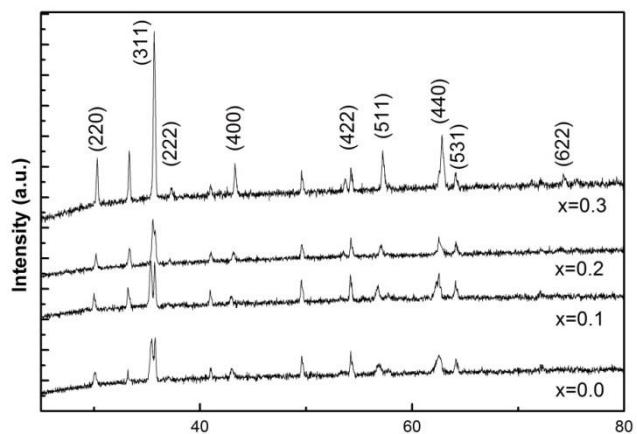


Figure 1: XRD spectra of  $Mn_{0.5-x}Ni_xZn_{0.5}Fe_2O_4$  ( $x=0.0, 0.1, 0.2, 0.3$ )

The patterns show usual reflection of (220), (311), (222), (400), (422), (511), (440), (531) and (622) planes reveal the configuration of cubic spinel structure of all the samples. The broad XRD peaks reveal the nanocrystalline nature of the samples. Some impurity reflections in XRD patterns are due to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> phase [13]

#### B. FTIR Studies

Figure 2 shows the Fourier Transform Infrared (FTIR) spectra of all the samples recorded in the range 350 cm<sup>-1</sup> – 2000 cm<sup>-1</sup>. The IR spectra show the two strong absorption bands in the range 350 cm<sup>-1</sup> – 500 cm<sup>-1</sup> and weak absorption bands in the range 500 cm<sup>-1</sup> – 1500 cm<sup>-1</sup>. The high frequency band ( $v_1$ ) observed in the range 360 cm<sup>-1</sup> – 500 cm<sup>-1</sup>, assigned to the intrinsic stretching vibrations of the metal ion-oxygen at the tetrahedral sites, while the low frequency band ( $v_2$ ) observed in the range 350 cm<sup>-1</sup> – 450 cm<sup>-1</sup>, corresponds to the octahedral metal ion stretching [ 14-15 ]. The vibrations  $v_1$  and  $v_2$  of different frequency may be attributed to the long bond length of oxygen – metal ions in the octahedral sites and shorter bond length of oxygen – metal ions in the tetrahedral sites. These two main absorption bands are common feature of ferrites which confirm the spinel ferrite structure [16]. It has been observed that both high frequency band  $v_1$  and low frequency band  $v_2$  are found to decrease with increase in Ni substitution. The spectra exhibit some absorption

bands from the range  $800\text{ cm}^{-1}$  –  $1100\text{ cm}^{-1}$ , which were ascribed to the stretching and H – O – H bending vibrations.

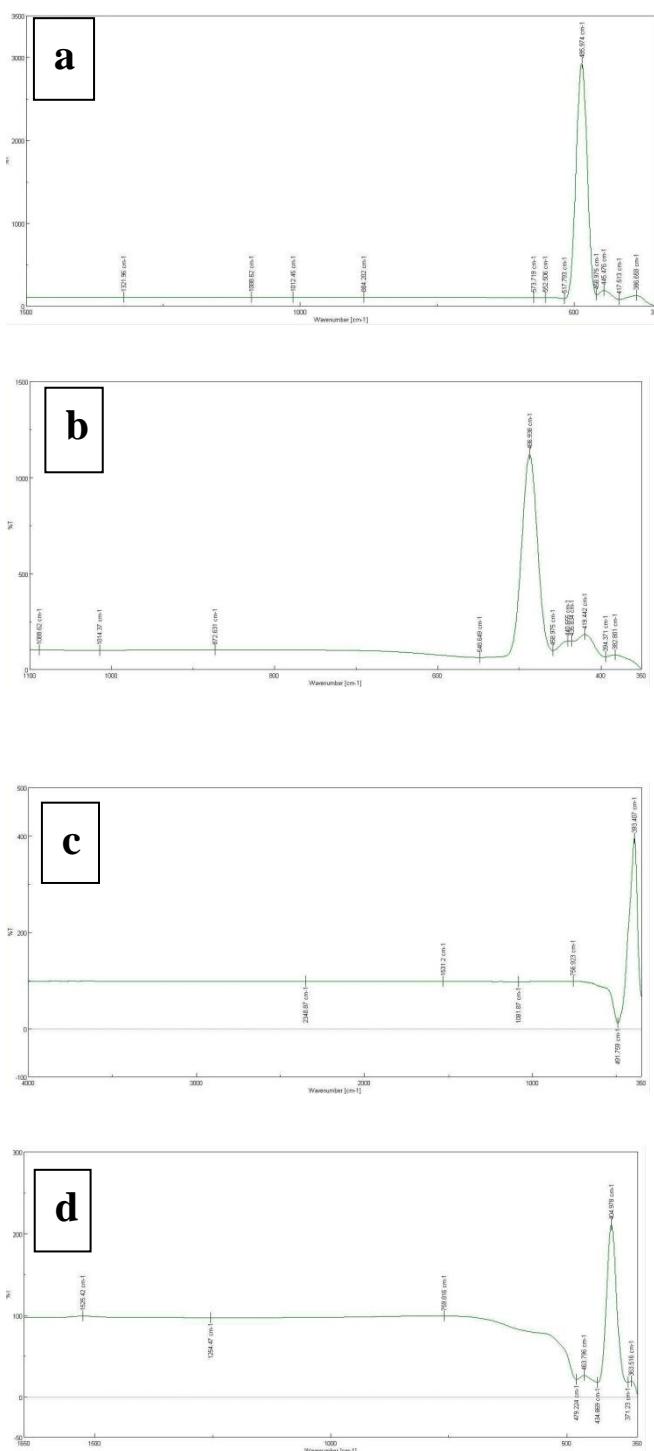


Figure 2: FTIR spectra of  $\text{Mn}_{0.5-x}\text{Ni}_x\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  ferrite nanoparticles, (a)  $x=0.0$ , (b)  $x=0.1$ , (c)  $x=0.2$ , (d)  $x=0.3$

### C. SEM and EDX Studies

Scanning Electron Microscope (SEM) micrographs of  $\text{Mn}_{0.5-x}\text{Ni}_x\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  ( $x=0.0, 0.1, 0.2, 0.3$ ) ferrite nanoparticles synthesized via sol-gel method is presented in figure 3. The SEM images of powder showed that the surface microstructure is porous and agglomerated. It is observed that all the samples are homogenous and nearly spherical in shape. The micrograph also shows the presence of more number of smaller grains. Smaller grains have large surface to volume ratio which have a direct effect on the properties of these ferrites [7].

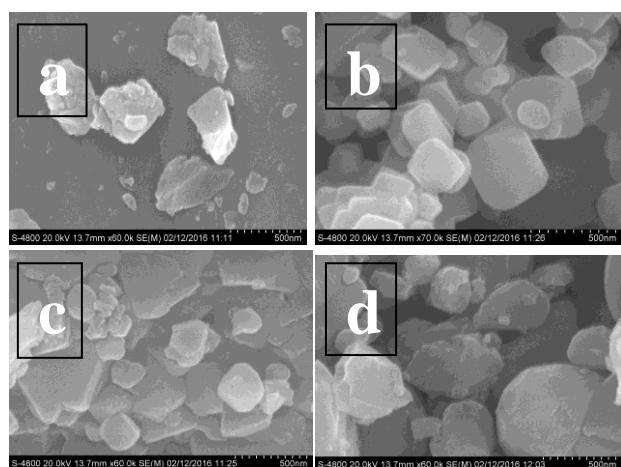
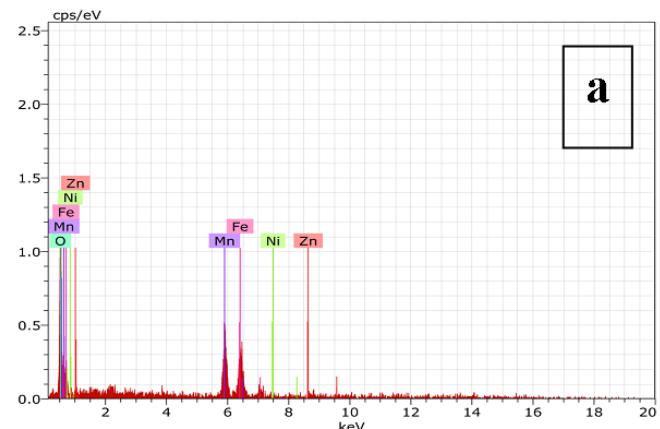


Figure 3: SEM micrographs of  $\text{Mn}_{0.5-x}\text{Ni}_x\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  ferrite nanoparticles, (a)  $x=0.0$ , (b)  $x=0.1$ , (c)  $x=0.2$ , (d)  $x=0.3$

The chemical compositional analysis of nickel substituted Mn-Zn ferrite nanoparticles have been observed by energy-dispersive detection X-ray spectra shown in figure 4 (a-d).



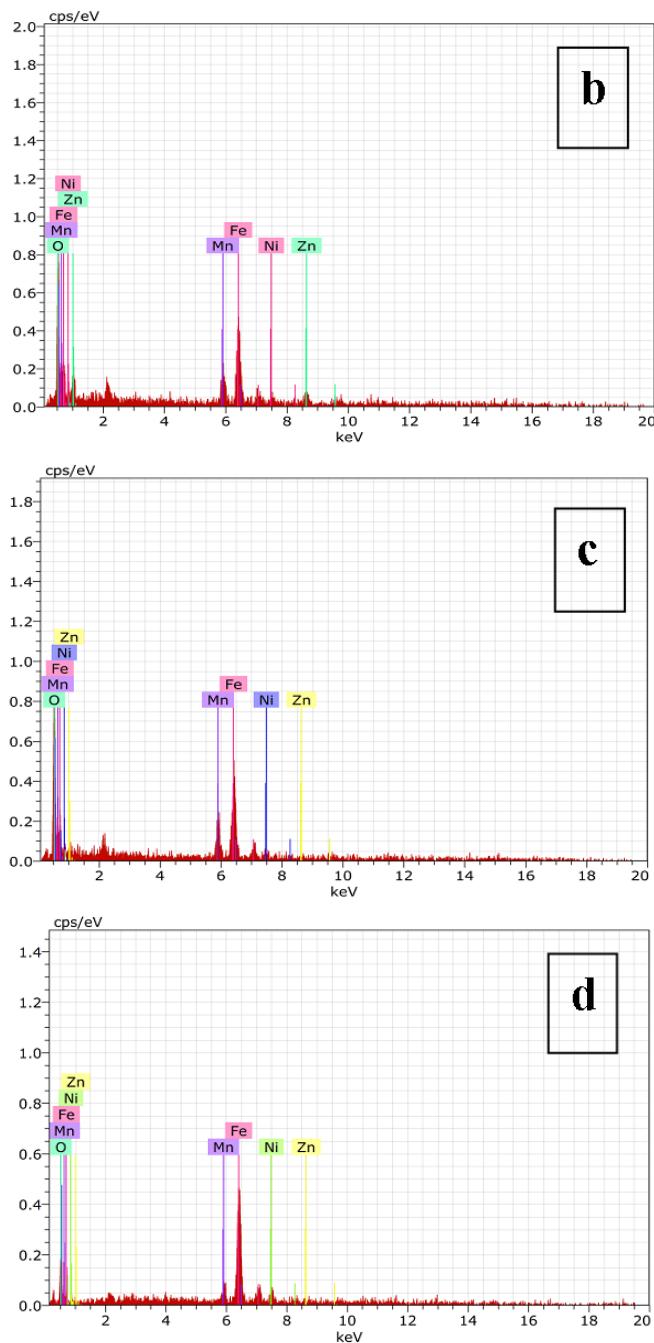


Figure 4: EDX pattern of  $\text{Mn}_{0.5-x}\text{Ni}_x\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  ferrite nanoparticles, (a)  $x=0.0$ , (b)  $x=0.1$ , (c)  $x=0.2$ , (d)  $x=0.3$

EDX characterization exhibits the compositional details of the prepared material. In this technique an electron beam of 10 – 20 kev strikes with the surface of the sample as a result x-ray to be emitted. The energy of the emitted x-ray is different for different elements and gives the presence of compositional element [17]. The elemental composition of the samples obtained by EDX spectrum confirms the

presence of O, Mn, Fe, Ni and Zn elements. The data obtained by the quantitative

analysis of EDX spectrum is presented in table 1. The elemental compositions correspond with the stoichiometric relations of the prepared material. There is no any impurity found in the samples which is indication of the purity of the prepared material. It has been observed that some Zn is lost in the samples [Table 1] probably due to  $\alpha\text{-Fe}_2\text{O}_3$  as confirmation from XRD peaks [Figure 1]. Due to some Zn lost an equivalent surplus iron hydroxide in the samples causes precipitation of  $\alpha\text{-Fe}_2\text{O}_3$  as an extra peak [18]. A. Kmita et al. [19] reported above 473 K the thermal decomposition process of zinc ferrite starts according to the following reactions,

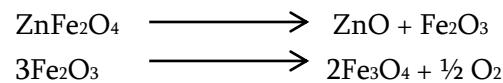


Table 2: EDX analysis of  $\text{Mn}_{0.5-x}\text{Ni}_x\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  ( $x=0.0, 0.1, 0.2, 0.3$ )

Ni Content (x)	Wt %	Element					Total %
		O	Mn	Fe	Ni	Zn	
0.0	Norm. c.	43.5 6	30.5 2	24.6 4	0.3 0	0.98	100
	Ato m. c.	72.8 0	14.8 6	11.8 0	0.1 4	0.40	100
0.1	Norm. c.	37.7 6	12.3 6	34.9 7	4.3 2	10.6 0	100
	Ato m. c.	68.4 7	6.53 7	18.1 7	2.1 3	4.70	100
0.2	Norm. c.	39.8 0	14.4 9	39.0 5	5.0 0	1.67	100
	Ato m. c.	69.8 5	7.41 3	19.6 9	2.3 9	0.72	100
0.3	Norm. c.	14.7 6	7.90 7	68.0 3	8.4 3	0.83	100
	Ato m. c.	37.7 9	5.89 2	49.9 8	5.8 8	0.52	100

#### IV. CONCLUSION

Nickel substituted Mn-Zn ferrite nanoparticles were synthesized by sol-gel method. The XRD patterns are characteristic of cubic spinel structure. SEM images reveal the surface microstructure of the prepared materials is porous and agglomerated. EDX spectra confirm there is no any impurity found in the samples. From FTIR spectra it has been observed that both high frequency band  $\nu_1$  and low frequency band  $\nu_2$  are found to decrease with increase in nickel substitution.

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