# PREPARATION, CHARACTERIZATION AND STUDY OF ELECTRICAL AND MAGNETIC PROPERTIES OF FINE PARTICLE Mn-Zn FERRITES BY NITRILOTRIACETATE PRECURSOR METHOD

**Project Investigator: S.H.P.Keluskar**

**Annual Report**

**2002-2003**

The phenomenon of magnetism and the behavior of magnetic materials are well known. The domain structure, magnetic interaction and the basic properties of these materials are of considerable importance in modern technology. Magnetic materials have gained importance due to variety of applications. They form essential components in the form of magnetic cores in audio-video equipment, high frequency transformers, memory units in computers, magnetic amplifiers, power inductors, broad band pulse transformers, electromagnetic interference (EMI) suppressors, asymmetric digital subscriber line (ADSL) and splitter application, mobile communication, microwave absorbers for special applications in defence etc.

**Ferrimagnetic Materials**

Ferrimagnetism exists in those materials in which atomic moments are aligned antiparallel but magnetic moments are unequal as shown in (fig.1.3).

↑ ↓ ↑ ↓ ↑ ↓ ↑ ↓

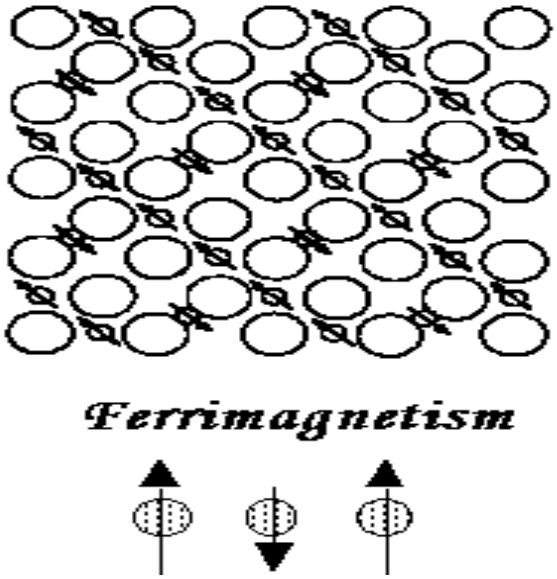



Fig.1.3

This is due to the fact that the opposing magnets have different moments or the number of atoms having one spin direction is different from that having opposite spin direction thus giving a magnetic moment.

Several magnetic oxides have been found to be magnetic; of these, ferrimagnetic oxides, popularly known as ferrites are of immense importance due to their special characteristics. Mixed metal oxides with iron oxides as their main components are known as mixed ferrites.

Ferrites crystallize in three different crystal types namely spinel, garnet and magnetoplumbite. The spinel and garnet have cubic structure while plumbite has hexagonal structure.

The general formula of simple spinel ferrites is $A^{II}\ Fe_rO_4$ where $A^{II}$ is divalent metal ion.

The interesting and useful electrical and magnetic properties of spinel ferrites are governed by distribution of the ion and divalent metal ions among octahedral and tetrahedral sites of spinel lattice [10-14]. The tetrahedral and octahedral sites are conventionally called A and B sites respectively.

**For this study the following work plan was executed**

Step1: Synthesis of ultra fine particle $Mn_x\ Zn_{(1-x)}Fe_2O_4$ with

x= 0.3,0.35,0.4,0.45,0.5,0.55,0.6,0.65,0.7

Step2: Characterization of powder samples.

Step3: Grain size measurements of powder samples.

Step4: Study of Magnetic properties of powder samples.

Step5: Study of Electrictic properties of powder samples.

## PREPARATION OF MATERIALS

The most important aspect is still the development of new strategies for the synthesis of nanomaterials, particularly soft chemical routes. Chemical methods of synthesis play a crucial role in designing and discovering new and novel materials and in providing less cumbersome methods for preparing known materials. Chemical methods also enable the synthesis of metastable materials, which are otherwise difficult to prepare. In this chapter, the various methods of synthesizing oxide materials are briefly summarized with emphasis on soft-chemical routes.

### 3.4 SYNTHESIS OF $Mn_{(x)}Zn_{(1-x)}Fe_2O_4$ MIXED FERRITE IN PRESENT STUDY

Calculated amounts of manganese nitrate tetrahydrate and zinc nitrate hexahydrate were taken as per the various proportions of $Mn^{+2}$ : $Zn^{+2}$ ions mentioned in the Table 3(a & b) and dissolved in sufficient volume of distilled water to obtain solution mixture of two ions. Calculated amount of ferrous sulphate heptahydrate was taken such that the proportion of $Fe^{+2}$ to $Mn^{+2}$ and $Zn^{+2}$ together was 2:1. This $Fe^{+2}$ salt was dissolved in sufficient amount of distilled water and requisite volume of aqueous solution of barium nitrate was added to precipitate $SO_4^{2-}$ (sulphate) as $BaSO_4$ (barium sulphate). Resultant mixture was filtered to remove insoluble

$BaSO_4$ and washed with water to remove last traces of $Fe^{2+}$ ions. Filtrate along with washings, which contained ferrous nitrate solution, was added to aqueous solution of manganese-zinc nitrate followed by appropriate amount of ligand solution.

This solution mixture was than processed to obtain the fine particle

$Mn_{(x)}Zn_{(1-x)}Fe_2O_4$ ferrite material by two different techniques.

(a) The solution mixture was then heated slowly to dryness. The solid mass was then ignited to decompose to obtain the Mn-Zn mixed ferrite product.

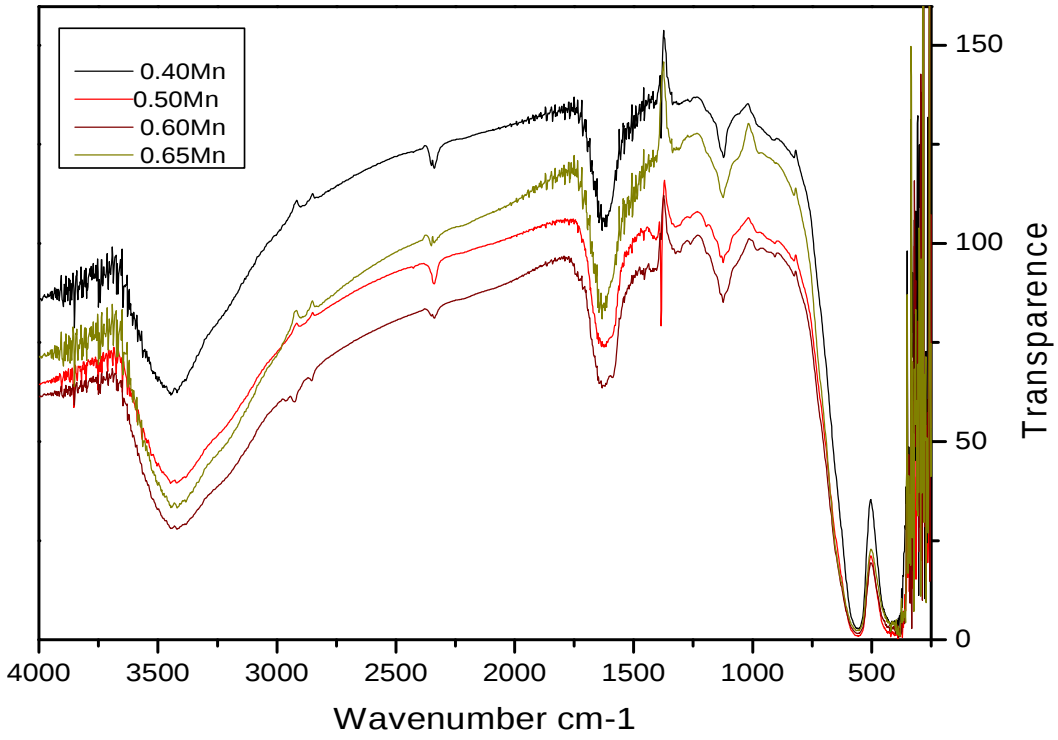
(b) The solution mixture was heated slowly to dryness and was allowed to decompose by self ignition in a microwave oven to obtain Mn-Zn mixed ferrite product.

| No. | Mn : Zn | Amount of $Mn(NO_3)_2$ . $4H_2O$g (moles) Molecular Weight =250.94g | Amount of $Zn(NO_3)_2$ .$6H_2O$ g (moles) Molecular Weight =297.38g | Amount of $FeSO_4 \cdot 7H_2O$ g (moles) Molecular Weight= 555.70g |
|---|---|---|---|---|
| 1 | 0.40:0.60 | 50.0376 (0.040) | 17.8428 (0.060) | 111.14 (0.2) |
| 2 | 0.45:0.55 | 11.2923 (0.045) | 16.3559 (0.055) | 111.14 (0.2) |
| 3 | 0.50:0.50 | 12.5470 (0.050) | 14.8690 (0.050) | 111.14 (0.2) |
| | | | | |

| | | | | |
|---|---|---|---|---|
| 4 | 0.55:0.45 | 53.8017 (0.055) | 13.3821 (0.045) | 111.14 (0.2) |
| 5 | 0.60:0.40 | 15.0564 (0.060) | 11.8952 (0.040) | 111.14 (0.2) |
| 6 | 0.65:0.35 | 16.3111 (0.065) | 10.4083 (0.035) | 111.14 (0.2) |

The samples obtained were characterized using XRD and IR.

IR spectra for all the samples were recorded on Shimadzu FTIR 8900 spectrometer.



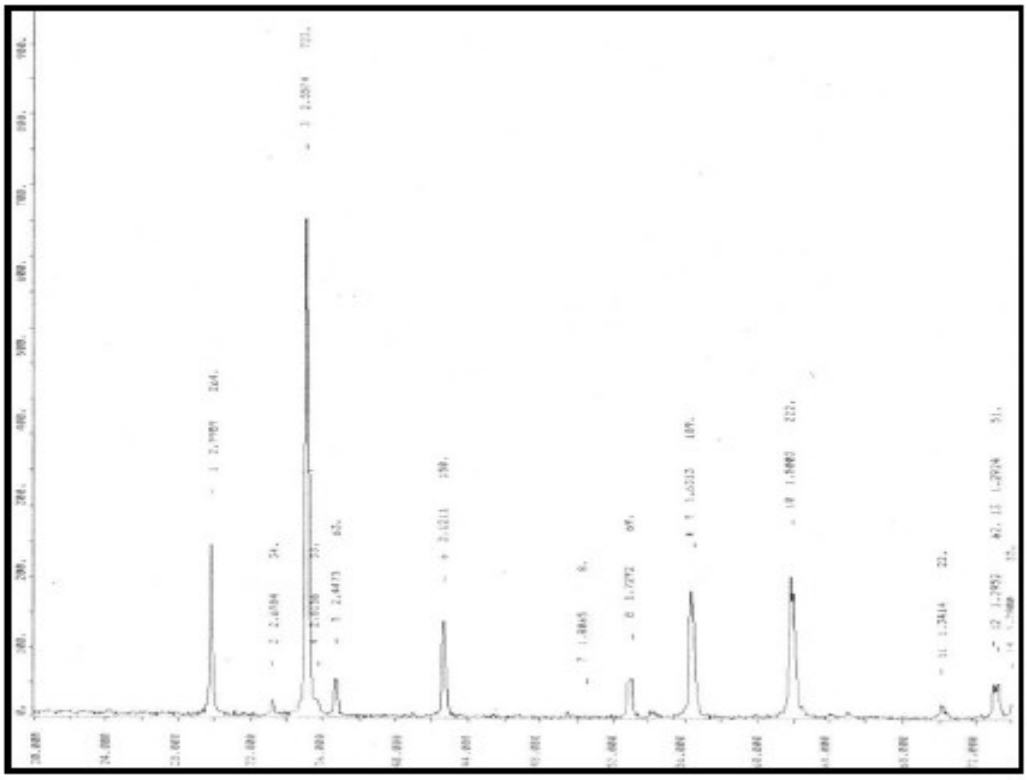

IR spectra of dried samples were recorded in the waverange of 4000-250 $cm^{-1}$.

The metal oxygen absorption bands at 600-550 cm-1 and 450-385cm-1 are characteristically pronounced for all spinel structures and for ferrites in particular. The band 350-330 cm-1 is less intense and sometimes it merges with the band 450-385 cm-1 giving a single wide band at 420-330 cm-1.This agrees with the reported values.

## X-Ray Diffraction Analysis

In the present study X-ray diffraction is used to confirm the formation of spinel structure, to determine interplanar distances and the lattice constants.

The XRD pattern clearly identifies a single phase of Mn-Zn ferrite. The patterns similar to the XRD patterns reported in the literature for Mn-Zn ferrite. The values of lattice constants *a* calculated from these were found to be in excellent agreement with reported values.



| Sample | Experimental value for 'a' in AU | Average particle diameter D in nm. |
|---|---|---|
| $ZnFe_2O_4$ | 8.440 | 26.195 |
| $Mn_{(0.3)}Zn_{(0.7)}Fe_{(2)}O_4$ | 8.455 | 7.20 |
| $Mn_{(0.35)}Zn_{(0.65)}Fe_{(2)}O_4$ | 8.456 | 11.07 |
| $Mn_{(0.4)}Zn_{(0.6)}Fe_{(2)}O_4$ | 8.457 | 21.461 |
| $Mn_{(0.45)}Zn_{(0.55)}Fe_{(2)}O_4$ | 8.458 | 4.464 |
| $Mn_{(0.5)}Zn_{(0.5)}Fe_{(2)}O_4$ | 8.459 | 18.543 |
| $Mn_{(0.55)}Zn_{(0.45)}Fe_{(2)}O_4$ | 8.461 | 11.074 |
| $Mn_{(0.6)}Zn_{(0.4)}Fe_{(2)}O_4$ | 8.463 | 25.278 |
| $Mn_{(0.65)}Zn_{(0.35)}Fe_{(2)}O_4$ | 8.470 | 10.7274 |
| $Mn_{(0.7)}Zn_{(0.3)}Fe_{(2)}O_4$ | 8.479 | 10.106 |
| $MnFe_2O_4$ | 8.510 | 15.720 |

The values of ‘a’ matches with the reported values and the estimated particle size is in the range 7-30nm.

This also confirms that the material prepared is a  ultrafine material.