ESTD. 2010

Crossian Resonance

A Multidisciplinary Research Journal

(A Refereed Biannual Published in June and December)

ISSN 0976-5417

Vol. 9 No. 1 June 2018

Synthesis of Magnesium Ferrite by Co-Precipitation Method

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ABSTRACT

Magnesium ferrite was synthesized by a simple co-precipitation method. The crystal structure and the metal-oxide phase formation were investigated by powder X-ray diffraction (PXRD) and Fourier Transform Infrared Spectroscopy (FTIR). Lattice parameter, volume of the unit cell and X-ray density were determined from PXRD pattern and the lattice parameter values were verified by Bradley-Jay and Nelson-Riley plots. Average crystallite size was estimated from the PXRD pattern using Scherrer equation. The FTIR spectra showed two principal absorption bands in the range of 400cm^{-1} to 1000 cm^{-1}. The values of force constant for tetrahedral (K_t) and octahedral (K_o) sites were calculated.

Keywords: *$MgFe_2O_4$, PXRD, FTIR, lattice parameter, X-ray density, force constant.*

I. INTRODUCTION

Ferrites are the ferromagnetic oxides containing iron oxide and another metal oxide in proper proportion. They have the general formula of MFe_2O_4 (where M is a divalent metal ion, eg. Fe, Co, Ni, Mg, etc.). In a spinel structure, there are 56 ions, 32 oxygen and 24 metal ions in a unit cell. In most ferrite materials, the substituents play an important role in determining the variation of the physical properties, the magnetic and electric transport properties are affected by the substituents. A general formula of ferrite structure is denoted as $(M_xFe_{1-x})[M_xFe_{2-x}]O_4$, in which M shows cations that occupy tetrahedron sites and x is degree of inversion. Among the ferro-spinels, the inverse type is particularly interesting due to its high magneto crystalline anisotropy, high saturation magnetization and unique magnetic structure. Magnesium ferrite ($MgFe_2O_4$) possesses cubic structure and has a normal spinel structure. At this structure, Mg^{2+} ions occupy octahedron B site and Fe^{3+} ions occupy both tetrahedron A and octahedron B-sites. Thus, the compound can be represented by the formula $(Fe^{3+}_{1-x})[Mg^{2+}_{x}Fe^{3+}_{1-x}]O^{2-}_4$, where the round and the square brackets represent A and B sites, respectively. There are various preparation methods such as solid state reaction, sol-gel, chemical co-precipitation, hydrothermal etc. Among them co-precipitation method is employed as it is simple, easy and economic, also it is a proper technique for making small size and monodisperse nanoparticles [1].

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Ferric nitrate nonahydrate ($Fe(NO_3)_3.9H_2O$), magnesium nitrate hexahydrate ($Mg(NO_3)_2 6H_2O$) are used as precursors; Sodium hydroxide (NaOH) and acetone of analytical reagent grade were used.

2.2 Sample Preparation

Magnesium ferrite nanoparticles ($MgFe_2O_4$) have been prepared by using co-precipitation method. Ferric nitrate nonahydrate and magnesium nitrate hexahydrate are used as precursors. 2M $Fe(NO_3)_3.9H_2O$ and 1M $Mg(NO_3)_2 6H_2O$ are dissolved in 40 ml of double distilled water and subjected to magnetic stirring. The pH of the solution is adjusted to 10 by adding drop wise 0.1M NaOH solution. Obtained brown precipitate was filtered and washed repeatedly with double distilled water and ethanol until a pH of 7 was achieved. The precipitate is dried at 100^{0}C for several hours and subsequently calcined at 400^{0}C and 600^{0}C for 3 hours to get magnesium ferrite nanoparticles as end product [2].Once again the final product is finely powdered. The synthesized sample was characterized by PXRD and FTIR. The synthesized nanoparticles are found to be dark brown in color.

3. RESULTS AND DISCUSSION

3.1. Powder X-ray Diffraction Analysis

Characterization of the final product was performed by Powder X-ray powder diffraction analysis. The PXRD pattern is shown in Fig.1 (a). It exhibits typical reflections (311), (220), (400), and (440) planes that are indications of the presence of the cubic spinel structure. This diffraction lines provide clear evidence on the formation of $MgFe_2O_4$. The entire diffraction peaks match well with the standard values (JCPDS file No: 89-3084) and are indexed. No secondary phase was detected in PXRD, ensuring the phase purity of the final product. The lattice parameter of $MgFe_2O_4$was obtained using UNITCELL software as a=8.3779 Å. The precise value of lattice parameter of pure $MgFe_2O_4$ was calculated using Bradley-Jay and Nelson-Riley extrapolation methods that minimize the influence of systematic errors. Extrapolation against $\cos^2\theta$ is called Bradley-Jay method whereas extrapolation against $\{(\cos^2\theta/\sin\theta) + (\cos^2\theta/\theta)\}$ is called Nelson-Riley plot. The lattice parameters calculated by different methods are tabulated in Table 1.

The particle size of magnesium ferrite nanoparticles was determined from the full width at half maximum (FWHM) of the XRD patterns using the Scherer formula $\mathbf{d = 0.9\lambda/\beta\cos\theta}$, where d is the crystallite size (nm), β is the full width of the diffraction line at half the maximum intensity measured in radians, λ is the X-ray wavelength and θ is the Bragg angle. The crystallite sizes estimated using the Scherer formula was ranging from 7 to 21 nm. The actual (X-ray) density of $MgFe_2O_4$ nanoparticles is calculated using the formula $\mathbf{P_x = 8M/Na^3}$, Where M is the molecular weight of the sample, N the Avogadro's number and a lattice constant [3] and it is found as 5.313g/cc.

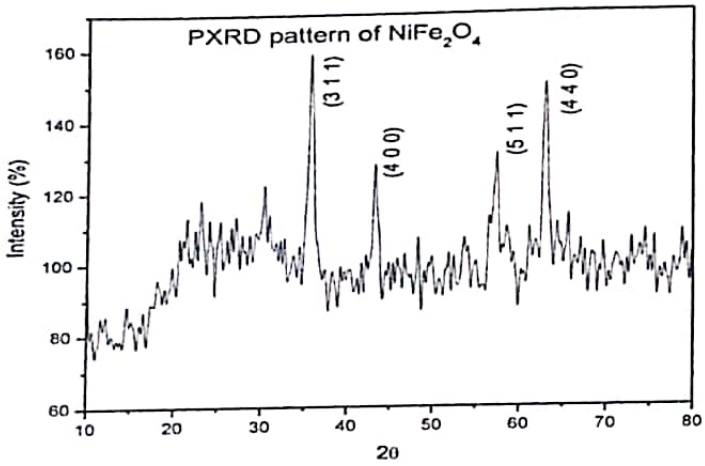

Fig. 1: (a) PXRD pattern of $MgFe_2O_4$nanoparticles

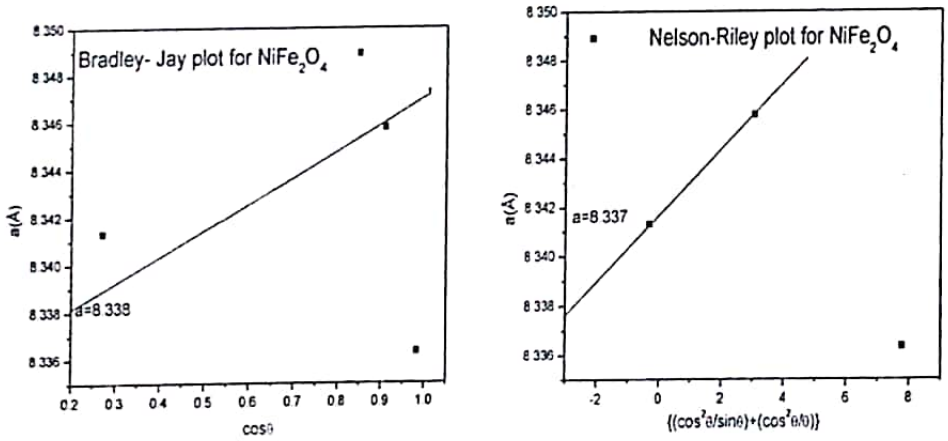

Fig. 1: (b) Bradley- Jay plot for $MgFe_2O_4$Fig. 1: (c) Nelson-Riley plot for $MgFe_2O_4$

Table 1: Lattice parameter 'a' by different methods

Method	Lattice parameter 'a' (Å)
JCPDS	8.3779
UNIT CELL	8.3779
By using formulaa $=d/(h^2+k^2+l^2)^{1/2}$	8.342
Bradley - Jay plot	8.3449
Nelson - Riley plot	8.3755

3.2. FTIR Studies

FTIR spectral analysis helps to confirm the formation of spinel structure in ferrite samples. In the FTIR spectrum two main broad metal–oxygen bands are observed in the $MgFe_2O_4$ spinels. The higher one (ν_1) observed in the wave number range 600–550 cm^{-1}, is caused by the stretching vibrations of the tetrahedral metal–oxygen bond. The lower band (ν_2) observed in the range 450–385 cm^{-1}[4].

The ν_1 and ν_2 for the as prepared $MgFe_2O_4$ sample was found to be 534cm^{-1} and 420cm^{-1}. The values of the force constants (K_T and K_O) for the band Fe^{3+}- O^{2-}at tetrahedral and octahedral sites were calculated using the relation $\mathbf{K = 4\pi^2\upsilon^2c^2m}$, where c is the speed of light, 9 is the band wave number in cm^{-1} and m is the reduced mass for Fe^{3+} ions and O^{2-} ions (2.061×10^{-23} g). The values of force constant for tetrahedral (K_T) and octahedral (K_O) sites were calculated as 2.086 $\times10^5$dyne.$cm^{-1}$and 1.29 x 10^5dyne.cm^{-1} for the as prepared sample. The band observed at 1382$cm^{-1}$was due to the surface co-ordinated O- H bond in ferrite [5].

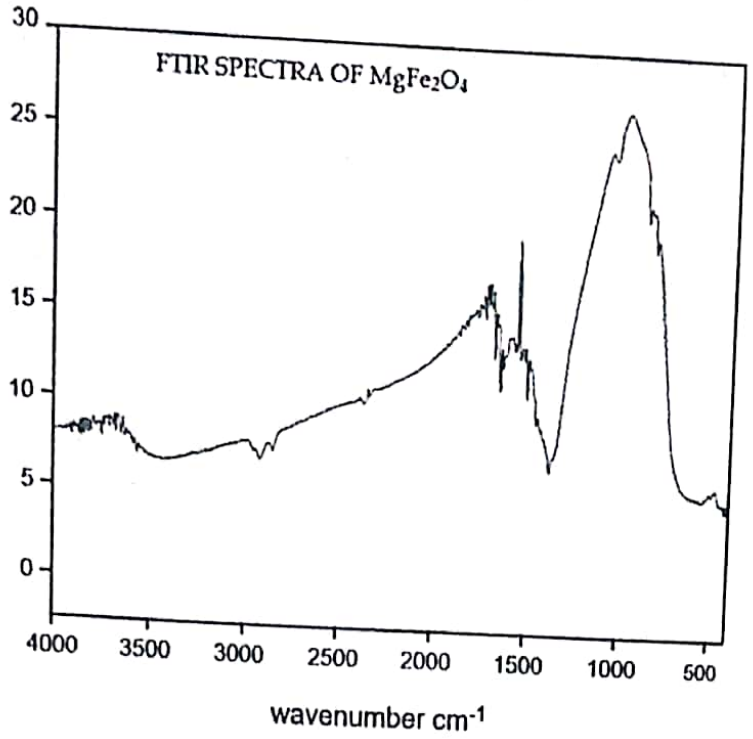

Fig. 2: FTIR spectrum of $MgFe_2O_4$nanoparticles

CONCLUSION

Magnesium ferrite nanoparticles (MgFe2O4) have been prepared using co-precipitation method. There is close agreement among the lattice parameter calculated from the JCPDS, XRD data through UNITCELL software, Bradley-Jay and Nelson-Riley extrapolation methods. The metal – oxide phase formation was confirmed through the FTIR band assignments. Force constant for the Fe – O bonds in the tetrahedral and octahedral sites of $MgFe_2O_4$was estimated.

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