J. Environ. Nanotechnol.
Volume 2, No.2 (2013) pp. 53-55
ISSN (Print) : 2279-0748
ISSN (Online) : 2319-5541
doi : 10.13074/jent.2013.06.132015

Synthesis and Characterization of Magnesium ferrite nanoparticles by Co-precipitation method

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Received: 05.04.2013 Revised : 20.04.2013 Accepted: 26.04.2013

Abstract

Magnesium ferrite nanoparticles are successfully prepared by co-precipitation method. Magnesium chloride [$MgCl_2$. $6H_2O$], anhydrous Ferric chloride [$FeCl_3$] and sodium hydroxide are used as raw materials. Magnesium ferrite samples sintered at 130 °C and 600 °C are subjected to X-ray diffraction to calculate the average nano-crystalline size using Debye – Scherrer formula. The FT-IR spectra of the sample are recorded to ensure the presence of the metallic compounds. The morphological analysis of the sample is done using Scanning electron microscope (SEM).

Keywords: *XRD; Debye-Scherrer formula; FT-IR; X-ray diffration.*

1. INTRODUCTION

Nanoparticles of magnetic materials have been the subject of detailed study for their interesting electric, magnetic and optical properties which are considerably different from those of their bulk counterparts. These materials have potential applications in various fields of modern technologies, viz., as magnetic resonance imaging contrast agents, in ferrofluid technology and in magnetic caloric refrigeration. The search for new good gas-sensing materials and study of the new properties of conventional materials has become an active research field.

Magnesium ferrite belongs to a class of compounds having the general formula $MgFe_2O_4$ crystallizing with spinel structure. It is a typical spinel in which the cation distribution in the crystal lattice site is very much sensitive to heat treatment due to the high diffusibility of Mg^{2+} ions. The physical and chemical properties of ferrites are dependent upon factors such as sintering temperature, sintering time, rate of heating, rate of cooling, etc. (Bonnenberg et al. 1970). Various methods such as ceramic method (Johnson et al. 1985), sol–gel method (Kim et al. 2001; Subhash et al. 2004), hydrothermal method (Li et al. 2001), citrate method (Lal et al. 2005) and combustion method (Fu et al. 2009) are used for preparation of spinel ferrites. The co-precipitation method is widely used for preparation of ferrites due to its overriding advantages such as composition flexibility.

As prepared magnesium ferrite samples are sintered at 130Ë%C and 600Ë%C and are subjected to X-ray diffraction. The FT-IR spectra of these samples and the morphological analysis of the sample done using SEM are reported in this paper.

2. EXPERIMENTAL

Magnesium ferrite $MgFe_2O_4$ nanoparticles are prepared by co-precipitation method. The desired composition is obtained by taking stoichiometric amounts of magnesium chloride [$MgCl_2$.$6H_2O$] and anhydrous ferric chloride [$FeCl_3$] dissolved in distilled water. The neutralization is

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carried out with sodium hydroxide solution and the reaction temperature is maintained at 60ËºC. The pH of the solution is maintained at 8 and stirred for 2hrs. The precipitate is thoroughly washed with distilled water until it is free from impurities. The product is dried at a temperature of 100ËºC to remove the water contents. The dried powder is mixed homogeneously and sintered at 130ËºC & 600ËºC.

These samples are subjected to X-ray diffraction to calculate the average particle size using Debye – Scherrer formula. The FT-IR spectra of these samples are recorded to ensure the presence of the metallic compounds. The magnetic properties of the copper doped cobalt ferrite nano particles are studied using Vibrating Sample Magnetometer (VSM).The morphological analysis of the sample is carried out using Scanning Electron Microscope (SEM).

3. RESULTS AND DISCUSSION

3.1 FT-IR Spectral analysis

Fig. 1 shows FT-IR spectra in the range 4000–400 cm^{-1} for the magnesium ferrite samples

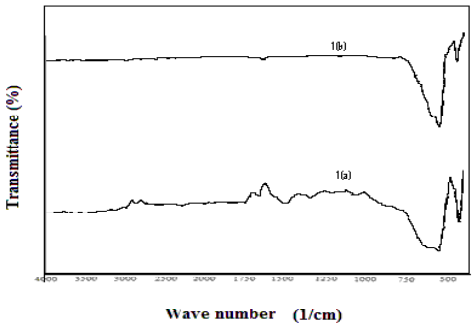


Fig 1(a & b) FT-IR spectra of Magnesium ferrite samples sintered at (a) 130ËºC (b) 600ËºC

sintered at 130ËºC and 600ËºC. The absorption band around 3560 cm^{-1} in fig1(a) indicates the presence of hydroxyl group and the absence of band around 3560 cm^{-1} in fig 1(b) implies that the hydroxyl group is completely removed when the sample is sintered at 600ËºC. The spectra exhibit two absorption bands around 595 and 440 cm^{-1} respectively. These spectra represent characteristic features of ferrospinels and the bands are assigned to metal oxygen stretching frequencies. Since tetrahedral M-O bonds are associated with higher force constants and lower bond lengths, the observed stretching frequency is expected to appear at a higher frequency compared to the M-O stretching frequency of octahedral (Oh M-O) sites (Evans and Hafner, 1968) .In view of this, the band appearing near 595 cm^{-1} is assigned to the M-O stretching mode of the tetrahedral group and that near the 440 cm^{-1} is assigned to the M-O stretching mode of octahedral group. The bands are usually assigned to vibrations of ions in the crystal lattice (Waldron 1955; Porta et al. 1974).

3.2 Structural Analysis

X-ray diffraction patterns of the $MgFe_2O_4$ samples sintered at 130ËºC and 600ËºC are presented in Fig. 2(a)&2(b). The well-defined (311) peak appears to be more intense. All the diffractograms showed the characteristic reflections of the spinel phase.

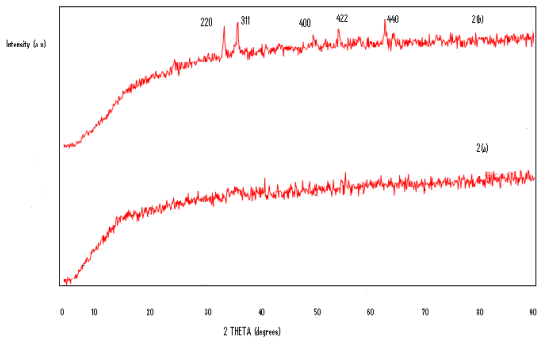


Fig 2(a & b) XRD analysis of Magnesium ferrite sample sintered at (a) 130ËºC (b) 600ËºC

diffractograms showed the characteristic reflections of the spinel phase. Fig. 2 also shows that the peaks become sharper and narrower with increasing

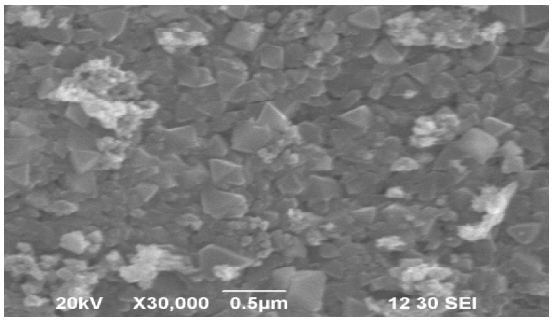
sinteringtemperature, indicating the enhancement of crystallanity. The average particle size (D) is calculated using the Scherrer formula [11].

D=0.9ë/âcosè

Where d is the mean crystallite size, ë is X-ray wavelength, â is the angular line width at half maximum intensity and è is the Bragg's angle. The average crystallite size from X-ray technique is found to vary from 5 to 14 nm.

3.3 Scanning Electron Microscope

Scanning electron micrograph for the sample $MgFe_2O_4$ sintered at 600ËC shown in Fig. 3 indicates the irregular shape of fine particles. It suggests the formation of grains by aggregation of small crystallites (Hankare et al. 2009).



ig. 3: SEM image of $MgFe_2O_4$ sample sintered at 600°C.

4. CONCLUSION

Magnesium ferrite nanoparticles are prepared by the co-precipitation method. The FT-IR spectra show main absorption bands around 595 cm^{-1} and 440 cm^{-1} corresponding to the vibration modes of the spinel compounds. XRD pattern reveals that the synthesized ferrites consist of nano crystalline particles with size in the range from 5 to 14nm and the crystalline nature of the sample is increased as the sintering temperature increases. It is also confirmed by the SEM micrographs.

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