ISSN : 0974 - 7486 Volume 7 Issue 5

Trade Science Inc.

Materials Science

An Indian Journal

Full Paper

MSAIJ, 7(5), 2011 [313-316]

Synthesis of nanocrystalline cobalt zinc ferrites by sol-gel auto combustion method and study of their properties

S.R.Kulal*, S.R.Bamane

Metal Oxide Research Laboratory, Dr. Patangrao Kadam Mahavidyalaya, Sangli, Maharashtra, (INDIA)

E-mail: srkulal@gmail.com

Received: 4th March, 2011 ; Accepted: 14th March, 2011

ABSTRACT

The ferrite compositions $Co_xZn_{1-x}Fe_2O_4$ with $x = 0.0, 0.25, 0.5$ 0.75 and 1.0, were synthesized by sol gel auto-combustion method. The powders were calcined at 950°C/6 h. These ferrite samples were characterized by using X-ray diffraction, Scanning electron microscopy, thermo-gravimetric and differential thermal analysis, FTIR spectroscopy, Electron dispersive analysis and transmission electron microscopy. The powders showed the presence of crystalline cubic spinel ferrite with about 28–32 nm crystallite sizes.

KEYWORDS

Ferrites;
Nitrates;
Glycine;
Sol gel auto combustion method;
Nanoparticles.

INTRODUCTION

The most important advances were made in ferromagnetism in the field of magnetic oxides. The advancement in the high frequency ferrite was initiated by Snock[1]. The chemistry of the ferrites with the spinel structure have technologically important potential applications such as magnetic resonance imaging[2], magnetic drug delivery[3], magnetic recording media, magnetic storage fluids[4], sensors[5], pigments[6] and Photo catalysts[7]. The fine particles of mixed metal oxides possessing spinel structure and its property have been investigated by number of workers[8-13]. These properties depend upon the nature of ions and their site distribution amongst tetrahedral and octahedral sites.

In recent years, synthesis and application of transition metal doped Zn ferrite nanoparticles is becoming a subject of intense research because of their properties, and variety of applications[14,15]. The polycrystalline Cu-Zn ferrites could be considered as the most versatile ferrites, due to their high resistivity and low eddy current losses. Generally Cu-Zn ferrites were commercially used in radio frequency circuits, transformer cores, antennas and read/write heads for high-speed digital tape and in high quality filters[16-18].

These applications mainly depend upon microstructure and surface properties of the fine powders which depend upon the method of preparation[19]. Mixed metal oxides are prepared by various methods such as sol-gel[20], hydrothermal[21], citrate precursor[22], co-precipitation[23] etc. The method of preparation plays a very important role with regard to the chemical, structural and magnetic property of mixed spinel ferrites[24-27]. In this investigation in order to understand behavior of these ferrites, Co-Zn ferrites were prepared by sol-gel auto combustion method and were characterized by using FTIR, SEM, TGA/DTA, EDS, XRD and TEM.

EXPERIMENTAL

Reagent grade purity chemicals were used. Zinc nitrate, cobalt nitrate, iron nitrate and glycine were prepared in deionized water. In a beaker 0.25 M zinc nitrate was taken, drop wise 0.25 M cobalt nitrate was added to it with contineous stirring at 80^0 C followed by addition of 0.25 iron nitrate. Glycine was added to this mixture and P^H was adjusted to 9 by adding ammonia solution. This content was stirred and heated at 80^0 c for 3 hours to obtain gel. This gel was heated on hot plate to obtain the Co Zn ferrite powder.

X-ray diffraction (XRD) study of the synthesized product was carried out by using Philips model PW-3710 x-ray diffract meter with Cu k ά=1.54 Ao. Scanning electron microscopy (SEM) observation was performed with a JEOL JSM-6360 scanning electron microscope. Electron dispersive X-ray spectrometry (EDS) was used for elemental analysis of these nanoparticles. A Perkin-Elmer Spectrum100 Fourier transform infrared spectrometer (FTIR) was used for IR study of these nanoparticles. The TGA-DSC measurements were carried out using SDTQ600V20.9 Build 20 thermo gravimetric analyser. Particle size of these materials were confirmed by the Transmission electron microscope working at 100 kV (JEOL- JEM 100 SX microscope).

RESULTS AND DISCUSSION

XRD analysis

Figure 1 shows the XRD pattern of calcined sample at 950^0C for 6 hours. This pattern corresponds to peaks of (111), (220), (311), (400), (422), (511), (440), (533). These polycrystalline nanoparticles exhibit cubic spinel structure $ZnFe_2O_4$ (JCPDS 89-1012) and $CoFe_2O_4$ (JCPDS.22-1080) card. Lattice parameter of $Co_xZn_{1-x}Fe_2O_4$ with $x = 0.50$ was found to be 8.4437 A°. The average grain size of $Co_xZn_{1-x}Fe_2O_4$ with $x = 0.50$ is calculated by Debye-Scherrer equation to the half intensity width of (311) peak.

$$\mathbf{d = 0.9\,\lambda / \beta \cos\theta}$$

where λ is the wave length of Cu k ά=1.54A°, β is the calibrated half intensity width of the selected diffraction peak (degrees) and θ is the Bragg's angle (half of the peak position angle). From this equation the average size of $Co_xZn_{1-x}Fe_2O_4$ with x = 0.0, 0.25, 0.5 nanoparticle was estimated to be 28 to 32 nm.

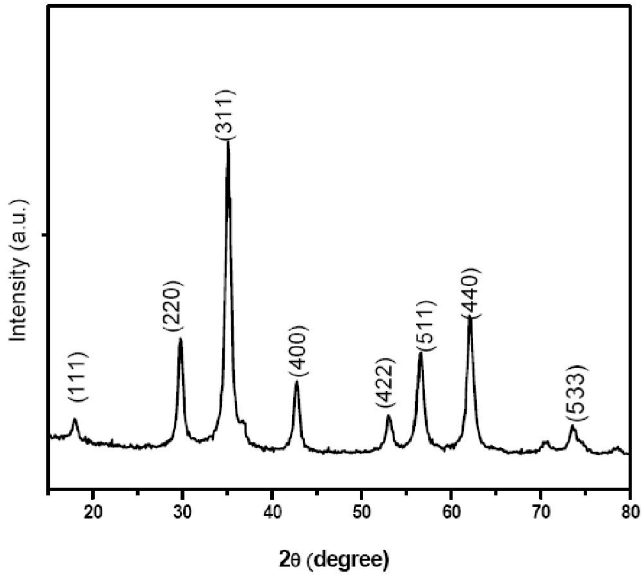

Figure 1 : XRD pattern of the Co Zn ferrite

TGA-DTA analysis

TGA- DTA analysis of these nanoparticles heated at the rate 10^0c / minute from 0 to 1000^0C, is given in figure 2. The DTA curve shows that thermal decomposition of $Co_xZn_{1-x}Fe_2O_4$ nanoparticles at 170^0C with an endothermic peak and this peak is ascribed due to thermal decomposition of residual water from gel. The dehydration also produces TGA weight loss in the region of 100 -200 ^{0} C may be due to autocatalytic oxidation reduction reactions of nitrates with glycine[28].

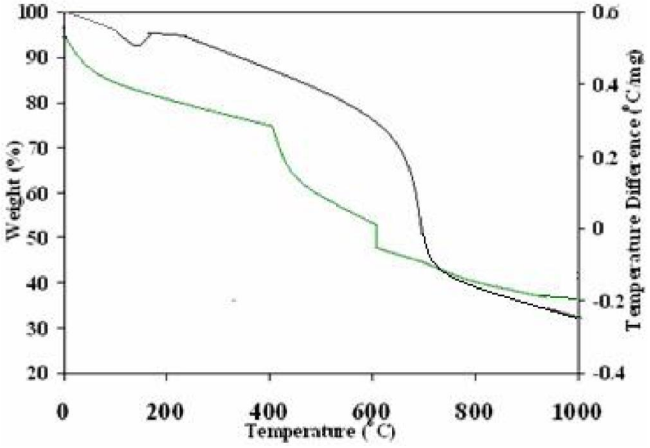

Figure 2 : TGA-DTA micrograph of as-prepared Co Zn ferrite

SEM and EDS analysis

The SEM micrographs of $Co_xZn_{1-x}Fe_2O_4$ synthesized by sol gel auto combustion method shows more interlinked interconnected beads (Figure 3). The elemental analysis of the sample was carried out by using energy dispersive X-ray spectrometer (EDS) and is shown in figure 4. The EDS result clearly shows Co and Zn doped in to ferrite crystal lattice. The Co and Zn doped into the ferrite crystal may be responsible for the observed morphology of the nanoparticle and it also minimizes the surface energy of these nanoparticles.

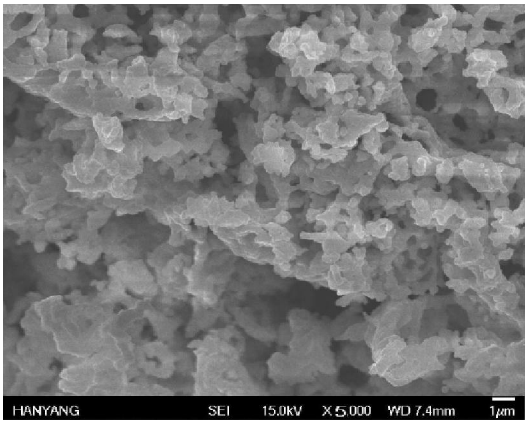

Figure 3 : SEM micrograph of Co Zn ferrite

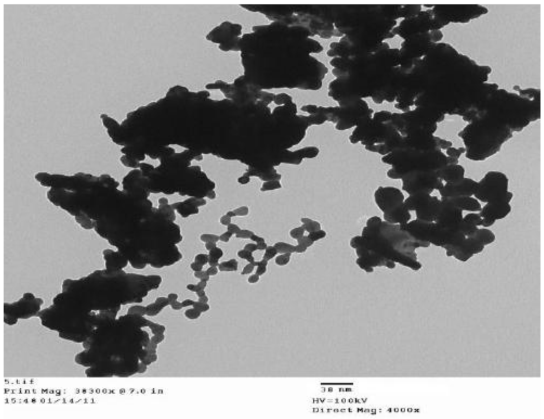

Figure 5 : TEM images Co Zn ferrite nanocrystals

CONCLUSIONS

A simple sol gel auto combustion process using glycine was employed to synthesize CoZn ferrite nanoparticles. Size of nanoparticles was found to be 28-32 nm and were formed by calcination at 950^{0}C for 6 hours. EDS analysis of the nanomaterial confirms that the material is composed of Co, Zn, O and Fe without any impurity. Particle size obtained from TEM analysis was found to be in good agreement with the value obtained from XRD pattern.

TEM and IR analysis

FTIR spectra of Co Zn ferrite nanoparticle is given in figure 5. It shows characteristic two strong absorption bands in the range of 600 to 450 cm^{-1} which are attributed to tetrahedral and octahedral site[29,30]. TEM image (Figure 6) of the Co Zn ferrite nanoparticle shows the agglomeration of these particles and have particle size 38 nm which is in agreement with the value obtained from XRD pattern.

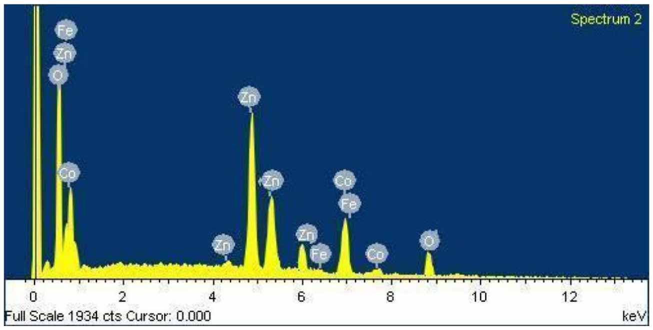

Figure 4 : EDS analysis of elements detected in the sample

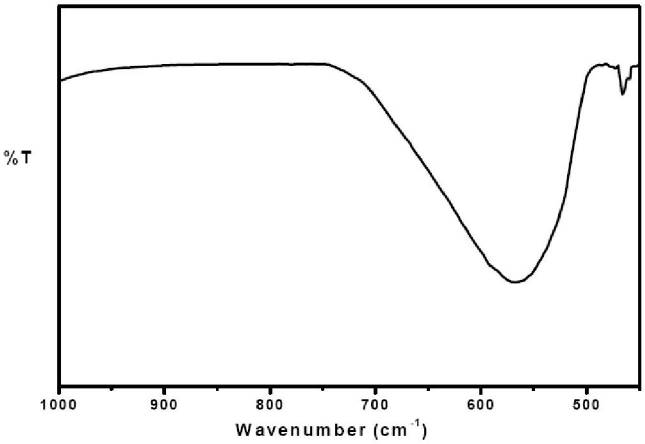

Figure 6 : FT-IR spectra of Co Zn ferrite

REFERENCES

[1] J.L.Snoek; 'New Developments of Ferromagnetic Materials', Elsevier Publisher Company, New York, **(1947)**.

[2] A.J.Fatiadi; Synthesis, 133 **(1976)**.

[3] G.Cainelli, G.Cardillo; Chromium Oxidation in Organic Chem., Springer Verlag, Berlin **(1984)**.

[4] J.I.Kroschwitz; Forth Ed. Kirk Othmer, Encyclopedia of Tech., Wiley Interscience Publications, New York, **4**, **(1992)**.

[5] J.Muzart; Bull.Soc.Chim.Fr., 65 **(1986)**.

[6] H.Van Bekkum; In: F.W.Lichtenthaler, (Ed); Carbohydrates as Org.Raw Mater., VCH, Weinheim, **(1991)**.

[7] S.Xu, W.Shangguan, J.Yuan, J.Shi; Chem.Sci.and Tech.of Advan.Mater., **8**, 40 **(2007)**.

[8] N.Okasha; J.Mater.Sci., **43**, 4192 **(2008)**.

[9] M.Banergee, N.Verma, R.Prasad; J.Mater.Sci., **42**, 1833 **(2007)**.

[10] M.R.Barati; J.Sol-Gel Sci.Tech., **52**, 171 **(2009)**.

[11] E.Melagiriyappa, H.S.Jayanna; J.of Alloy and Comp., **482**, 147 **(2009)**.

[12] T.Mathew, B.S.Rao, C.S.Gopinath; J.Catal., **210**, 405 **(2002)**.

[13] I.Z.Rahman, T.T.Ahmed; J.Magn.Magn.Mater., **290**, 1576 **(2005)**.

[14] X.Wei Qi, J.Zhou, Z.Yue, Z.L.Gui, L.T.Li; J.Magn.Magn.Mater., **251**, 316 **(2002)**.

[15] A.C.F.M.Costa, E.Tortella, M.R.Morelli, R.H.G.A.Kiminami; J.Magn.Magn.Mater., **256**, 174 **(2003)**.

[16] H.A.Dawoud, S.K.Shaat; The Isla.Univ.Jour., (Series of Nat.Studies and Engg.), **14**, 165 **(2006)**.

[17] J.Kulikowski; J.Magn.Mater., **41**, 56 **(1984)**.

[18] P.Ravindranathan, K.C.Patil; Mater.Sci., **22**, **(1987)**.

[19] B.K.Das; Preparation and Characterization of Mater., J.M.Honing, C.N.R.Rao, (Eds); New York, Academic Press, **(1981)**.

[20] S.Sunder, B.K.Srivastava, D.Krishnamurthy; Ind.J. Pure and Appl.Phys., **42**, 366 **(2004)**.

[21] T.Pannaparagil, S.Komarneni; IEEE Trans.Mag., **15**, 4233 **(1989)**.

[22] N.S.Gajbhiye, U.Bhattacharya, V.S.Darshane; Thermochim.Acta, **264**, 219 **(1995)**.

[23] B.A.Mulla, V.S.Darshane; Ind.J.Chem., **22A**, 143 **(1983)**.

[24] E.Wolska, W.Wolski, J.Kaczmarek, E.Riedel, D.Prick; Solid State Ionics, **51**, 231 **(1992)**.

[25] Idem, ibid., **101**, 1069 **(1997)**.

[26] M.A.Amer, O.M.Hamada; Hyperfine Interact., **96**, 99 **(1995)**.

[27] D.Ravinder, T.Alivelu; Mag.Mater.Lett., **41**, 254 **(1999)**.

[28] N.S.Gajbhiye, U.Bhattacharyya, V.S.Darshane; Thermochim.Acta, **264**, 219 **(1995)**.

[29] A.M.Shaikh, S.A.Jadhav, S.C.Watawe; Mater.Lett., **44**, 192 **(2000)**.

[30] H.M.Kazi, S.F.Mansour; J.Phys.Chem.Solids, **67**, 1643 **(2006)**.