

Preparation and Characterization of Cu-Zn ferrites by Combustion method

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Abstract

Cu-Zn ferrite nanoparticles have been synthesized by Combustion method under ambient /temperature..atmosphere. The phase of the prepared nanopowder was confirmed using X-ray diffraction (XRD). The surface morphology was examined by Scanning Electron Microscope (SEM) and Vibrating Sample Magnetometry (VSM) was used to investigate the magnetic behaviour of Cu-Zn ferrite nanoparticles at low temperature. Fourier Transform Infrared Spectroscopy (FT-IR) yields the occurrence of the absorption bands. The interesting results of the synthesized specimen are reported in the manuscript.

Keywords: Ferrites, Cu-Zn ferrites, XRD, SEM, VSM, FTIR.

1. Introduction

Advancements in scientific technology have been focused more attention on mixed ferrite systems at nano-dimension. Spinel ferrites are significant because of their extraordinary electrical and magnetic properties and potential applications in storage systems, magnetic cores, sensors, microwave absorbers and high frequency devices [1-4]. The chemical formula of mixed spinel structure of soft Cu-Zn ferrites is of the form $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$, where the cation Zn^{2+} occupies the tetrahedral sublattices, whereas cation Cu^{2+} occupies the octahedral sublattices and Fe^{3+} is shared to both octahedral and tetrahedral sublattices [5-7]. Crystalline Cu-Zn spinel ferrites have been investigated

because of their applications in non-resonant devices, radio frequency circuits, read/write heads for high speed digital tapes and microwave devices [8]. The synthesis of ferrites by conventional techniques always has limitations with parameters such as experimentation period, high temperature, grain size and pH of the solution. The properties of ferrites are sensibly tailored by suitable composition, sintering temperature and sintering time [9-10]. The experimental methods like double sintering ceramic technique [11], flash combustion technique [12], solgel technique [13], electrospinning method [14], sonochemical method and microemulsion technique [15] are various methods available for the synthesis of ferrite nanoparticles. Combustion technique has advantages compared to other techniques such as rapidly producing fine and homogeneous nanoparticles by consuming less time and energy. In combustion synthesis, an exothermic reaction begins at the ignition temperature and generates a certain amount of heat that is manifested in the maximum temperature [16].

This manuscript elucidates the synthesis of $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles via combustion technique and their characterization using powder X-ray diffraction, Fourier transform infrared spectroscopy, Scanning electron microscopy and temperature dependent magnetic behavior at low temperatures.

2. SAMPLE PREPARATION

Analytical grade Zinc nitrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], Copper nitrate [$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$], Iron nitrate [$\text{Fe}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$] and Citric Acid [$\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$] were used to prepare $(\text{Cu}_{0.5}\text{Zn}_{0.5})\text{Fe}_2\text{O}_4$ combustion method. Metal nitrates and citric acid were dissolved in deionized water with 1:1 molar ratio of nitrates to citric acid and well mixed. The mixture was then subjected to the thermal agitation at 650°C . The metal nitrates served as oxidizers and citric acid served as a fuel. The precursors immediately suffered to the oxidization in due process under thermal environment. The pristine powder was granulated and sintered at 900°C for 4 hours under air atmosphere. The sintered powder was subjected to various analyzes to understand the phase identification, lattice parameter determination, surface morphology and magnetic behavior.

3. RESULTS AND DISCUSSION

3.1 Powder X-ray Diffraction Analysis

The phase formation and crystallinity of the sample were identified using BRUKER DRX500 X-ray diffractometer with CuK_α radiation of wavelength 1.5406\AA over the angular range $10\text{-}70^\circ$ by scanning at the step of 0.04° per minute. Fig.1 shows the powder X-ray diffraction pattern of $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles. The obtained pattern was indexed using JCPDS Card No. 897409 and reveals the fact that the synthesized sample is of face centered cubic structure with space group $\text{Fd}\bar{3}\text{m}$. All the crystal planes are indexed with miller indices (111), (220), (311), (222), (400), (422), (511), and (440) without additional peaks.

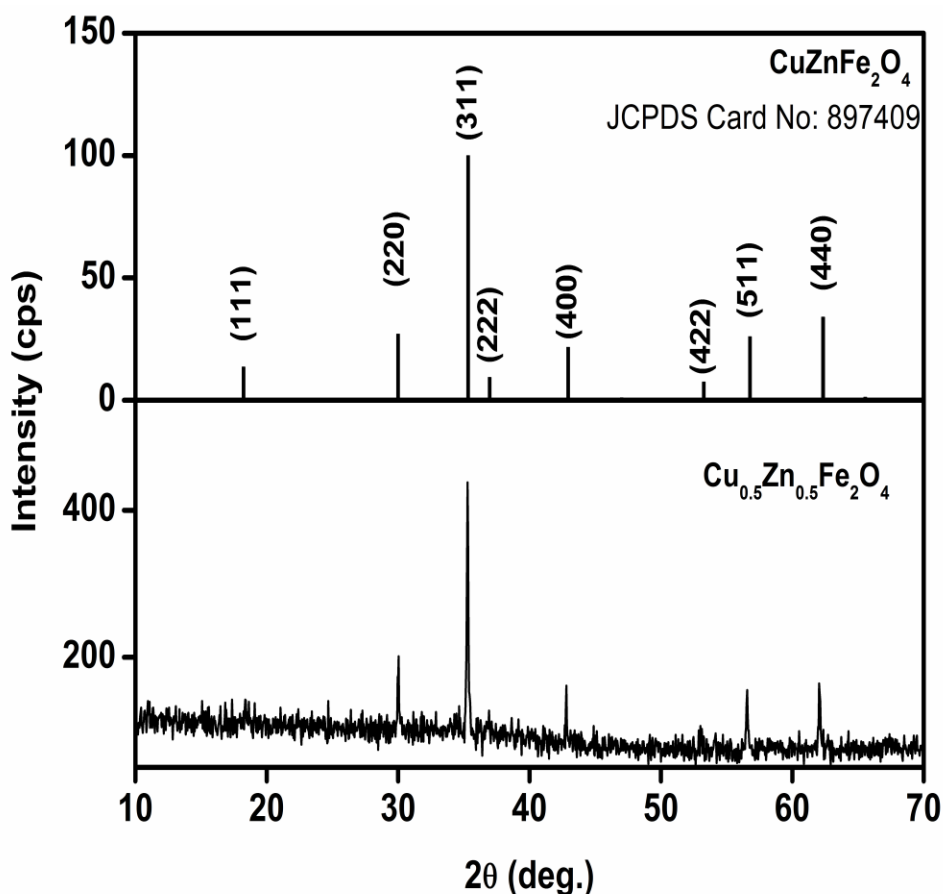


Fig.1: Powder X-ray diffraction pattern of $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles

The calculated lattice parameter is $a=8.419\text{\AA}$ and the crystallite size was estimated using Scherrer formula,

$$D = \frac{k\lambda}{\beta \cos \theta}$$

Where, D is the crystallite size, k is the geometry factor, λ is the wavelength used, β is the Full width at half maximum and θ is the angle of diffraction. The estimation provided that the synthesized Cu-Zn nanoparticles possessing the crystallite size 29 nm. The internal strain (ϵ) was estimated using Williamson-Hall equation $\epsilon = \frac{\beta_{hkl}}{\tan\theta}$ [17] as 0.0182.

3.2 Fourier Transform Infrared Spectroscopy

The synthesized specimen was subjected to FTIR analysis to confirm the formation of spinel structure. The FTIR measurement ranges from the frequency 400-4000 cm^{-1} . Fig.2 shows the FTIR spectrum of Cu-Zn ferrite nanoparticles. The transmittance spectrum exemplifies that the prominent bands occur at the positions 3693.62, 2985.37, 1525.53, 1362.85, 986.10 cm^{-1} and 563.63 cm^{-1} . The absorption bands occur at around 400-500 cm^{-1} are related to spinel cubic structure. The band at 563.63 cm^{-1} is assigned to the vibrations between the tetrahedral metal ion and the oxygen ion ($M_{\text{tetra}}\text{-O}$) and the position at around 450 cm^{-1} is assigned to the vibrations of the bond between octahedral metal ion and oxygen ion ($M_{\text{octa}}\text{-O}$).

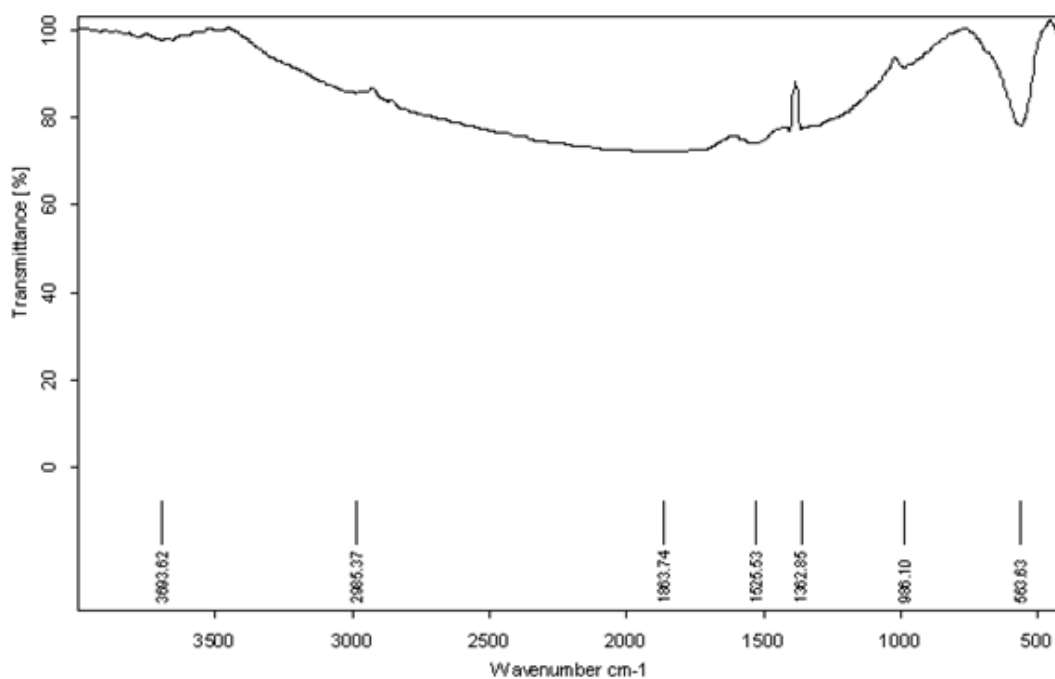


Fig.2: FT-IR spectrum of Cu_{0.5}Zn_{0.5}Fe₂O₄ nanoparticles

3.3 Morphology analysis

Fig.3 shows the surface morphology of $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles obtained with the help of scanning electron microscope. The SEM micrographs show the different morphology with well grain boundaries and aggregation of nanoparticles which is attributed to the Vander Waals force of attraction among nanoparticles. The SEM results to the average particle size of 80 nm.

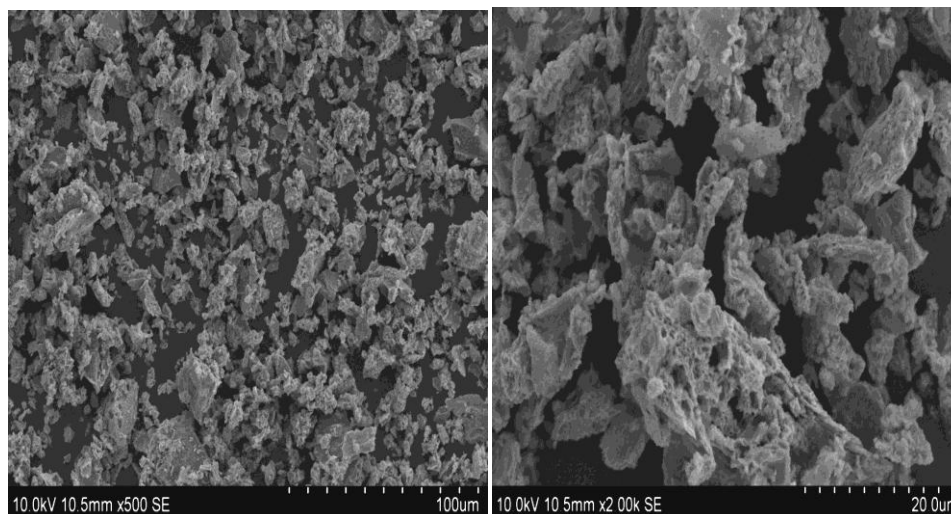


Fig.4: SEM images of $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles

3.4 Vibrating Sample Magnetometry

Vibrating sample magnetometry analysis was used to study the magnetic response of the sample at low temperatures and the change in magnetic moment per mass with temperature is shown in figure 3. The temperature was varied from 19 K to 300 K and the magnetic field was kept constant (1 tesla) for the measurement. The decrease of magnetic moments at low temperatures is due to the inter-particle interactions among the superspins of nanoparticles [18]. The magnetic moment was observed at 300 K as 29 emu/g.

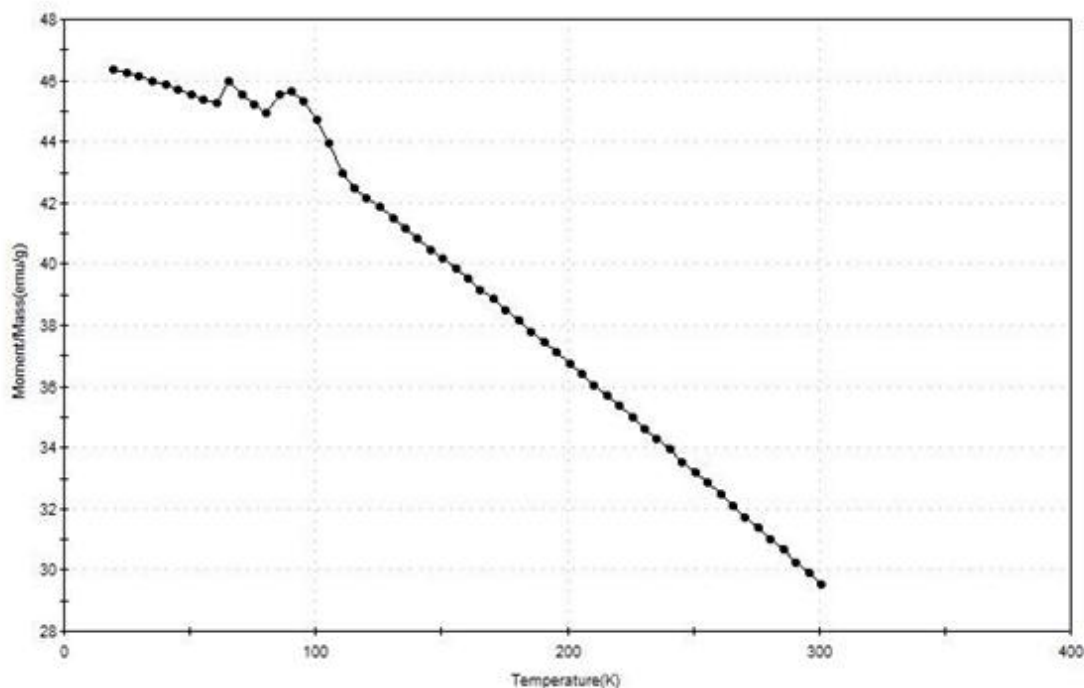


Fig.3: Temperature dependent Magnetic behavior of $\text{Cu}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles

4. Conclusions

Cu-Zn ferrite nanoparticles were successfully synthesized by Combustion method under ambient atmosphere. The synthesized sample has FCC structure and acquires crystallite size of 29 nm. The characteristic bondings were observed by FTIR and the surface morphology reveals the agglomeration of particles with average particle size of 80 nm. The temperature dependent magnetic behavior yields that the magnetic moment is 29 emu/g at 300K.

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References

- [1] Gholizadeh, A and Jafari, E, Effects of sintering atmosphere and temperature on structural and magnetic properties of Ni-Cu-Zn ferrite nano-particles: Magnetic enhancement by a reducing atmosphere, *Journal of Magnetism and Magnetic Materials*, 422, (2017), pp.328-336.
- [2] Mürbe, J and Töpfer, J, Low temperature sintering of sub-stoichiometric Ni-Cu-Zn ferrites: Shrinkage, microstructure and permeability, *Journal of Magnetism and Magnetic Materials*, 324, (2012), pp.578-583.
- [3] Caltun, O.F *et al.*, Study of the microstructure and of the permeability spectra of Ni-Zn-Cu ferrites, *Journal of Magnetism and Magnetic Materials*, 242-245, (2002), pp.160-162.
- [4] LUO, G.S *et al.*, Effect of Cu ion substitution on structural and dielectric properties of Ni-Zn ferrites, *Transactions of Nonferrous Metals Society of China*, 25, (2015), pp. 3678-3684.
- [5] Pradhan, S.K *et al.*, Microstructure characterization and cation distribution of nanocrystalline magnesium ferrite prepared by ball milling, *Materials Chemistry and Physics*, 93, (2005), pp. 224–230.
- [6] Tan, M *et al.*, Overlapping large polaron tunneling conductivity and giant dielectric constant in $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_{1.5}\text{Cr}_{0.5}\text{O}_4$ nanoparticles (NPs), *Journal of Alloys and Compounds*, 509, (2011), pp.9399-9405.
- [7] Kurian , M and Divya, S. Nair, Effect of preparation conditions on Nickel Zinc Ferrite nanoparticles: A comparison between sol-gel auto combustion and co-precipitation methods, *Journal of Saudi Chemical Society*, 20, (2016), pp. S517-S522.
- [8] Lamani, A.R *et al.*, Microcrystalline parameters of Cu-Zn ferrites using X-ray line profile analysis, *Indian Journal of Pure & Applied Physics*, 47, (2009), pp.715-718.
- [9] Verma, A *et al.*, Dielectric properties of Ni-Zn ferrites prepared by the citrate precursor method, *Materials Science and Engineering B*, 60, (1999), pp. 156-162.
- [10] Rao B.P and Rao P.S.V.S, X-ray and magnetic studies of scandium substituted Ni-Zn ferrites, *IEEE Transactions on Magnetics*, 33, (1997), pp. 4454-4458.

- [11] Jadhav S.A, Structural and magnetic properties of Zn substituted Li-Cu ferrites, *Materials Chemistry and Physics*, 65, (2000), pp. 120-123.
- [12] Mangalaraja, R.V *et al.*, Initial permeability studies of Ni-Zn ferrites prepared by flash combustion technique, *Materials Science and Engineering A*, 355, (2003), pp. 320-324.
- [13] Isfahani, M.J.N *et al.*, Magnetic behavior of nickel-bismuth ferrite synthesized by a combined sol-gel/thermal method, *Ceramics International*, 39, (2013), pp. 1163-1167.
- [14] Xiang, J *et al.*, One-dimensional NiCuZn ferrite nanostructures: Fabrication, structure and magnetic properties, *Journal of Solid State Chemistry*, 183, (2010), pp. 1239-1244.
- [15] Das, N *et al.*, Nanosized bismuth ferrite powder prepared through sonochemical and microemulsion technique, *Materials Letters*, 61, (2007), pp. 2100-2104.
- [16] Kopp Alves, A *et al.*, Novel Synthesis and Characterization of Nanostructured Materials, *Engineering Materials*, DOI: 10.1007/978-3-642-41275-2-2, Springer-Verlag Berlin Heidelberg, (2013).
- [17] Anupama, M.K. *et al.*, Investigation of impedance response and dielectric relaxation of Ni-Zn ferrites prepared by self-combustion technique, *Journal of Alloys and Compounds*, (2017), Accepted Manuscript, DOI: 10.1016/j.jallcom.2017.02.241.
- [18] Antic, B *et al.*, An integrated study of thermal treatment effects on the microstructure and magnetic properties of Zn–ferrite nanoparticles, *Journal of Physics: Condensed Matter*, 25, (2013), 086001 (13pp)