Structural and electrical properties of copper ferrite 
(CuFe$_2$O$_4$) NPs

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Abstract. Spinel ferrites are the important class of soft magnetic material composed of iron
oxide and metal oxide as their main compound. Copper ferrite (CuFe$_2$O$_4$) is one such spinel
ferrite with great potential technological applications. It is well-known about ‘Jahn-Teller
distortion’ that the copper ferro-spinel are at room temperature has a co-operative distortion
to the tetragonal site-symmetry which depends only upon the copper ion distribution. The
structural and electrical properties of Copper ferrite were investigated by X-ray diffraction and
DC-resistivity studies. The room temperature XRD data revealed that the formation of
tetragonal structure of (CuFe$_2$O$_4$) NPs. The crystallite size (t) nm was obtained from Debye-
Sherrer’s formula indicated that the (CuFe$_2$O$_4$) NPs shows nanocrystalline nature with
crystallite size 14 nm. The lattice constant (a) was found to be in the reported range (~8.380
Å). The DC-resistivity parameters were drawn from the two point probe device. An activation
energy $\Delta E$ of the copper ferrite nanoparticles was found to be 0.01 eV.

1. Introduction

The spinel ferrites are characterized by the formula AFe$_2$O$_4$ (Where, A is a divalent metal ions like Co,
Ni, Zn, Cd, Mg, Cu etc. and B is a trivalent Fe$^{3+}$ ions) [1] and their crystal structure belongs to the
space group fd$_3$mOh7 [2]. Since the last several decades, good quality and upgrade work on the
investigation of structural, electrical and magnetic properties of spinel ferrites has been carried out by
several workers [3]. Cubic spinel structured ferrite nanoparticles have facilitated new advances in the
field of nanoscience and nanotechnology. Nanoparticles of this spinel ferrite have been creating
novelty in their physical properties, which makes them more advanced and useful in this technological
world [4]. As such new materials evolving with the novel structural, electrical, magnetic and dielectric properties has great importance in the scientific application of ferrite materials in various fields [5]. Some of the unoptional applications include gas sensors, catalysis, magnetic resonance imaging (MRI) [6], magnetic recording media [7], drug delivery systems [8], ferro-fluids [9], microwave devices [7], antenna rods [10], storage devices [11], water purification etc. The synthesis conditions and preparative methods of ferrite nanoparticles play a crucial role to achieve this place in the current scenario. In general well-known synthesis methods were employed for the fabrication of such ferrite materials viz. conventional ceramic technique [12], sol-gel method [13], hydrothermal method [14], micro-emulsion [15], auto-clave [16], chemical synthesis method [17], ball milling technic [18] by many researchers. Here, we are fascinated with the demand of the various industries having futuristic view towards the physical properties of the ferrite nanoparticles. To encounter this, we have selected one of the wet-chemical methods i.e. sol-gel auto-combustion for the fabrication of Copper ferrite (CuFe$_2$O$_4$) Nps [Cu$_{1-x}$Fe$_{2+x}$O$_4$ ($x=0.04$)]. Snock and Bertaut [19] in their literature has reported that the CuFe$_2$O$_4$ has a cubic spinel structure above 750° C and having less than 1 axial ratio (c/a) below this temperature representing tetragonal structure [20]. Aiming to the structural and electrical parameter studies we have synthesised and characterised the CuFe$_2$O$_4$ Nps and reported the findings of the work.

2. Experimental

2.1 Raw Materials

For the present investigation, we have applied sol-gel auto-combustion method. So, inorder to compete the circumstances of wet-chemical reactions in sol-gel auto-combustion, we have selected AR grade copper nitrate [Cu(NO$_3$)$_2$.6H$_2$O], ferric nitrate (Fe(NO$_3$)$_3$.9H$_2$O) and citric acid (C$_6$H$_8$O$_7$) [21] were used for the synthesis. Citric acid was used as a fuel/chelating agent. Ammonia (NH$_3$) was used for maintaining pH of the solution during the synthesis.

2.2 Synthesis Method

Initially, we added specific amount of 99.9% pure AR grade granule form of copper nitrate in to a deionised water and obtained a clear solution of [Cu(NO$_3$)$_2$.6H$_2$O] named as solution1. Next to this, we add granule form of ferric nitrate (Fe(NO$_3$)$_3$.9H$_2$O) in to another beaker with a deionised water and solution 2 is created. Similarly, citric acid solution was obtained as a solution 3. The molar ratio of nitrates to citric acid was taken as 1:3. Thereafter, we mixed all these three solution in one beaker and mix them all with the help of stirrer. Then we mount this mixed solution on a hot plate magnetic stirrer for the evaporation of excess water. We adjusted the stirring temperature to 80° C. During the stirring and heating ammonia was also slowly added to the sol to adjust the pH value at about 8. When most of the water gets evaporated the solution turns to viscous gel which then continued heating and stirring till it gets nearly dry gel formed. This makes us increase the temperature at the 110° C to push the sol-gel reaction to the next level. At a critical temperature the dry gel of mixed metal-nitrate-citrate solution get ignited and starts burning in a self-propagating manner, which gives us loose powder of (CuFe$_2$O$_4$) Nps. This powder was grinded and annealed and used for the characterization purpose for the studies.

3. Characterization

3.1 X-Ray Diffraction

X-ray diffraction pattern of Copper ferrite (CuFe$_2$O$_4$) Nps was taken by using Cu-kt radiation (λ = 1.5405 Å) on Philips PW-1730 X-ray diffractometer. The X-ray diffractometry was performed in a room temperature. The scanning rate of 0.02 deg/s was kept and the major Bragg’s reflections of CuFe$_2$O$_4$ Nps were recorded in the 20 range of 20° to 80° for the analysis of structural parameters.
3.2 Electrical property

The DC-electrical properties of the prepared copper ferrite (CuFe₂O₄) Nps were measured in the temperature range 300 - 800 K by using home made two-probe technique device.

4. Results and discussion

4.1 Structural Properties

The prepared Copper ferrite (CuFe₂O₄) Nps samples were studied for a structural characterization by X-ray diffractrometry. The XRD patterns show the reflections (220), (311), (222), (400), (422), (511) and (440). No extra peak was observed in the XRD pattern. Phase purity, crystallite size (t) of the particles and lattice constant (a) etc. various structural parameters were calculated using XRD data on employing the related formula.

4.1.1 Lattice constant (a)

The lattice constant (a) of (CuFe₂O₄) Nps was calculated by was determined from XRD data using the relation.[22]

\[ a = d \sqrt{N} \quad (1) \]

Where, \( a \) is lattice constant, ‘d’ is inter planar spacing and \( N = (h^2+k^2+l^2) \); (h k l) are Miller indices.

The lattice constant (a) of (CuFe₂O₄) Nps samples was obtained to be 8.380 Å.

4.1.2 Crystallite size (t)

For calculating the crystallite size of (CuFe₂O₄) Nps, (311) peak in the XRD pattern is found to be most intense amongst the other existing peaks. This peak was used to determine the crystallite size (t) of the samples. The crystallite size (t) of (CuFe₂O₄) Nps was calculated by using Debye-Scherrer method, which is mentioned by eq. [23];

\[ t = \frac{K \lambda}{\beta \cos \theta} \quad (2) \]

Where, (t) is a crystallite size (nm), \( \lambda \) is a wavelength of X-ray (\( \lambda=1.5405 \) Å), \( \beta \) is broadening of peak at diffraction angle \( \theta \). The crystallite size (t) of copper ferrite (CuFe₂O₄) Nps samples was found to be 14 nm, which is confirming the nanocrystalline nature.

4.1.3 X-ray density (d_x)

The X-ray density (\( d_x \)) was calculated from lattice constant by the following relation [22]. The X-ray density for (CuFe₂O₄) Nps was obtained as 5.39 (g/cm³). The values of crystallite size lattice constant (a), unit cell volume (V) and X-ray density (d_x) are listed in table 1.

\[ dx = \frac{8M}{N_A a^2} \quad (3) \]

Where, M is molecular weight and \( N_A \) is the Avogadro's number, \( a \) is lattice constant.

| Table 1. | lattice parameter \( a \)(Å), X-ray density \( d_x \)(g/cm³), crystallite size \( t \)(nm) and volume \( V \)(Å³) of (CuFe₂O₄) Nps |
|-----------|------------------|-----------------|-----------------|-------------------|
| (CuFe₂O₄) Nps | \( a \)(Å) | \( d_x \)(g/cm³) | \( t \)(nm) | \( V \)(Å³) |
| 8.380 | 5.39 | 14 | 293.70 |
4.2 DC-resistivity

Electrical properties of spinel ferrites have been the subject of interest many researchers since the artificial production of spinels by Snoek [24]. DC electrical resistivity was measured by home made two point probe device. The samples of (CuFe₂O₄) Nps were used in the form of pellets of 10 mm diameter and of 3 mm thickness. The pellets were prepared at room temperature by compressing at 6 tons in a hydraulic press. For making the smooth surface of the pellets zero polish paper was used. After smoothening of the surfaces silver paste is applied on it for good ohmic contact. The DC-resistivity was measured in the 300 K- 800 K range. The resistivity of the sample was calculated using the relation [25];

$$\rho (dc) = \frac{R_0 r^2}{t}$$  \hspace{1cm} (4)

where, R is the ohmic resistance of the sample, r is the radius of the sample in meter.

| (CuFe₂O₄) Nps | ΔE (eV) | R (K) (at 300 K) | ρ (Ω cm) (at 300 K) |
|---------------|--------|----------------|------------------|
|               | 0.01   | 1038.96        | 0.7153           |

The plots of log ρ versus 1000/T were plotted and the resultant activation energies for conduction were computed in table 2. This resistivity is calculated by the relation given by [26];

$$\rho = \rho_0 \exp\left(\frac{\Delta E}{KT}\right)$$  \hspace{1cm} (5)

The resistivity ρ of (CuFe₂O₄) Nps at 300 K was reported as 0.7153 (Ω cm). The value of activation energy ΔE which is reported as 0.1 Ev.

5. Conclusions

We have successfully prepared nanocrystalline (CuFe₂O₄) Nps by employing sol-gel auto-combustion method using citric acid with the molar ratio 1:3. The crystallite size (t) of the prepared (CuFe₂O₄) Nps was recorded as 14 nm. The structural parameters were derived from XRD data and the values of (CuFe₂O₄) Nps are in good agreement with the reported literature. The prepared (CuFe₂O₄) Nps belongs to the distorted, tetragonal phase below certain temperature above which it starts showing cubic spinel structure. Bragg’s angle (2θ) reflections are in very good agreement with the reported literature. X-ray density ($d_\chi$) of CuFe₂O₄ Nps was obtained as 5.39 (g/cm³). The lattice constant α was reported as 8.380 (Å) for CuFe₂O₄ Nps. The resistance (R) was calculated at 300 K as 1038.96 Ω and resistivity ρ at 300K is 0.7153 Ω cm. We have concluded that the (CuFe₂O₄) Nps is very useful for the technological advancement in the coming future to be studied.

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