Ceramic Synthesis and X-ray Diffraction Characterization of Copper Ferrite

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Abstract: Copper ferrite (CuFe$_2$O$_4$) particles have been synthesized by the standard ceramic technique. The prepared copper ferrite particles were sintered at 800 °C for 12h. The pre-sintered sample is again reground in fine powder and pelletized using a hydraulic press. These pallets were finally sintered at 1050 °C for 12 h and the structural properties of copper ferrite were investigated and reported in this work. The X-ray diffraction analysis revealed that synthesized copper ferrite particles possess a single phase cubic spinel structure. A keen observation of the X-ray diffraction pattern showed the average crystallite size (t) of the copper ferrite obtained from Scherrer’s formula as 140 nm. The lattice constant (a) and other structural parameters are in the reported range.

INTRODUCTION

Ferrites are the iron oxide mixed with the transition metal ions, which are technologically important magnetic materials from the last several decades [1]. According to L. Neel, ferrites are the ferromagnetic oxides [2]. Ferrite materials have unique electrical and magnetic properties best suited for high-frequency applications [3]. They are used in magnetic ink [4] and magnetic fluids [5] and for the fabrication of magnetic core of reading and write heads of high-speed digital tapes [6]. The important structural, electrical, and magnetic properties of ferrites arise from the distribution of cation over the available (A)-site and [B]-site [7]. The factors like preparation method and preparative condition play an important role in governing the properties of ferrite. They are classified into three groups namely spinel ferrite [8], garnets [9] and hexaferrite [10]. Among these three types of ferrites, “spinel ferrites” are an important class of magnetic materials and are widely used in many technological aspects [11]. The crystal structure of the spinel ferrite belongs to a “cubic” spinel with space group $Fd\bar{3}m$ [12]. The crystal structure possesses two sub-lattices namely tetrahedral (A) and octahedral [B] in which cations of different ionic radii can accommodate [13]. The variation in the physical properties of spinel ferrite can be brought by either change in method of preparation or selecting appropriate cation on large scale [14]. Various physical and chemical methods have been employed to obtain crystalline spinel ferrite [15]. Ceramic technology has its own advantage over the other synthesis methods as the high sintering temperature the density of the sample increases which is useful in some application [16]. The stoichiometric and homogeneity of the samples is maintained in the ceramic method. Many efforts have been made to improve the basic properties of ferrite by the synthesis conditions also important in governing the properties of ferrites and are rarely studied [17].
CERAMIC METHOD

The most popular and commonly used method of preparation is the solid-state reaction/ceramic method [18]. In the ceramic method, very pure and fine grain constituents in oxide forms are taken as raw material for the synthesis. A poly-crystalline sample of copper ferrite (CuFe$_2$O$_4$) was prepared by the standard ceramic method. The Analytical Reagent (AR) grade oxides (CuO) and (Fe$_2$O$_3$) were mixed as per stoichiometric proportions. Then they are thoroughly, uniformly mixed, and ground for 3 to 4 h using agate mortar pestle. This mixture is sintered at a specific temperature to facilitate solid-state chemical reaction among the oxides. Pre-sintering of the samples can be done at about lower temperature 800 °C for 12 h and final sintering of the sample can be done at much higher temperature 1050 °C for 12 h depending on the constituents of the compound and then slowly cooled to room temperature.

CHARACTERIZATION TECHNIQUES

The XRD patterns of CuFe$_2$O$_4$ were recorded in the 20 range of 20 - 80° using Cu-ka ($\lambda = 1.5418$ Å) radiation source BRUKER axs D8 ADVANCE X-ray diffractometer; characterized at room temperature. The entire X-ray diffraction pattern was set with the scanning rate of 0.02 deg/s and the major Bragg’s reflections were recorded for the CuFe$_2$O$_4$ analysis of structural parameters.

Figure 1. The steps followed in the synthesis of CuFe$_2$O$_4$ via the ceramic method
RESULTS AND DISCUSSION

The XRD patterns show the reflections (220), (311), (222), (400), (422), (511) and (440) well match with JCPDS no. 039-1346 [19]. All these reflections belong to the cubic spinel structure [20]. No extra peak other than cubic spinel was observed in the XRD pattern.

Lattice constant (a) of CuFe$_2$O$_4$

The calculated lattice constant (a) was calculated using the following [21];

$$ a = d_{hkl} \sqrt{h^2 + k^2 + l^2} \tag{1} $$

Where $d_{hkl}$ the interplanar spacing of two planes, (a) is the lattice constant, and (hkl) is the miller indices of each plane. The lattice parameters of CuFe$_2$O$_4$ calculated from the XRD data for the spinel structure is $a = 8.380$ Å which is in good agreement with the literature value. The values of lattice constant for CuFe$_2$O$_4$ sample determined using the above relation are summarized in Table 1.

<table>
<thead>
<tr>
<th>NiFe$_2$O$_4$ NPs</th>
<th>a (Å)</th>
<th>t (nm)</th>
<th>$d_X$ (g/cm$^3$)</th>
<th>$d_B$ (g/cm$^3$)</th>
<th>P (%)</th>
<th>V (Å$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuFe$_2$O$_4$</td>
<td>8.380</td>
<td>140</td>
<td>5.403</td>
<td>3.55</td>
<td>34.26</td>
<td>588.48</td>
</tr>
</tbody>
</table>

The crystallite size of the prepared samples (CuFe$_2$O$_4$) was obtained by using Scherer’s formula [22].

$$ t = \frac{0.9\lambda}{\beta \cos \theta} \tag{2} $$
Where \( \lambda \) is the X-ray wavelength, \( \beta \) is the full width at half maximum, \( \theta \) is the Bragg’s angle, and \( k = 0.89 \). The average crystallite size \( (d) \) obtained from XRD data is 140 nm. The (311) reflection in the XRD pattern is found to be most intense amongst the other existing peaks and is used to determine the crystallite size of the samples. The unit cell volume \( (V) \) was determined from the values of lattice constants \( (V = a^3) \) for the \( \text{CuFe}_2\text{O}_4 \) sample. The X-ray density was calculated using the following relation and their values are given by formula [23];

\[
dx = 8M / N_A \alpha^3
\]  

(3)

Where, \( M \) is molecular weight and \( N_A \) is the Avogadro's number, \( \alpha \) is lattice constant. The bulk density was measured using Archimedes principle and values are presented in Table 1. The percentage porosity \( (P) \) was calculated using the values of X-Ray density and bulk density for the \( \text{CuFe}_2\text{O}_4 \) sample. The values of porosity are listed in Table 1 it is also in good agreement.

**CONCLUSIONS**

In this article, we have reported the successful preparation of \( \text{CuFe}_2\text{O}_4 \) using the ceramic method. The ceramic synthesis of the copper ferrite \( \text{CuFe}_2\text{O}_4 \) was found to be better than the other methods as it is very simple and releases less toxic gasses in an environment in comparison with the chemical reactions as well. The X-ray diffraction study confirms the formation of single-phase materials with a particle size of 140 nm. Lattice constant \( \alpha = 8.380 \) which is in good agreement with the reported literature. In the \( \text{CuFe}_2\text{O}_4 \) synthesized via the ceramic method, porosity was recorded to be 34.26%, which can be further considered for the gas sensing applications of magnetic ferrites.

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**REFERENCE**

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