Low temperature synthesis and investigations of magnetic properties of cobalt ferrite nanoparticles

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Abstract. In the present study we report the synthesis of cobalt ferrite nanoparticles using one of the well-known wet chemical method i.e. sol-gel auto combustion technique. The synthesis was carried out at sufficiently low temperature of 100°C. Citric acid was used as a fuel in the synthesis process. The obtained nanoparticles were sintered at 550°C for 4 h and then used for structural and magnetic investigations. The phase pure nature and nano crystalline nature was investigated through X-ray diffraction technique. Room temperature X-ray diffraction pattern show well defined reflections oriented at different Bragg’s angle corresponding to Miller indices (220), (311), (222), (400), (422), (511) and (440). All this reflections belongs to cubic spinel structure. Thus, XRD analysis confirms the formation of single phase compound. The particle size was obtained through Scherrer’s equation and found to be 21 nm, indicating the nanocrystalline nature. The magnetic properties were investigated using pulse field hysteresis loop tracer at room temperature. The saturation magnetization show increased values as compared to the bulk cobalt ferrite. The coercivity found to be less which exhibits the superparamagnetic behaviour. The obtained structural and magnetic parameters are useful in biomedical applications.

1. Introduction

Over the past decades ferrites have proved a prominent magnetic material used in many applications due to their excellent magnetic as well as electrical properties [1, 2]. They have the applications in the field of antenna rods, transformer cores, magnetic data storage, high frequency devices etc [3, 4]. In the recent decades, ferrites in nanocrystalline form have attracted many researchers as these nanoparticles exhibit smaller size, large surface to volume ratio and superparamagnetic behaviour [5, 6]. These characteristics of nanoparticles are useful in targeted drug delivery, hyperthermia, magnetic sensors, catalyst and many other areas [7]. Ferrite crystallizes in cubic spinel structure, cubic garnet structure and hexagonal structure. Spinel ferrite structure is very much attractive and show better
properties and applications compared to others. Spinel ferrite has the general formula $\text{MFe}_2\text{O}_4$ where \( \text{M} \) stands for divalent metal ions like cobalt, nickel, manganese, copper etc. The crystal structure of spinel ferrite possesses two interstitial sites namely tetrahedral (A) and octahedral (B) sites in which cations of different valence and size can accommodate bringing wide variation in the magnetic and electric properties. Many researchers have focused their attention on the synthesis and characterizations of spinel ferrite nanoparticles for various applications. Cobalt ferrite nanoparticles were also synthesized and studied for many properties by different researchers. According to the literature cobalt ferrite is an inverse spinel ferrite in which cobalt ions mostly occupy octahedral B site. The important magnetic properties of the ferrites are mostly depends on method of preparation, type and nature of dopant. The synthesis method influences the size and shape of the nanoparticles. In the conventional approach, the ferrites were prepared by ceramic technique mostly in the bulk form. This conventional technique mainly requires high temperature and longtime grinding for the preparation of samples. Also, sample prepared by these materials are chemically less stable and inhomogeneous. In contrast with this conventional approach, recently low temperature wet chemical methods such as sol-gel auto combustion method, chemical co-precipitation, hydrothermal etc. are emerged as an interesting methods to prepare ferrite nanoparticles in nano scale form [8, 9]. Among these methods sol-gel auto combustion methods is of great interest to the scientist as it yields fine and homogeneous nanopowder with greater chemical stability and lower size. Further, the method is simple, cheaper and requires minor equipments which are available easily and operates at low temperature of the order of 80°C to 120°C. In light of this we have prepared cobalt ferrite nanoparticles by low temperature wet chemical method i.e. sol-gel auto combustion using citric acid as a fuel. Further, the prepared sample was characterized by X-ray diffraction and pulsed field hysteresis loop technique to study the structural and magnetic properties.

2. Experimental

The nanocrystalline spinel structured cobalt ferrite ($\text{CoFe}_2\text{O}_4$) sample was prepared by sol-gel auto combustion method using citric acid as a fuel. AR grade chemicals such as cobalt nitrate ($\text{Co(NO}_3\text{)}_2$), ferric nitrate ($\text{Fe(NO}_3\text{)}_3$) and citric acid ($\text{C}_6\text{H}_8\text{O}_7$) were used for the synthesis. The metal nitrates to fuel ratio was taken as 1:3. Ammonia solution was added to adjust the pH of the solution at 7. The as-synthesized powder is sintered at 550°C for 4 h and then used for further investigations. The prepared samples were characterized by X-ray diffraction (XRD) technique by Regaku model. The XRD patterns were recorded at room temperature in the 20 range of 20° to 80° using Cu-Kα radiation ($\lambda = 1.54056 \ \text{Å}$). The magnetic properties of the sample were measured using pulse field hysteresis loop technique (Magnata Company) at room temperature. Using M-H plot, the various magnetic parameters such as saturation magnetization, remanence magnetization and coercivity were determined.

3. Results and discussion

3.1. X-ray diffraction analysis

The prepared cobalt ferrite nanoparticles were characterized by X-ray diffraction (XRD) technique. The powder XRD pattern of is shown in Fig. 1. It is observed from the pattern that all the peaks correspond to cubic spinel ferrite without any additional peaks. The average crystallite size was calculated using the Debye-Scherrer’s formula which is found to be 21 nm for cobalt ferrite nanoparticles. Using the interplanar spacing (d) and the corresponding Miller indices (h k l), the lattice constant (a) of the sample was calculated using standard relation and found to be $8.378 \ \text{Å}$. The value of lattice constant of cobalt ferrite well matches with that of reported in the literature [10].
Fig. 1 Flowchart for sol-gel auto combustion synthesis of CoFe$_2$O$_4$ NPs

Fig. 2 X-ray diffraction pattern of cobalt ferrite nanoparticles
3.2. **Magnetic properties**

The spinel ferrites are useful magnetic materials used in many technological applications. The study of various magnetic properties is of prime importance to understand their use in desired application. In the present study the magnetic properties were studied by pulse field hysteresis loop technique at room temperature. The M-H plot shows a typical hysteresis curve indicating the ferromagnetic behaviour of the sample. The M-H plot was used to know the values of saturation magnetization ($M_s$), coercivity ($H_C$) and remanence magnetization ($M_r$). The obtained values are 89.24 emu/gm, 384.62 Oe and 20.56 emu/gm for cobalt ferrite. It is evident from the values magnetic parameters that, wet chemical method significantly influenced the magnetic properties of cobalt ferrite nanoparticles.

![M-H plot of nickel ferrite nanoparticles](image)

**Fig. 3** M-H plot of nickel ferrite nanoparticles

4. **Conclusion**

Cobalt ferrite ($\text{CoFe}_2\text{O}_4$) nanoparticles were successfully synthesized using citric acid assisted sol-gel auto combustion technique. XRD analysis confirmed the formation of single phase cubic spinel structure. The lattice constant and X-ray density calculated from XRD data are in well agreement with the reported data. The crystallite size obtained through Debye-Scherrer’s formula found to be 22 for cobalt ferrite nanoparticles. The values of saturation magnetization, coercivity and remanent magnetization were deduced from M-H hysteresis loop technique and are found to be in the reported range. All the magnetic parameters were influenced by the low temperature wet chemical synthesis.

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