A comparison between structural and magnetic behavior of cobalt ferrite synthesized via solid state and chemical methods

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Abstract

Cobalt ferrite oxide (CoFe₂O₄) was synthesized by two different methods of mechanochemical and combustion. The phase composition and the chemical bonding information of the two powders were investigated by x-ray diffraction (XRD) method and Fourier Transform Infrared Spectroscopy (FT-IR). Field emission scanning electron microscopy (FESEM) was used to study the microstructure and morphology and elemental analysis was performed with EDS. The magnetic properties were analyzed at room temperature using a vibrating sample magnetometer (VSM). According to the structural and elemental analyses, cobalt ferrite was directly synthesized in the combustion method. However, the mechanochemical route was not successful in phase formation, even after heating at high temperatures. The hysteresis loops of VSM analysis showed that the CoFe₂O₄ powder from combustion method resulted in higher saturation magnetization and coercive field than the powder synthesized by mechanical milling. The inferior magnetic behavior of the powder from solid state method is attributed to the presence of Co³⁺ in the starting powder, which results in a non-spinel complex between cobalt and iron ions.

1. Introduction

In recent years magnetic materials including spinel ferrites have been attracting an ever growing interest. From a technological point of view ferromagnetic nanoparticles are regarded as potential candidates for fundamental constituents of future magnetic storage media. Therefore, many attempts have been made to improve the magnetic and electrical properties of ferrite nanoparticles [1]. Among spinel ferrites, cobalt ferrite (CoFe₂O₄; CFO) is an attractive material due to its admirable properties such as high magnetic residue, medium saturation magnetization, high magneto-crystalline anisotropy, high coercivity, reasonable mechanical hardness and proper chemical stability [2]. As a result, cobalt ferrites are regarded as promising candidates for the development of modern electronic devices, such as capacitors, sensors or high capacity batteries [3–5]. Additionally, they are used in many technological application including high-density recording systems, microwave adsorbents, drug delivery systems, cell identification and separation, catalyst for water separation, magnetic switches, and chemical sensors. The properties of spinel ferrite nanoparticles are highly sensitive to the various factors such as preparation techniques, particle size and morphology, annealing temperature and cation distribution at tetrahedral and octahedral sites [6]. The spinel ferrite has a face centered cubic (fcc) structure filled with oxygen ions and the unit cell contains octahedral and tetrahedral interstitial sites occupied by metal cations, resulting in a different local symmetry [7]. Bulk CoFe₂O₄ possesses an inverse spinel structure with Co²⁺ ions in octahedral site and Fe³⁺ ions equally distributed in tetrahedral and octahedral sites. However, CFO nanoparticles exhibit cationic inversion and the amount of Co²⁺ and Fe³⁺ ions at both sites depends on the method of preparation [8]. Cationic distribution in octahedral and tetrahedral sites may be quantified by the inversion degree (γ), which can be defined as the fraction of Fe³⁺ ions in the tetrahedral sites [7, 9]. The magnetic properties of spinel ferrites are affected by the interactions between cations at the octahedral and tetrahedral sites with oxygen ions [10]. Additionally, the ferromagnetic behavior is due to the antiparallel spins between the Fe³⁺
ions at the octahedral sites and the Co$^{2+}$ ions at the tetrahedral sites. It is also affected by the distribution of cations in A and B sites and the ferrite preparation methods [11, 12].

Cobalt ferrite is synthesized by various methods such as sol-gel [12] coprecipitation [13], hydrothermal [14], combustion [15], mechanical alloying [16], etc. In one hand, the wet chemical methods often require low processing temperature. For example, CoFe$_2$O$_4$ nanoparticles can be synthesized in the temperature range of 164°C–192°C by a microwave assisted hydrothermal process, while the solid-state methods necessarily requires a high temperature of about 800°C [17]. On the other hand, the wet chemical methods suffer from some limitations such as high preparation cost and complex precursor solutions, which do not exist in solid state methods [18].

Mechanical alloying using high energy mills is an efficient solid state route that results in ultrafine particles with increased coercivity due to higher induced strain [9]. The milling energy is provided by some factors including milling media, ball to powder ratio, milling speed and milling time [19]. Among the chemical methods, combustion synthesis is increasingly used in the production of oxide catalysts due to relatively low production cost, high purity and homogeneous nanoparticle. The combustion method mainly involves combining the reactants in an aqueous medium using complex agents such as glycine as well as oxidizing agents such as metal nitrate [20].

In this study, cobalt ferrite was synthesized with two different routes: mechanochemical reaction using mechanical alloying, as a conventional solid-state method, and combustion route as a wet chemical method. The phase structure and microstructure of the two powders as well as the magnetic behavior were studied in details. The Previous works about mechanochemical synthesis of CFO mainly used Co$^{2+}$ in the forms of CoCO$_3$, CoO as cobalt sources in raw materials. According to the thermodynamic calculations, the type of raw materials and the molar ratios play an important role in the nature of reactions and the subsequent phase formation. Therefore, in this study we tried to synthesis the CFO powder by using Co$_3$O$_4$ as cobalt source in starting materials. The starting powders were milled for 5 and 10 h then the as-milled samples heated isothermally in air at different temperatures. Additionally, the combustion method was also performed to synthesize CFO using different raw materials to see the differences between the synthesized powders in these two methods.

2. Experimental

In this research, cobalt ferrite was synthesized by two different methods: the mechanochemical reaction using planetary mill and the combustion route. High purity Fe$_2$O$_3$ (≥99) and Co$_3$O$_4$ (≥99) powders supplied by Merck Company were used as starting materials in the mechanochemical method. In previous studies the source of cobalt ions were cobalt oxide (CoO) [21, 22] or cobalt carbonate [16, 23], therefore, in this work we decided to try a different source of cobalt, i.e., Co$_3$O$_4$, which is rarely used in previous works [24, 25]. The three different reactions about CFO formation based on the cobalt sources are as the following:

\[
\text{CoO} + \text{Fe}_2\text{O}_3 \rightarrow \text{CoFe}_2\text{O}_4 \tag{1}
\]

\[
\text{CoCO}_3 + \text{Fe}_2\text{O}_3 \rightarrow \text{CoFe}_2\text{O}_4 + \text{CO}_2 \tag{2}
\]

\[
\frac{1}{3} \text{Co}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \rightarrow \text{CoFe}_2\text{O}_4 + \frac{1}{6} \text{O}_2 \tag{3}
\]

As it is seen from equation (3), in order to obtain one mole of stoichiometric CoFe$_2$O$_4$, the molar ratio of Co$_3$O$_4$ to Fe$_2$O$_3$ should be 1/3. Thermodynamic assessments using HSC software (version 6.12, Outotec Research Oy, 1974–2007) show that the reaction between Co$_3$O$_4$ and Fe$_2$O$_3$ to form CoFe$_2$O$_4$ (equation (3)) is exothermic ($\Delta H^0_{298K} = -13.4 \text{ kJ}$) and favorable at room temperature ($\Delta G^0_{298K} = -26.3 \text{ kJ}$). The raw materials were milled for 5 h at 300 rpm using a high energy mill (Amin asiya fanavar, NARYA MPM, Iran). Milling was performed in steel containers with steel balls and the ball to powder ratio (BPR) of 20. Since the milling time of 5 h was not enough for phase formation, another 5 h was added and the milling time increased to 10 h. The as-milled powder was then calcined at 600°C–1000°C for 3 h in air.

In the combustion route, analytical grade cobalt nitrate [Co(NO$_3$)$_2$·6H$_2$O] (99.8%) ferric nitrate [Fe(NO$_3$)$_3$·9H$_2$O] (99%) were taken as raw materials while glycine [CH$_2$NH$_2$COOH] (98%) was employed as fuel to drive the combustion reaction. In typical procedure, the stoichiometric amounts (G/N = 1.48) of cobalt nitrate, ferric nitrate and glycine were taken in a glass container. In the present work, glycine to nitrate (G/N) molar ratio was 2.22 to obtain fuel rich condition [12]. Due to the hygroscopic nature of the metal nitrates, after mixing for 15 min the precursors resulted in brown viscous slurry. The container was then preheated to 300°C using a hot plate. The whole combustion reaction process was complete in less than 15 min, whereas actual time of ignition was less than 5 s. During combustion, a large amount of foams was produced and some sparkles appeared at one corner which spread out through the whole mass and yielded a bulky and fluffy product. This synthesis route was accompanied by a subsequent heating at 800°C for 2 h in air.
Phase structure was characterized with XRD (Philips PW 1730, Netherlands) using Cu-Ka radiation and MAUD software (version 2.99, July 2021) was used to perform the Rietveld refinement. In order to characterize the chemical bonding, FTIR measurements were performed using a FTIR spectrometer (Shimadzo 8300, Kyoto, Japan). Microstructure and elemental composition of the powders were investigated by a field emission scanning electron microscope (FESEM, MIRA III, Republic Czech). Room temperature magnetic properties were studied using a VSM device (VSMFT, Daneshpajouh Kashan Company, Iran).

3. Results and discussion

The XRD patterns of as milled CFO powders are demonstrated in figure 1. After 5 h milling, the peaks of iron oxide and cobalt oxide (raw materials) are observed in the corresponding XRD pattern. By increasing milling time to 10 h, the XRD pattern was similar to 5 h as-milled sample and the peaks of raw materials (i.e., cobalt oxide and iron oxide) were still observed in figure 1. The XRD results of as-milled samples in figure 1 show that cobalt ferrite (CFO) cannot be formed after 10 h milling.

Figure 2 shows the XRD patterns of as-milled and annealed CoFe2O4 powders prepared with mechaochemical method. The initial structural analysis was performed with Xpert High score software and in order to better identify the phases, Rietveld refinement was performed using MAUD software.

The phase analysis in XRD pattern of the milled samples (figure 1) confirms the presence of cobalt oxide (CoCo2O4; 01-080-1541), maghemite (Fe72O96; 96-900-6319) and iron oxide (Fe2O3; 01-089-2810). At 600 and 700 °C we still have these previous phases, but after heating at 1000 °C for 3 h in air, the phases are mainly consisted of tetragonal maghemite and cobalt ferrite (96-591-0064). Although at 1100 °C the powder started to sinter, the phases are the same as 1000 °C and single phase CFO was not formed at this temperature. As it is seen in the XRD patterns of high temperatures (figure 2), the majority of the maghematite peaks are overlapped with cobalt ferrite (CFO) and the corresponding peaks are hardly separated. Maghemite as a Fe$^{2+}$ deficient spinel ferrite consisting Fe$^{2+}$ and Fe$^{3+}$ ions [26], can be considered as an intermediate phase before CFO formation.

Although combustion method resulted in a single-phase spinel CFO (reference code: 96-591-0064) with no sign of secondary phases, the mechanical alloying method did not yield the single-phase CFO even after heating the 10 h milled sample at 1000 °C. Phase analysis revealed that the powder from combustion method exhibited cubic spinel structure CFO (Co16Fe8O32) with Fd-3m space group, which is confirmed with Rietveld refinement. As it is clearly seen in figure 3, the XRD pattern of CFO powder produced from combustion method did not change after annealing at 800 °C and the former CFO phase still exists in this powder.

A precise Rietveld refinement with maghemite and CFO phases (figure 4) indicated pure maghemite phase in the powder form mechanochemical method and the combustion method resulted in pure CFO. Due to the proximity of the ionic radius of cobalt (Co$^{2+}$) and iron (Fe$^{3+}$), the peak positions in CFO are slightly shifted to lower angles compared with iron oxide and it is difficult to distinguish between CFO and maghemite. Nevertheless, the presence of cobalt ions will be confirmed by FTIR (figure 5) and EDS elemental analysis (figure 7) of the following sections.

Figure 1. The XRD patterns of as milled powders after milling for 5 and 10 h.
It should be noted that the refinement results are in agreement with initial phase estimations and the low values of χ parameter ($\chi = \sqrt{\frac{RSSR}{RSSP}}$) is an indication of good fitting between measured and calculated patterns (table 1). Accordingly, though the mechanical alloying as a solid state method requires a very high energy to form cobalt ferrite oxide, the more important parameter is the valence of initial ions. Regarding the stoichiometric formula of cobalt ferrite, the valence of cobalt and iron needs to be +2 and +3, respectively; Otherwise, the CFO composition would not follow the stoichiometric formula. In this study, Co$_3$O$_4$ as the initial source of cobalt is a combination of +2 and +3 ions, in which the Co$^{3+}$ ions cannot fill the interstitial sites of spinel CFO. Therefore, a non-spinel structure will be formed instead of stoichiometric CFO. It should be noted that the raw materials of combustion method include cobalt and iron nitrates with valence of +2 and +3 for Co and iron ions respectively, which can easily form stoichiometric CFO. Ding et al.[27] used similar raw materials to synthesize the CFO composition via mechanical alloying. They used Co$_3$O$_4$ and Fe$_2$O$_3$ with molar ratio of 1:2, which is different from the molar ratios in this research (based on equation (3)). Ding et al.[27] concluded that CFO is not

Figure 2. The XRD patterns of as synthesized and annealed CFO sample prepared by mechanochemical method. The 10 h milled sample has been annealed at different temperatures for 3 h in air.

Figure 3. The XRD patterns of as synthesized and annealed (800 °C, 2 h, Air) CFO powders prepared with combustion method.
formed during milling, even after 24 h of milling with high energy Spex mill, however, a single phase spinel CFO was formed after annealing at 1000 °C.

Figure 5 compares the FTIR spectra of the as-synthesized CFO powders from combustion method and the annealed powder from mechanochemical route. The peaks at 400–412 and 597–615 cm⁻¹ are attributed to vibration bonds at octahedral sites of Fe-O and tetrahedral sites of Co-O, respectively [28]. As it is shown in figure 5, these vibrations are observed in the two powders of combustion and mechanical alloying methods. In the FTIR spectra of combustion method there is a small peak at about 990 and a wide one exists around 1100, which are stronger in the spectra of mechanical alloying method (≈970 and ≈1050 cm⁻¹). The only difference between these two spectra is the peaks located at 454, 615 and 876 cm⁻¹, which are observed in the FTIR spectra of the CFO powder from mechanical alloying route. Nazari et al [29], studied the structural and magnetic properties of maghemite nano particles and reported similar peaks in the FTIR spectra of iron oxide. Accordingly, these two peaks are attributed to pure maghemite. As it is clearly seen, the FTIR spectra well confirms the XRD results; a pure spinel CFO exists in the powder from combustion method, whilst, the presence of maghemite and CFO in the powder from mechanical alloying is well recognized. It should be noted that there were no peaks in the high wavenumber region of the FTIR spectra of both powders, which implies the complete removal of organic compounds from the powders.

The microstructures of the synthesized CFO powders from combustion method and conventional solid state (mechanical alloying) methods are shown in figure 6. The microstructures are compared at two different magnifications. According to the FESEM images, the combustion method resulted in CFO powder with smaller particle size. The average particle size of the annealed powders in mechanical alloying is estimated to be about 300 nm, whilst the microstructure of the as-synthesizes powder in combustion method shows much smaller

Figure 4. Rietveld refinement results of CFO powders obtained from MAUD software. The 10 h milled powder of mechanical alloying route was heated at 1000 °C for 3 h.
particles, which are closely attracted to each other and formed aggregates. The average particle size is not clearly distinguished in this magnification and the agglomerates seem to be about 700 nm. The results of figures 6(c)–(d) reveal that a high energy is provided by combustion method, which is enough to form crystalline spinel CFO without further annealing. It is clearly seen in figure 6(b) that the powders from mechanical alloying have a cuboid morphology, that is consisted with the crystal structure estimated from XRD analysis (results of figure 2). According to figure 6(d), the particles obtained from the combustion method have foam like and non-uniform morphology.

Prabhakaran et al synthesized CFO powder with combustion method. They declared that the released temperature during combustion process and the magnetic interactions between particle resulted in such aggregates [30]. In another study, Salunkhe et al, synthesized CFO nano particles with different G/N ratios and reported the same observations. They also declared that by increasing the G/N ratio to 2.22, a porous foam like morphology was observed which is attributed to the escape of combustion gases during the process [12]. According to the work done by Kaufmann et al, the combustion method yields to a spongy CFO microstructure in which the porosities are the product of gas involved reactions and the evolution of the gases results in such morphology [20].

The EDS spectra of the two powders are shown in figure 7. The characteristics peaks of Co, Fe and O are observed in the spectra and the elemental mapping corroborates the uniform distribution of the constituents. According to table 2, the Co/Fe ratio of about 0.5 is reached in the combustion method, while the nonstoichiometric formula of the powder from mechanical alloying (mechanochemical reaction) is confirmed by the Co/Fe ratio of about 1.5, which implies that the synthesized cobalt ferrite is based on the Co3Fe2O4 formula not the stoichiometric CoFe2O4. As it is discussed in previous sections, the raw materials have a predominant role in mechanochemical synthesis of cobalt ferrite. The presence of Co2+ and Fe3+ ions is vital to get the stoichiometric CoFe2O4 and other valences may result in non-stoichiometric compositions. The Co/Fe ratio of 1.5 obtained in the powder from mechanochemical reaction (solid-state method) is consistent with Co3Fe2O4 formula, which is not identified in structural analysis.

The room temperature magnetic hysteresis loops of the powders synthesized by mechanical alloying and combustion methods are shown in figure 8. The values of saturation magnetization (Ms), coercivity (Hc), and remanent magnetization (Mr) are shown in table 3. As it is seen, the magnetic behavior of CFO powder from
The combustion method is comparable to the values reported in literature [11]. However, for the milled and annealed powder, the corresponding magnetic parameters are inferior to the typical cobalt ferrite. Based on the structural and elemental studies of previous sections, the presence of Co$^{3+}$ ions resulted in an intermediate phase between cobalt and iron oxides, which is regarded as a magnetic composition. However, the presence of maghemite and cobalt oxide with inherent magnetic behavior [26, 31], which is lower than spinel CFO is another indication for the inferior magnetic properties in the powder from mechanochemical method.

Table 4 compares the magnetic and structural properties of the CFO powder synthesized from combustion method of this study with similar works in other papers. Since the mechanochemical method did not result in single phase CFO, the structural parameters are calculated for the sample of combustion method. The lattice parameters, lattice strain and density of CFO were calculated from the XRD results of as-synthesized CFO powder. As it is seen, the lattice parameter of the synthesized CFO powder is about 8.333 nm and it is in the range of the results of Prabhakaran et al [30]. In the other study, Salunkhe et al [12] reported a different value for lattice parameter of CFO, which resulted in lower x-ray density (table 4). The crystallite size and lattice strain of synthesized CFO were calculated using Williamson-Hall method and the amount of crystallite size was about 33.6 nm. As it seen in table 4, the amount of crystallite size of CFO in this research is in good agreement with the results of previous works. Accordingly, the magnetic parameters of the synthesized CFO powder are summarized table 4. While the remanent magnetization and coercive field of our synthesized CFO is larger than the other results, the value of saturation magnetization is between the reported values of other studies. It should be noted that the G/N (Glycine to nitrate) ratio of our study is similar to that of Salunkhe et al, and is lower than the value of Prabhakaran et al. Since this G/N ratio plays an important role in the combustion method and severely affects the magnetic properties [12], it makes sense to compare the results with the ones from Salunkhe et al. This comparison implies that our synthesized CFO powder with higher $H_C$, $M_r$, and $M_s$ and accordingly larger loop squareness ratio ($M_r/M_s$) is a good candidate for hard magnetic applications.

![Figure 6. FESEM images of (a) and (b) the CFO particles of mechanical alloying after heating at 1000° for 3 h in air, (c) and (d) the as-synthesized CFO powders from combustion method.](image)
Figure 7. EDS analysis and elemental mappings of CFO particles (a) synthesized with mechanical alloying (10 h milled, annealed at 1000 °C for 3 h), and (b) as-synthesized powder from combustion method.

Table 2. Summary of the data from EDS analysis of figure 6 for synthesized CFO powders.

| Element | Mechanical alloying Atomic % | Combustion Atomic % |
|---------|-----------------------------|---------------------|
| O       | 58.03                       | 45.48               |
| Fe      | 16.87                       | 35.03               |
| Co      | 25.10                       | 19.49               |
| Total   | 100                         | 100                 |
| Co/Fe   | 1.49                        | 0.56                |
4. Conclusion

In this research, cobalt ferrite was synthesized via two different methods: mechanochemical and combustion. Our results revealed that in spite of having a negative $\Delta G_{298 \text{K}}^{0}$, a spinel CFO is not formed after 10 h milling. Furthermore, the subsequent heating of 10 h milled sample at 1000 °C for 3 h was not successive in phase formation. The presence of Co$^{3+}$ was regarded as the dissuasive factor of spinel CFO formation in the mechanochemical route. In the combustion method, the situation was different and due to the presence of the Co$^{2+}$ ions in the starting powder (cobalt nitrate), the as-synthesized sample was consistent with spinel cobalt ferrite of XRD analysis. These results were confirmed with the elemental analysis of EDS analysis. The VSM results showed lower magnetic properties for the powder from mechanochemical method, which is attributed to the non-spinel nature of the synthesized cobalt ferrite. The synthesized powder of the combustion method resulted in saturation magnetization of 62.3 emu g$^{-1}$ and the coercive field of 1.34 kOe, which are in good agreement with the previous studies.

\begin{table}[h]
\centering
\caption{Summary of magnetic properties of CFO powders obtained from VSM analysis based on figure 7.}
\begin{tabular}{lccc}
\hline
Sample & $M_s$ (emu g$^{-1}$) & $M_r$ (emu g$^{-1}$) & $E_c$ (Oe) \\
\hline
CFO-mechanical alloying & 20.4 & 9.23 & 656.5 \\
CFO-Combustion & 62.3 & 33 & 1338 \\
\hline
\end{tabular}
\end{table}

\begin{table}[h]
\centering
\caption{The structural and magnetic properties of the as-synthesized CFO powders of this research prepared from combustion method (the second row) and the results of other works.}
\begin{tabular}{lccccccccc}
\hline
Sample & $a$ (nm) & $d$ (nm) & $\varepsilon$ (%) & $\rho$ (g cm$^{-3}$) & $M_r$ (emu g$^{-1}$) & $M_s$ (emu g$^{-1}$) & $E_c$ (Oe) & $M_r/M_s$ \\
\hline
CFO-combustion method, as-synthesized & 8.333 & 33.6 & 0.2 & 5.4737 & 33 & 62.3 & 1338 & 0.53 \\
Salunkhe et al\cite{12} & 8.377 & 38 & — & 5.1213 & 28.09 & 59 & 820.4 & 0.47 \\
Prabhakaran et al\cite{30} & 8.330 & 37.8 & 0.28 & — & 29.21 & 71.1 & 603.28 & 0.41 \\
\hline
\end{tabular}
\end{table}
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Data availability statement
All data that support the findings of this study are included within the article (and any supplementary files).

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