Original Article

Changing the Magnetic Properties of Cobalt Ferrite Nanoparticles with Different Fabrication Conditions

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Abstract: In this study, crystalline nanoparticles $CoFe_2O_4$ with a spinel structure were prepared by hydrothermal methods. The magnetic properties of non-calcined cobalt ferrite formed from nanocrystalline powders. The dependence of the particle size and crystalline structure of obtained nanoparticles in the synthesis conditions was examined and characterized using field emission scanning electron microscope (FESEM), and X-ray diffraction analysis (XRD). The XRD analysis revealed a high degree of crystallinity and confirmed the spinel structure of crystalline nanoparticles $CoFe_2O_4$. The FESEM image shows the presence of spherical ferrite particles with an average diameter of about 13-18 nm. The results also show that the formation of cobalt ferrite spinel structures was affected by fabrication conditions. Magnetic hysteresis loop data confirm that the magnetic properties of nanoparticles depend on the synthesis conditions. The material prepared by the hydrothermal route and calcination at 150ºC with molar ration Co^{2+}: Fe^{3+} = 1:2.2 for 2 hours has higher magnetic saturation than that of the surveyed samples.

Keywords: Cobalt ferrite, hydrothermal, magnetic properties.

1. Introduction

In recent years, nanocrystalline materials are becoming a subject of intense research because of their unique properties. Magnetic nanoparticles have been of interest for their typical physical and chemical

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properties as well as their potential applications in various fields such as high-density magnetic recording, magnetic fluids, data storage, spintronics, solar cells, sensors, and catalysis.

Among many ferrites, cobalt ferrite magnetic nanoparticles are attracting much attention because of their high coercivity, magnetocrystalline anisotropy, moderate saturation magnetization, chemical stability, wear resistance, electrical insulation, and structure [1]. Structurally, in the inverse spinel of the ferrite, tetrahedral sites are generally occupied by Fe^{3+} ions, whereas octahedral sites (B-sites) are inhabited by Co^{2+} and Fe^{3+} ions [2]. To alter the structure and magnetic properties of ferrite nanoparticles, it is necessary to modify their composition and microstructures via different preparation routes [2]. $CoFe_2O_4$ nanoparticles were previously prepared by a wide array of synthesis routines, such as chemical co-precipitation [3, 4], sol-gel [5, 6], solid-state method [7], solvothermal [8], solution combustion [9–12] and hydrothermal method [13, 14]. For each synthesis method, it was found that the fabrication condition played a key role in determining the structure and magnetic properties of the obtained product. Among these techniques, chemical co-precipitation has been reported to be the most economical one. In addition, the hydrothermal method has been confirmed to be a high rate of production and simplicity.

This paper reports on the effect of fabrication condition on the magnetic properties of cobalt ferrite samples, prepared by hydrothermal processes for the purpose of creating highly magnetic $CoFe_2O_4$ nanoparticles to be used as a raw material to synthesize multi-phase ferroelectric/ferromagnetic material from applications in energy storage devices.

2. Material and Methods

Cobalt ferrite nanoparticles ($CoFe_2O_4$) were synthesized by using the hydrothermal method. Cobalt nitrate hexahydrate ($Co(NO_3)_2.6H_2O$), ferric nitrate nonahydrate ($Fe(NO_3)_3.9H_2O$) and potassium hydroxide (KOH) with a purity of 98% were used as the precipitating agents and deionized water as solvent. All chemicals were purchased from Guangdong Guanghua Sci-Tech Co., Ltd. The chemical reaction is described by:

$$Co^{2+} + 2Fe^{3+} + 8OH^- \longrightarrow CoFe_2O_4 + 4H_2O \qquad (1)$$

* Effect of Mole Ratio of Co^{2+}:Fe^{3+}

Initially, we fixed the number of moles of $Co(NO_3)_2.6H_2O = 0.02$ mol and then changed the number of moles of $Fe(NO_3)_3.9H_2O$ from 0.032 mol, 0.036 mol, 0.04 mol, 0.044 mol and to 0.048 mol corresponding to the molar ratio of Co^{2+}: Fe^{3+} = 1:1.6; 1:1.8; 1:2.0; 1:2.2 and 1:2.4.

Then, $Co(NO_3)_2.6H_2O$ mixed with $Fe(NO_3)_3.9H_2O$ was dissolved in 80 ml of distilled H_2O. The solution was mixed and stirred at room temperature (25°C) using a stirring rate of 120 rpm for 15 minutes. KOH was dissolved in distilled H_2O and then added stepwise to the reaction mixture until pH = 12 was reached. When precipitate was observed at the bottom of the reaction mixture, we transferred all the mixture and precipitate obtained into an autoclave and treated them at the temperature of 150°C for 2 hours. The precipitate separated from the solution was washed several times with distilled H_2O and then was dried at 80°C for 12 hours to get the final product, $CoFe_2O_4$.

* Effect of Reaction Temperature

The procedure for making CFO samples is done as stated in the effect of the molar ratio of Co^{2+}:Fe^{3+} above. The samples were synthesized for 2 hours at different reaction temperatures (110°C, 130°C and

150°C) with the molar ratio of $Co^{2+}:Fe^{3+}$, which is the ratio for the highest magnetic CFO result obtained from the survey according to the effect of molar ratio $Co^{2+}:Fe^{3+}$.

* Effect of Reaction Time

The procedure for making CFO samples is done as stated in the effect of the molar ratio of $Co^{2+}:Fe^{3+}$ above. The samples were synthesized during different reaction times (from 1 hour to 8 hours) with the molar ratio of $Co^{2+}:Fe^{3+}$ and reaction temperature, which is the ratio and temperature for the highest magnetic CFO result obtained from the survey according to the effect of molar ratio $Co^{2+}:Fe^{3+}$ and effect of reaction temperature.

The crystal structures of the samples were characterized by XRD using diffractometer XRD EQUINOX 5000 with Cu-Kα radiation (λ=1.5406 Å) and the morphology (size and shape) of the particle materials was obtained by field emission scanning electron microscopy FESEM (Hitachi S-4800) at the Institute of Materials Science, Vietnam Academy of Science and Technology. Hysteresis loops were measured at room temperature to the highest field of 8000G using a vibrating sample magnetometer (VSM) at VNU Key Laboratory for Micro and Nanotechnology - VMINA.

3. Results and Discussion

3.1. Effect of Mole Ratio of $Co^{2+}:Fe^{3+}$

Magnetic cobalt ferrite ($CoFe_2O_4$) nanocrystals were synthesized via the hydrothermal method with reaction conditions: Hydrothermal Time 2h, Hydrothermal Temperature 150°C and the number of moles of Co^{2+} is kept constant (0.02 mol), and the number of moles of Fe^{3+} varies in such a way that the corresponding molar ratio of $Co^{2+}: Fe^{3+}$ = 1:1.6; 1:1.8; 1:2.0; 1:2.2 and 1:2.4.

Crystal Structure

XRD patterns of some $CoFe_2O_4$ at different molar ratios are shown in Figure 1.
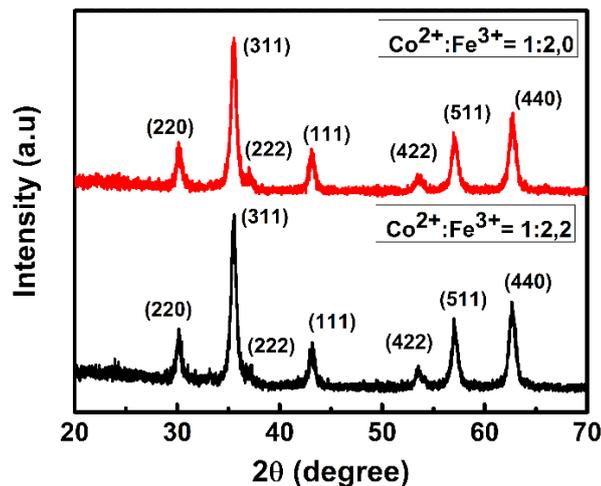
Figure 1. XRD patterns of some $CoFe_2O_4$ at different molar ratios.

Different XRD patterns of cobalt ferrites corresponding to different molar ratios $Co^{2+}:Fe^{3+}$ are shown in Figure 1. The reflection peaks correspond to the characteristic spacing between (220), (311), (222), (111), (422), (511) and (400) planes of a cubic spinel structure, providing clear evidence of the formation of cobalt ferrite (JCPDS standard card for $CoFe_2O_4$ No. 01-077-0426). $CoFe_2O_4$ ferrite samples in fine crystalline phase, single-phase, and the crystal phase formed completely during hydrothermal processes. It is believed that in a thermos flask, $CoFe_2O_4$ formation reactions can take place as follows: [15]

$$KOH \longrightarrow K^+ + OH^- \qquad (2)$$

$$Fe^{3+} + 3OH^- \longrightarrow Fe(OH)_3 \qquad (3)$$

$$Co^{2+} + 2OH^- \longrightarrow Co(OH)_2 \qquad (4)$$

$$Fe(OH)_3 \longrightarrow \beta FeOOH \qquad (5)$$

$$2FeOOH + Co(OH)_2 \longrightarrow CoFe_2O_4 + 2H_2O \qquad (6)$$
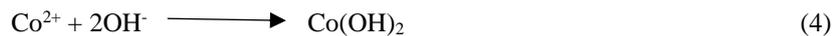
The average crystallite size of as-prepared $CoFe_2O_4$ ferrite particles was estimated through analysis using the classical Scherrer formular [16] $D_{hkl} = k\lambda/\beta\cos\theta$, where D_{hkl} is the crystallite size derived from the (311) peak of the XRD profiles, k is the sphere shape factor (0.89), λ is the wavelength of X-ray (1.54056Å), β is the full-width at half-maximum (FWHM) of the peak in radians and θ is the Braggs angle in radians. The obtained average crystallite size of as-prepared $CoFe_2O_4$ ferrite particles is about 15 nm. Based on the peak central positions obtained from the high-intensity low-angle (311) and high-intensity high-angle (440) peaks, the ferrite lattice parameter was estimated at 8.267Å, which is in good agreement with the bulk value of 8.377Å [17].

Magnetic Properties

Various magnetic properties including saturation magnetization (Ms), coercivity (Hc), and remanent magnetization (Mr) are listed in Figure 1 and Table 2. From the values obtained, it was found that $CoFe_2O_4$ samples with the molar ratio of $Co^{2+}:Fe^{3+}=1:2.2$ gave good magnetic results with the highest magnetization saturation Ms = 58.86 emu/g, which is smaller than the bulk value (74.08 emu/g) [18]. The remanence magnitude, Mr, can be extracted from the hysteresis loop at the intersections of the loop with the vertical magnetization axis. The Mr value of 16.05 emu/g. for nanosized ferrite particles, the surface areas are larger and thus the surface energy and surface tension are high. This results in changes in cationic preferences and leads to an increased degree of antisite defects and thus lesser magnetizations [19,20]. This result is used to carry out the next surveys.

3.2. Effect of Reaction Temperature

Temperature is one of the determinants of crystal structure and size. Since the formation of ferrite crystals has been formed during hydrothermal processes, we have conducted a hydrothermal temperature survey. We conducted a sample survey at a molar ratio of $Co^{2+}:Fe^{3+}=1:2.2$, hydrothermal time for 2 hours and hydrothermal temperatures of 110℃, 130℃, and 150℃.

Crystal Structure

XRD patterns of $CoFe_2O_4$ at different reaction temperatures are shown in Figure 3. Comparing the XRD results of the sample of ferromagnetic materials made by $CoFe_2O_4$ to the standard XRD data of the $CoFe_2O_4$ sample, it was found that the samples all showed diffraction peaks and these peaks completely coincided with the standard data of the $CoFe_2O_4$ sample. However, in Figure 3, $CoFe_2O_4$ samples made at 150℃ for diffraction peaks, are stronger and sharper than $CoFe_2O_4$ samples

manufactured at 110℃ and 130℃. It is shown that at 150℃, $CoFe_2O_4$ samples are the best crystallized in single-phase, and the crystalline phases formed completely during hydrothermal processes.

Table 1. Summary of the magnetic property values of $CoFe_2O_4$ patterns at different molar ratios

Sample	Ms (emu/g)	Mr (emu/g)	Hc (G)
Co^{2+}: Fe^{3+} = 1:1.6	51.07	14.19	650.27
Co^{2+}: Fe^{3+} = 1:1.8	54.34	13.08	551.44
Co^{2+}: Fe^{3+} = 1:2.0	55.38	15.61	665.15
Co^{2+}: Fe^{3+} = 1:2.2	58.86	16.05	663.85
Co^{2+}: Fe^{3+} = 1:2.4	51.44	15.89	690.20
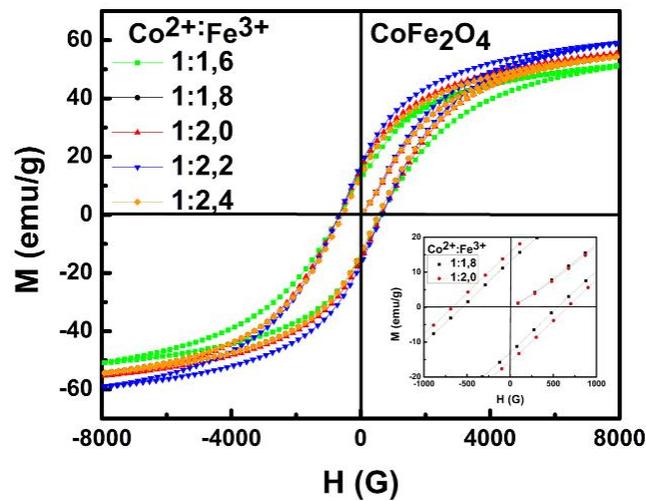
Figure 2. Hysteresis loop of $CoFe_2O_4$ sample at different molar ratios. The inset shows magnification around applied field of 500 G of their loops with molar ratios of Co^{2+}: Fe^{3+} are 1:1.8 and 1:2.0.
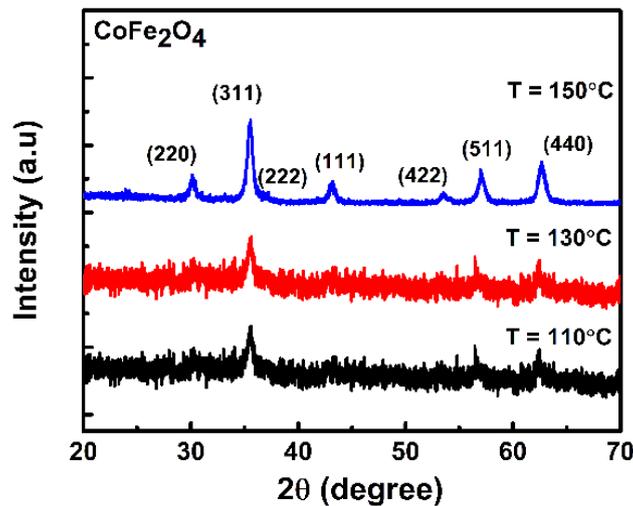
Figure 3. XRD patterns of $CoFe_2O_4$ sample at different reaction temperatures.

Magnetic Properties

The hysteresis loop of $CoFe_2O_4$ sample at different reaction temperatures is shown in Figure 4.
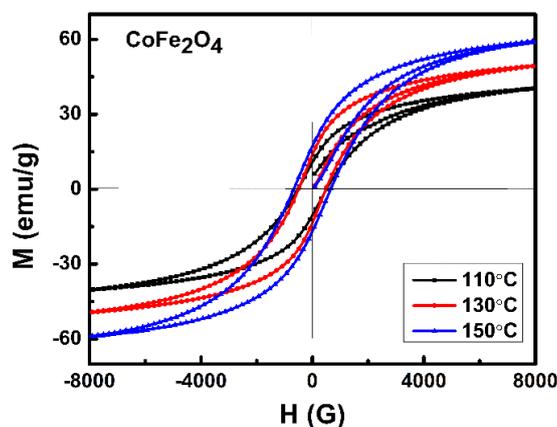
Figure 4. Hysteresis loop of $CoFe_2O_4$ sample at different reaction temperatures.

Figure 4 and Table 2 show that $CoFe_2O_4$ samples made at the temperatures of 110 ℃, 130 ℃, and 150 ℃ have an average coercive field (Hc) from 490.05 - 663.85 G, saturation value from 40.26 to 58.86 emu/g, and residual value from 10.61 - 16.05 emu/g. $CoFe_2O_4$ samples have good magnetism with the highest magnetization saturation Ms = 58.86 emu/g, remanence magnitude Mr = 16.05 emu/g and coercivity field Hc = 663.85 G during hydrothermal process at 150℃. It was observed that Ms value showed a positive correlation with the particle size. This was in line with Kumar et al., suggesting that increased particle size could lead to improved magnetization. This result is used to carry out subsequent surveys.

Table 2. Values of $CoFe_2O_4$ magnetic properties pattern at different reaction temperatures.

Sample	Ms (emu/g)	Mr (emu/g)	H_C (G)
110℃	40.26	10.61	513.62
130℃	49.28	13.63	490.05
150℃	58.86	16.05	663.85

3.3. Effect of Reaction Times

In this context, we investigate the effect of reaction time on the structure and magnetism of $CoFe_2O_4$ ferrite nanoparticles with the reaction temperature of 150℃, the molar ratio of Co^{2+}: Fe^{3+} = 1:2.2 and time varies from 1 hour to 8 hours.

Crystal Structure

The XRD structural properties of the synthesized $CoFe_2O_4$ pattern in Figure 5 show the characteristic peaks of the following reflection planes (220), (311), (222), (111), (511), and (440). These planes prove the presence of a spinel cubic structure. As all the $CoFe_2O_4$ samples, made during the raction times of 2

hours, 4 hours, 6 hours, 7 hours and 8 hours, show good results (Figure 5), we choose the reaction time of 2 hours to perform the next experiment to shorten the experimental time.
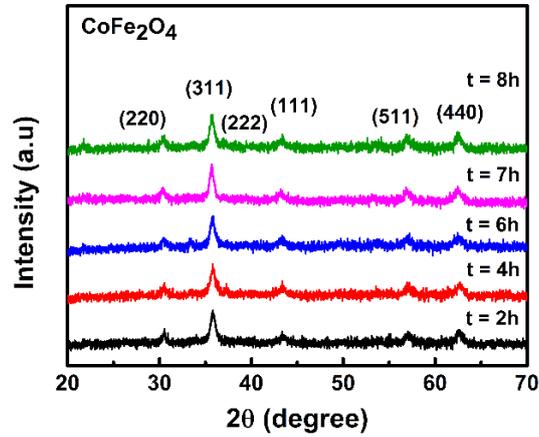
Figure 5. XRD pattern of $CoFe_2O_4$ at different reaction times.

Magnetic Properties

The hysteresis loop of $CoFe_2O_4$ sample at different reaction times is shown in Figure 6.
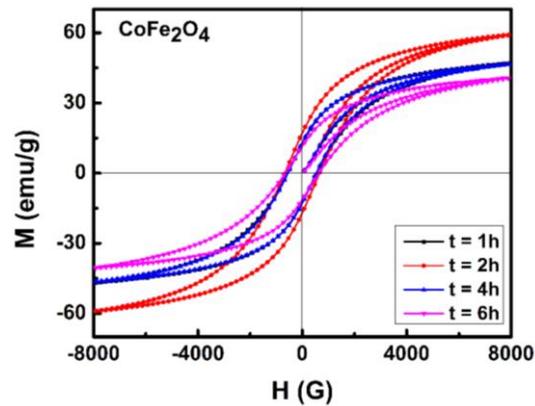
Figure 6. Hysteresis loop of $CoFe_2O_4$ sample at different reaction times.

Table 3. Values of the $CoFe_2O_4$ magnetic properties pattern at different reaction times.

Sample	Ms (emu/g)	Mr (emu/g)	Hc (G)
1h	46.86	12.62	557,93
2h	58.86	16.05	663.85
3h	51.86	15.75	606.14
4h	46.52	12.91	544.75
5h	42.59	11.73	628.63
6h	40.63	11.35	644.73
7h	47.02	14.07	703.67
8h	42.96	13.65	781.54

Figure 6 and Table 3 show that the $CoFe_2O_4$ samples are made during timespans from 1 hour to 8 hours. The $CoFe_2O_4$ samples have good magnetism with the highest magnetization saturation Ms = 58.86 emu/g, remanence magnitude Mr = 16.05 emu/g and coercive field Hc = 663.85 G during the hydrothermal process at 150℃.

3.4. The Morphology of CoFe₂O₄ Grains

The findings in Sections 3.1, 3.2 and 3.3 show that the $CoFe_2O_4$ sample made with the molar ratio of $Co^{2+}:Fe^{3+}$ = 1:2.2 at 150℃ during 2 hours gives the best magnetic properties; therefore, we chose this sample to examine the morphology of the $CoFe_2O_4$ sample (Figure 7). The results show that the morphology of the $CoFe_2O_4$ sample particle is spherical, nanorod-shaped with the size ranged from 13-18nm, which is suitable for calculating by the Scherrer equation.
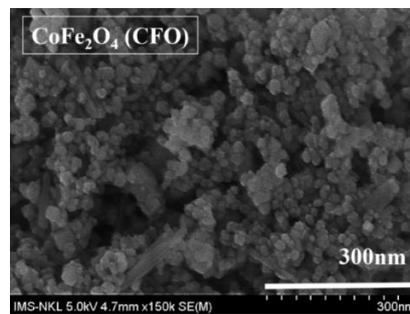
Figure 7. FE-SEM image of the $CoFe_2O_4$ sample.

The $CoFe_2O_4$ particles obtained from the experimental process show that the result of the synthesis is equivalent to or better than the results by some other recent authors. Table 4 provides comparative data on particle size and magnetic properties of as-prepared $CoFe_2O_4$ ferrite particles and published results by some other authors.

Table 4. Values of the $CoFe_2O_4$ particle magnetic properties in recent researches.

No.	Material	Method	d (nm)	Ms (emu/g)	Author
1	$CoFe_2O_4$	Hydrothermal	13-18	58.86	This work
2	$CoFe_2O_4$	Hydrothermal	21.32	53.48	L.T. Tam [21]
3	$CoFe_2O_4$	Hydrothermal	30	22-30	Chao quan Ho [22]
4	$CoFe_2O_4$	Hydrothermal	50	~ 50	Nhan, D. T. T [23]
5	$CoFe_2O_4$	Chemical precipitation	10-25	42.38	Al Lehyani [24]
6	$CoFe_2O_4$	Chemical coprecipitation	20-30	61.77	Zhenfa.zi [25]
7	$CoFe_2O_4$	Wed Chemical route	15-48	68	Maaz K. [26]

4. Conclusions

In this study, $CoFe_2O_4$ spinel nanoparticles were successfully synthesized by the hydrothermal method. The effect of molar ratio, reaction temperature, and reaction time on the crystal structure, morphology, and magnetic properties of $CoFe_2O_4$ materials was studied. The crystallite size calculated by the Scherer formula increased from 13 to 18nm under different synthesis conditions. The synthesized

material has the best saturation magnetization Ms = 58.86 emu/g when fabricated with molar ratio $Co^{2+}:Fe^{3+}$ = 1: 2.2 at 150°C for 2 hours.

Acknowledgments

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