Application of $[M(en)_3]_3[Fe(ox)_3]_2$ ($M = Zn, Cd, Ni$) complexes as new precursor for the synthesis of ferrite micro/nanostructures

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Abstract

This work presents solid-state thermal decomposition synthesis of transition metal ferrites (MFe_2O_4 , $M = Zn, Ni, Cd$). In this method, binary complexes of $[Zn(en)_3]_3[Fe(ox)_3]_2$, $[Cd(en)_3]_3[Fe(ox)_3]_2$ and $[Ni(en)_3]_3[Fe(ox)_3]_2$ (where en = ethylenediamine and ox = oxalate) are introduced as new single-source precursors to fabricate $ZnFe_2O_4$, $CdFe_2O_4$ and $NiFe_2O_4$ micro/nanostructures, respectively. X-ray powder diffraction (XRD) patterns show that pure $CdFe_2O_4$ and $NiFe_2O_4$ are formed by thermal decomposition of the single-source precursors at 800 °C. When thermal decomposition of $[Zn(en)_3]_3[Fe(ox)_3]_2$ complex is carried out at 700 and 800 °C, the final products are composed of $ZnFe_2O_4$ and metal oxides. It is interesting that pure ferrite materials are not formed by using metal salts instead of the binary complexes. Additionally, the particles prepared by using metal salts are highly agglomerated, and it is difficult to measure the individual particle size. The magnetic properties of the products measured by vibrating sample magnetometer (VSM) indicate that the zinc ferrite synthesized at 800 °C is superparamagnetic while the cadmium and nickel ferrites fabricated at 800 °C have ferromagnetic nature.

Keywords: Ferrite; Nanostructures; Thermal decomposition; Complex; Electron microscopy.

1. Introduction

Transition metal ferrites (MFe_2O_4 , $M = Ni, Cd, Zn, etc$) with spinel structure are attractive owing to their extensive applications in electronics [1], ferrofluids [2], magnetic resonance imaging (MRI) [3], drug delivery systems [4], hyperthermia treatments [5], catalysts [6] and gas sensors [7]. Among the ferrites, zinc and cadmium ferrites ($ZnFe_2O_4$, and $CdFe_2O_4$ shorted as ZFO and CFO) have normal spinel structure. The spinel structure contains eight tetrahedral coordination sites and sixteen octahedral coordination sites for the occupancy of divalent and trivalent cations, respectively [8]. In the normal spinel structure of ZFO and CFO, divalent zinc and cadmium cations occupy tetrahedral sites, but trivalent iron cations occupy octahedral sites. On the other hand, nickel ferrite ($NiFe_2O_4$, NFO) is an inverse spinel in which half of the ferric (Fe^{3+}) ions fill the tetrahedral sites and the rest ferric ions plus nickel ions occupy the octahedral sites. $NiFe_2O_4$, in the bulk state, possesses ferrimagnetism that originates from the antiparallel orientation of spins between Fe^{3+} ions at tetrahedral sites and Ni^{2+} ions at octahedral sites [9].

Ferrites in nano scales have been produced by a large number of methods. For example, nanocrystalline CFO has been synthesized by high-energy ball milling of CdO and $\alpha-Fe_2O_3$ [10]. Sol-gel combustion synthesis of CFO has been reported by Sutka *et al.* [11]. Anchietia and co-worker synthesized ZFO nanoparticles by solvothermal method using ethylene glycol and 1,4 butanediol as solvent [6]. Among the methods applied for the preparation of nanosized materials, thermal decomposition of coordination compounds is considered as an efficient method to control shape and particle size of the final products [12-15]. So far, thermal decomposition of various binary complexes including $[Co(En)_3][Fe(CN)_6].2H_2O$, $[Co(En)_3]_4[Fe(CN)_6]_3.15H_2O$ [16] and $[Co(en)_3][Fe(CN)_6].xH_2O$ [17, 18] led to the preparation of transition metal ferrites.

In the present work, for the first time, the $[Zn(en)_3]_3[Fe(ox)_3]_2$, $[Cd(en)_3]_3[Fe(ox)_3]_2$ and $[Ni(en)_3]_3[Fe(ox)_3]_2$ binary complex precursors were used to fabricate the $ZnFe_2O_4$, $CdFe_2O_4$ and $NiFe_2O_4$ micro/nanostructures, respectively. Thermal decomposition of the as-synthesized complexes at solid-state was carried out at different temperatures. The

effect of calcination temperature on the morphology and chemical composition of ferrites was studied. The final products were analyzed by several techniques such as SEM, XRD, VSM and EDS, techniques.

2. Experimental

2.1 Materials

$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, ethylenediamine ($\text{NH}_2\text{CH}_2\text{CH}_2\text{NH}_2$) and $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ were purchased from Merck Company (pro-analysis) and used without further purification.

2.2 Synthesis of $\text{K}_3[\text{Fe}(\text{ox})_3] \cdot 3\text{H}_2\text{O}$ complex

To prepare $\text{K}_3[\text{Fe}(\text{ox})_3] \cdot 3\text{H}_2\text{O}$ complex, 10 mmol of $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ was dissolved in 50 mL of distilled water in a 250 mL beaker, and, then, 30 mmol of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ initially dissolved in 50 mL of distilled water was added into the $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ solution under magnetic stirring. After evaporating of the solvent for 2 days, green $\text{K}_3[\text{Fe}(\text{ox})_3] \cdot 3\text{H}_2\text{O}$ crystals were filtered on Buchner funnel [19].

2.3 Synthesis of $[\text{M}(\text{en})_3](\text{NO}_3)_2$ complexes

In order to synthesize $[\text{M}(\text{en})_3](\text{NO}_3)_2$ ($\text{M} = \text{Zn}, \text{Cd}, \text{Ni}$) complexes, 1.0 mmol of metal nitrate salt was first dissolved in 25 mL of distilled water in a 250 mL beaker, and, then, 3.0 mmol of ethylenediamine was added into the metal nitrate solution. The beaker was placed in an ice bath to induce the $[\text{M}(\text{en})_3](\text{NO}_3)_2$ crystal precipitation. Afterward, the white $[\text{Zn}(\text{en})_3](\text{NO}_3)_2$ and $[\text{Cd}(\text{en})_3](\text{NO}_3)_2$ crystals, and purple $[\text{Ni}(\text{en})_3](\text{NO}_3)_2$ crystals were filtered on Buchner funnel [20].

2.4 Synthesis of $[\text{M}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$ binary complexes

In a typical procedure, 3.0 mol of the as-synthesized $[\text{M}(\text{en})_3](\text{NO}_3)_2$ complex and 2.0 mol of $\text{K}_3[\text{Fe}(\text{ox})_3] \cdot 3\text{H}_2\text{O}$ complex were separately dissolved in 50 mL of distilled water, and, finally, the $\text{K}_3[\text{Fe}(\text{ox})_3] \cdot 3\text{H}_2\text{O}$ solution was gradually added into the $[\text{M}(\text{en})_3](\text{NO}_3)_2$ solution to form $[\text{M}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$ binary complexes. At last, the obtained precipitates were collected by filtration, washed three times with distilled water, and dried at 50 °C for 5 h in vacuum.

2.5 Solid-state thermal decomposition of binary complexes

In a typical synthesis, 0.3 g of the as-prepared $[M(en)_3]_3[Fe(ox)_3]_2$ was loaded into a platinum crucible, and, then, it was placed in oven and heated at the rate of 5 °C/min in the air. Thermal decomposition process was carried out at two different temperatures (700 and 800 °C) to fabricate ferrite materials. According to Table 1, the samples ZFO3-4, CFO3-4 and NFO3-4 have been formed by thermal decomposition of the metal salts as precursors.

2.6 Solid-state reaction between metal salts (blank tests)

To examine the role of precursor on the morphology and chemical composition of ferrites, solid-state thermal decomposition process was done by using metal salts ($Zn(NO_3)_2 \cdot 6H_2O$, $Cd(NO_3)_2 \cdot 4H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 6H_2O$) instead of the binary complexes at the same conditions. For this purpose, $Zn(NO_3)_2 \cdot 6H_2O$, $Cd(NO_3)_2 \cdot 4H_2O$ and $Ni(NO_3)_2 \cdot 6H_2O$ were separately mixed with $Fe(NO_3)_3 \cdot 6H_2O$ in 3:2 molar ratio. The obtained mixtures were ground for 30 min. The resultant semisolid-state mixtures were heated at 700 and 800 °C for 4 h. According to Table 1, the samples ZFO3-4, CFO3-4 and NFO3-4 have been formed by thermal decomposition of the metal salts as precursor.

2.7 Characterization

Fourier transform infrared (FT-IR) spectra of the complexes were recorded on Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range of 400–4000 cm^{-1} . The as-produced metal ferrites were characterized by X-ray diffraction (XRD, X'PertPro, Philips) and scanning electron microscopy (SEM, HITACHI S4160, Japan). The magnetic properties of the samples were detected at room temperature using a vibrating sample magnetometer (VSM, Meghnatis Kavir Kashan Co., Kashan, Iran).

3. Results and discussion

3.1 Characterization of the as-synthesized $[M(en)_2]_3[Fe(ox)_3]_2$ binary complexes

To determine the formation of the complexes used as metal precursors, FT-IR spectra of $[\text{Zn}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$, $[\text{Cd}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ and $[\text{Ni}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complexes were taken, and the curves are shown in Fig. 1a-c, respectively. In the FT-IR spectrum of $[\text{Zn}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex, the adsorption peaks in the regions of 1626, 1317, 1026, 800 and 500–620 cm^{-1} are attributed to the C=O stretching, C–O stretching, C–N stretching, CH_2 rocking and Zn–N (or Fe–O) stretching vibrations, respectively [21]. In the FT-IR spectrum of $[\text{Cd}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex, the adsorption peaks at 1674, 1302, 1003, 787 and 500–600 cm^{-1} can be related to the C=O stretching, C–O stretching, C–N stretching, CH_2 rocking and Cd–N (or Fe–O) stretching vibrations, respectively [17]. In the FT-IR spectrum of $[\text{Ni}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex, the adsorption peaks centered at 1682 cm^{-1} can be assigned to the C=O stretching. In addition, the stretching vibrations of C–O and C–N bands in the range of 1000–1280 cm^{-1} , Ni–N and Fe–O bands in the range of 500–600 cm^{-1} are also seen. These observations proved the formation of the binary complexes.

3.2 Characterization of zinc ferrites

Figs. 2a and 2b display XRD patterns of the products synthesized by thermal decomposition of $[\text{Zn}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex at 700 (sample ZFO1) and 800 °C (sample ZFO2), respectively. The XRD pattern of sample ZFO1 (Fig. 2a) indicates the formation of cubic phase ZnFe_2O_4 (in majority) along with hexagonal phase ZnO (in minority). On the other hand, the reflection peaks of sample ZFO2 (Fig. 2b) show the formation of cubic phase ZnFe_2O_4 plus rhombohedral phase Fe_2O_3 . However, when the temperature is increased to 800 °C, the ZnO phase disappears, and the Fe_2O_3 phase is observed. Similar results have been found about the synthesis of zinc ferrite by Yang and Yen [22] and Ping *et al.* [23]. Figs. 2c and 2d show XRD patterns of the products synthesized by thermal decomposition of metal salts at 700 (sample ZFO3) and 800 °C (sample ZFO4), respectively. When the thermal decomposition process was carried out using $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ salts instead of the binary complexes, $\text{ZnO}/\text{Fe}_2\text{O}_3/\text{ZnFe}_2\text{O}_4$ and $\text{Fe}_2\text{O}_3/\text{ZnFe}_2\text{O}_4$ composites were formed. From comparison of the XRD patterns of the products synthesized by $[\text{Zn}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex (Figs. 2a and 2b) and metal salts (Figs. 2c and 2d), it is evident that the formation of

ZnFe_2O_4 decreases by using metal salts, especially at lower temperature. This phenomenon is introduced as disadvantage of using metal salts as precursor.

SEM images of samples ZFO1 and ZFO2 prepared at 700 and 800 °C using the $[\text{Zn}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$ complex are seen in Figs. 3a and 3b, respectively. Formation of very fine and homogeneous particles in sizes is clear in these images. Dominantly, the particles of samples ZFO1 and ZFO2 range from 8–10 nm and 12–15 nm, respectively. Figs. 3c and 3d show SEM images of samples ZFO3 and ZFO4 fabricated at 700 and 800 °C by using metal salts (blank tests), respectively. When the blank test was carried out at 700 °C, the particle sizes were in the range of 50–150 nm (Figs. 3c). By the blank test temperature increase from 700 to 800 °C, the $\text{Fe}_2\text{O}_3/\text{ZnFe}_2\text{O}_4$ bulk has been formed (Figs. 3d). Finally, it is concluded that nano-sized particles with narrow size distribution can be obtained using $[\text{Zn}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$ complex as precursor.

3.3 Characterization of cadmium ferrites

The XRD patterns of the products synthesized by thermal decomposition of $[\text{Cd}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$ complex at 700 (sample CFO1) and 800 °C (sample CFO2) are shown in Figs. 4a and 4b, respectively. The reflection peaks of sample CFO1 (Fig. 4a) can be attributed to cubic phase CdFe_2O_4 and CdO . As shown in Fig. 4b, the reflections of sample CFO2 are in excellent accordance with the standard pattern of cubic phase CdFe_2O_4 (JCPDS No. 01-1083), demonstrating the pure-phase product formation. XRD patterns of the products prepared by $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ salts (blank tests) at 700 (sample CFO3) and 800 °C (sample CFO4) are seen in Figs. 4c and 4d, respectively. The XRD patterns of these samples can be attributed to CdFe_2O_4 (in minority) and metal oxides (in majority). By comparing the XRD patterns of the products prepared at different conditions (Fig. 4), it can be seen that the main products formed by thermal decomposition of metal salts at 800 °C are metal oxides. Nevertheless, the metal salts thermal decomposition at lower temperature (700 °C) led to the cadmium ferrite formation.

Figs. 5a and 5b show SEM images of samples CFO1 and CFO2 synthesized by $[\text{Cd}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex at 700 and 800 °C, respectively. As shown in Fig. 5a, aggregation of very fine grains led to the preparation of ununiformed particles in particle sizes. The particle sizes of sample CFO1 are between 30–80 nm. Further increase in temperature under the same conditions led to the growth of non-smooth rod-like structures. Most of the rods ranged from 40–80 nm in diameter and 250–600 nm in length. The formation mechanism of CdFe_2O_4 nanorods can be proposed as follows. There are mainly two formation mechanism for rod-like nanostructures. One is ostwald ripening and the other is oriented attachment [24, 25]. According to the ostwald ripening model, in a particle system with different sizes, large particles grow at the expense of small ones. On the other hand, oriented attachment model involves spontaneous self-organization of adjacent particles. They share a common crystallographic orientation, followed by joining of these particles at a planar interface. By considering the SEM images presented in Figs. 5a and 5b, it is concluded that the self-organization of the particles formed at 700 °C leads to the preparation of nanorods at higher temperature (800 °C). Based on our observations in Figs. 5c and 5d plus XRD analyses, micro-sized and very high agglomeration particles of metal oxides mixed by a few amounts of CdFe_2O_4 are obtained through thermal decomposition of metal salts.

3.4 Characterization of nickel ferrites

The XRD patterns of the products synthesized by thermal decomposition of $[\text{Ni}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex at 700 (sample NFO1) and 800 °C (sample NFO2) are shown in Figs. 6a and 6b, respectively. All the reflections can be indexed by cubic phase NiFe_2O_4 (JCPDS No.44-1485), indicating the pure nickel ferrite fabrication using $[\text{Ni}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex as precursor. In Fig.6a, only one reflection located at $2\theta = 31.6^\circ$ is related to cubic phase NiO . XRD patterns of the products synthesized by $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ salts (blank tests) at 700 (sample NFO3) and 800 °C (sample NFO4) are seen in Figs. 6c and 6d, respectively. The reflection peaks of these samples can be attributed to NiFe_2O_4 , NiO (at 700 °C) and Fe_2O_3 (at 800 °C).

Figs. 7a and 7b present SEM images of the products synthesized by $[\text{Ni}(\text{en})_3][\text{Fe}(\text{ox})_3]_2$ complex at 700 (sample NFO1) and 800 °C (sample NFO2), respectively. The particle sizes of samples NFO1 and NFO2 are about 18–25 and 30–100 nm, respectively. According to SEM images of samples NFO3 (Fig. 7c) and NFO4 (Fig. 7d), agglomeration and particle sizes of the products increased by using $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ salts as precursors. Additionally, the particle size increased by the temperature increase from 700 to 800 °C.

3.5 Magnetic properties of ferrite materials

The magnetic properties of the as-produced ferrites were studied by a VSM. Figs. 8a-c show the 300 K hysteresis loops of samples ZFO2, CFO2 and NFO2, respectively. As shown in Fig. 8a, the superparamagnetic nature of zinc ferrite is clear. The superparamagnetic nature of zinc ferrite nanocrystals at room temperature has been found by Yao et al [26]. The coercive field (H_c), saturation magnetization (M_s) and remanent magnetization (M_r) values for samples CFO2 and NFO2 were illustrated in Table 2. The poor ferromagnetic nature of cadmium and nickel ferrites is observed in Figs. 8b and 8c. The increased H_c and M_r values of sample NFO2 may be related to the increased particle sizes.

3.6 Possible formation mechanism of ferrites

The formation mechanism of ferrites, which is closely related to diffusion of metal ions, was first studied by Koch and Wagner [27]. Following Wagner model, the formation of ferrites was explained as a result of the counter diffusion of cations within a rigid oxide lattice formed at the interface of constituent oxides [28]. Based on the presented formation mechanisms and the XRD results obtained in this work, we can propose a possible formation mechanism for ferrites. At first, $[\text{M}(\text{en})_3][\text{Fe}(\text{ox})_3]$ complex, as a single-source precursor, decomposes under thermal process and, then, metal oxides including MO ($\text{M} = \text{Zn}, \text{Cd}, \text{Ni}$) and Fe_2O_3 produce as intermediate compounds (reaction 1). Finally, MFe_2O_4 is formed by reaction between Fe_2O_3 and MO (reaction 2). The main stages are seen as follows:

4. Conclusions

In this work, new single-source precursors with general formula of $[M(en)_3]_3[Fe(ox)_3]_2$ (where M = Zn, Cd, Ni and en = ethylenediamine, ox = oxalate) have been introduced to fabricate ferrite micro/nanostructures via a solid-state thermal decomposition process. According to the XRD results, it was found that by increasing the temperature from 700 to 800 °C, solid-state reactions between Fe_2O_3 and MO (M = Cd, Ni) formed by thermal decomposition of the precursors completed, and pure $CdFe_2O_4$ and $NiFe_2O_4$ were obtained. On the other hand, thermal decomposition of $[Zn(en)_3]_3[Fe(ox)_3]_2$ complex at 700 and 800 °C led to the preparation of $ZnFe_2O_4/ZnO$ and $ZnFe_2O_4/Fe_2O_3$ composites, respectively. Besides, it was observed that pure ferrite materials could not be obtained by using metal salts instead of the binary complexes.

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Figure captions:

Fig. 1. FT-IR spectra of the as-synthesized complexes: (a) $[\text{Zn}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$, (b) $[\text{Cd}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$ and (c) $[\text{Ni}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$.

Fig. 2. XRD patterns of zinc ferrites: (a) ZFO1, (b) ZFO2, (c) ZFO3 and (d) ZFO4.

Fig. 3. SEM images of zinc ferrites: (a) ZFO1, (b) ZFO2, (c) ZFO3 and (d) ZFO4.

Fig. 4. XRD patterns of cadmium ferrites: (a) CFO1, (b) CFO2, (c) CFO3 and (d) CFO4.

Fig. 5. SEM images of cadmium ferrites: (a) CFO1, (b) CFO2, (c) CFO3 and (d) CFO4.

Fig. 6. XRD patterns of nickel ferrites: (a) NFO1, (b) NFO2, (c) NFO3 and (d) NFO4.

Fig. 7. SEM images nickel ferrites: (a) NFO1, (b) NFO2, (c) NFO3 and (d) NFO4.

Fig. 8. Magnetization curves of different ferrites: (a) ZFO2, (b) CFO2 and (c) NFO2.

Table 1. Preparation conditions of various metal ferrites by thermal decomposition method.

Table 2. The coercive field (H_c), saturation magnetization (M_s) and remanent magnetization (M_r) values of the ferrites synthesized at 800 °C by $[\text{M}(\text{en})_3]_3[\text{Fe}(\text{ox})_3]_2$ complex.

Table 1

Sample	Precursor	Temperature (°C)	Morphology of product	Chemical composition
ZFO1	Complex	700	Nanoparticles; 8-10	ZnFe ₂ O ₄ and ZnO
ZFO2	Complex	800	Nanoparticles; 12-15	ZnFe ₂ O ₄ and Fe ₂ O ₃
ZFO3	Nitrate salts	700	Nanoparticles; 50-150	ZnFe ₂ O ₄ , ZnO and Fe ₂ O ₃
ZFO4	Nitrate salts	800	Bulk	ZnFe ₂ O ₄ and Fe ₂ O ₃
CFO1	Complex	700	Nanoparticles; 30-80	CdFe ₂ O ₄ and CdO
CFO2	Complex	800	Nanorods; 40-80 (diameter), 250-600 (length)	CdFe ₂ O ₄
CFO3	Nitrate salts	700	Microparticles; Less than 1 µm	CdFe ₂ O ₄ , CdO and Fe ₂ O ₃
CFO4	Nitrate salts	800	Bulk	CdFe ₂ O ₄ , CdO and Fe ₂ O ₃
NFO1	Complex	700	Nanoparticles; 18-25	NiFe ₂ O ₄ and NiO (Trace)
NFO2	Complex	800	Nanoparticles; 30-100	NiFe ₂ O ₄
NFO3	Nitrate salts	700	Nanoparticles; 50-80	NiFe ₂ O ₄ and NiO
NFO4	Nitrate salts	800	Bulk	NiFe ₂ O ₄ and Fe ₂ O ₃

Table 2

Sample	Morphology	H _c (Oe)	M _r (emu/g)	M _s (emu/g)
ZFO2	Nanoparticle	0.00	0.00	3.50
CFO2	Nanorod	38	0.01	0.61
NFO2	Nanoparticle	125	3.50	23.00

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8