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Magnetic Heating of Fe–Co Ferrites: Experiments and Modeling

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Abstract

Magnetic nanoparticle hyperthermia uses magnetically-induced heat to kill cancer cells. In an alternating magnetic field, the induced heat depends strongly on particles' absorption properties. In order to achieve and maintain therapeutic temperatures inside a tumor and to minimize damage to normal tissues due to induced eddy currents, there is a need to develop new magnetic nanoparticles with improved heating characteristics. This paper investigates the magnetic heating properties of composite iron-cobalt ferrite nanoparticles $\text{Co}_x\text{Fe}^{\text{II}}_{1-x}\text{Fe}^{\text{III}}_2\text{O}_4$ with $0 \leq x \leq 1$. These composite materials are synthesized using a precipitation method. First, the Fe-Co nanoparticle synthesis is described, then their structure, size, magnetic and heating properties are measured and analyzed. The resulting nanoparticles were treated at temperatures 100–600°C in order to study any structural transformations and changes of physical properties. Finally, an empirical model is used to calculate both the nanoparticles' coercivity and their specific absorption rates for different Co concentrations.

Keywords

Fe–Co ferrites; hyperthermia; magnetic nanoparticles; modeling

1. Introduction

Nanoparticle magnetic hyperthermia has emerged as a promising technology^{1–4} for the treatment of cancers. Recent studies suggest that some cancer cells can accumulate magnetic nanoparticles (MNPs) preferably compared to normal cells.^{3,5} During magnetic hyperthermia treatment, magnetic MNPs are conveyed inside tumor cells, and then activated using an alternating magnetic field (AMF). The MNPs absorb the magnetic field energy and turn it into heat. This heat kills the tumor cells although the exact mechanism is unclear. Delivering therapeutic doses of MNPs inside cells remains one of the major challenges faced by magnetic hyperthermia technology. Thus, there is a need to develop new MNPs that will provide desirable therapeutic effects during cancer treatment with a minimum number of MNPs and at lower field strengths in order to avoid eddy current heating.⁶

In the present work, we synthesized Fe–Co ferrites and characterized their structure, magnetic behavior and heating properties. We also modeled their heating in a high frequency AMF. The main objective of this paper is to investigate the heating behavior of

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$\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$ MNPs. Recent studies^{6–9} have shown that small additions of Co can double the specific absorption rate (SAR), which is a measure of the amount of magnetic field energy converted into heat, of $\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$ in an AMF at a frequency of 425 Hz and at field strengths of 200–1000 Oe.

The conversion of magnetic field energy into heat by nanoparticles can arise through different processes: (1) hysteresis, (2) Néel relaxation, (3) Brownian motion and (4) frictional losses in viscous suspensions.¹⁰ This conversion depends on both the nanoparticle size and composition.¹¹ Studies show that the SAR can vary significantly in the nanoparticle size range from 5–100 nm that is of interest for hyperthermia treatment. Since, there is no universal theory that describes the specific power loss mechanisms of MNPs, in this paper we used an empirical model¹⁰ to calculate the SAR for different $\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$ materials under different applied AMF strengths at high frequencies.

2. Experimental

2.1. Synthesis of nanoparticles

MNPs of Fe–Co ferrites ($\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$) with $x = 0, 0.02, 0.05, 0.1, 0.2, 0.5$ and 1 were synthesized using a classical precipitation method.¹² Briefly, a mixture of 10% solutions of both FeCl_3 and FeSO_4 (or CoSO_4) in the ratio 2.1 was added to a 25% ammonia solution under vigorous stirring. The resulting black precipitate was washed three times using magnetic decantation. Particles were dried in air at 20°C and subsequently heated to 100°C, 200°C, 300°C, 400°C, 500°C or 600°C for 1 h in air in order to study any phase transformations and change in particle size.

2.2. Characterization

Bright field (BF) transmission electron microscope (TEM) images of the nanoparticles, fixed on a carbon-coated copper grid, were taken using a FEI Tecnai F20ST field emission gun TEM operated at 200 kV. For each particle size distribution 500 particles were measured. X-ray diffraction (XRD) measurements were performed using a Siemens diffractometer with $\text{Cu-K}\alpha$ radiation (range 2θ from 20–140°, a step size of 0.02°, a scan time of 1 s per step, 40 kV and 300 mA).

The quasi-static magnetic properties of the nanoparticles were determined (saturation magnetization, M_s , remanence magnetization, M_r , and coercivity, H_c) from hysteresis loop measurements using a Lakeshore model 7300 vibrating sample magnetometer (VSM).

The SAR was calculated based on temperature measurements recorded when the samples were subjected to AMF strengths of 200, 300, 400, 500 and 600 Oe at a frequency of 166 KHz applied using a Huttinger TIG 10/300 generator (Huttinger, Freiburg, Germany) coupled to a Fluxtrol Inc. water-cooled coil set at 20°C (Fluxtrol Inc, Auburn Hills, MI). The water-cooled copper solenoid coil had a 32 mm inner diameter and was 44 mm in length. A fiber optic temperature probe (FISO, Inc., Quebec, Canada) was positioned to measure the temperature in the sample. The sample was placed at the middle of the coil, where the field strength was greatest. The field there is uniform for ~6 cm of the 7 cm length of the coil (falling at the ends). The temperature was recorded at 1 s interval throughout the

experimental period and monitored in real-time via software supplied by the FISO temperature monitoring system.

3. Results and Discussion

3.1. Size and shape

TEM observations of the MNPs with 0–50 at.% Co (Fig. 1) shows that they have average magnetic core diameters of 8–9 nm. The size distribution is relatively narrow: for example, 50% of the MNPs (Co-free sample) have sizes 8 ± 1 nm; 90% particles have size 8.5 ± 2.5 nm. For other MNPs, a similar distribution was found. The only MNPs that differed from this range are those with 100% Co ($x = 1$) for which most crystals were smaller at 5–6 nm.

The MNPs of all compositions had irregular shapes (Fig. 1). We found that the sizes of the MNPs increased rapidly as the temperature of the subsequent heat treatment increased. Nanoparticles up to 70 nm were produced by sintering for $x = 0$ and up to 40 nm for $x = 1$ (Figs. 1 and 2). Broadening of the particle size distribution accompanied the increase in particle size (Figs. 1 and 2). The MNPs had irregular shapes both before and after the heat treatment. Treatment at 400°C slightly increased the particle size from the as-synthesized size. A stronger effect of particles sintering is observed for the 600°C treatment, with the greatest influence on the particles with $x = 0$ and $x = 1$ when the average particle sizes increased to 49 nm and 25 nm, respectively.

3.2. Structure

The formation of unalloyed magnetite, cobalt-doped magnetite and the effect of calcinations were investigated using XRD. Representative XRD patterns showing the effect of cobalt substitution on the ferrite are shown in Fig. 3. The crystal structure of both the undoped and doped ferrite was f.c.c. in agreement with ICDD card No. 01-075-1609. Additions of Co up to $x = 0.5$ did not affect the crystal structure of $\gamma\text{-Fe}_2\text{O}_3$ (Fig. 3). For $x = 1$ there are wide peaks at the locations corresponding to the CoFe_2O_4 (ICDD card No. 00-0003-0864). The size of the precipitated particles with $0 < x < 0.5$, calculated using the Scherrer formula¹³ from the widths of the peak with highest intensity (35.6°), was estimated to be 13–18 nm. The XRD pattern of the cobalt-doped magnetite with $x = 1$ shows a very fine particle size of ~5 nm.

The diffraction peaks become narrower as the annealing temperature was increased from room temperature to 600°C (Figs. 4–6) indicating that the particle sizes of all samples increased with increasing temperature due to sintering (Fig. 1). For MNPs with $0 < x < 0.5$ increasing the calcination temperature above 400°C produced new peaks at 24.1° , 33.1° , 40.9° , 49.5° and 64.0° , indicative of the transformation from $\gamma\text{-Fe}_2\text{O}_3$ to $\alpha\text{-Fe}_2\text{O}_3$. On comparing the relative intensities of peaks at 33.2° and 35.6° for samples with $0 < x < 0.5$ (Figs. 4 and 5) it is evident that as x decreases, the more the transformation proceeds to completion. For the sample with $x = 1$ the phase transformation did not occur at all (Fig. 6). Thus, for $x = 0$ and $x = 1$, the crystal structures are f.c.c. and inverse spinel structure, respectively.

3.3. Magnetic properties

Figure 7 shows the room temperature magnetic behavior of MNPs with different amounts of Co. For the MNPs with $x = 0$, superparamagnetic behavior was found for annealing temperatures up to 400°C. Above 400°C, the γ -Fe₂O₃ transforms to the nonmagnetic α -Fe₂O₃.

The M_s of the MNPs increased with increasing crystal size up to 20–30 nm depending on the composition,¹⁴ see Fig. 7. The transformation from γ - to nonmagnetic α -phase (Figs. 4 and 5) starts upon annealing above 400°C. Thus, two processes occur during sintering, i.e., an increase in particle size and a phase transformation. This explains why nanoparticles heat-treated at 400°C have the highest M_s of the MNPs studied here, i.e., they have the largest particle size and are γ -Fe₂O₃.

The M_s for all the precipitated samples was in the range 25–55 emu/g. Interesting behavior was observed for the mNPs with $x = 1$ (CoFe₂O₄). The as-precipitated nanoparticles show a very small M_s . However, annealing at > 400°C increased M_s dramatically (Fig. 8). This was likely due to the dramatic increase in particle size.¹⁴

The sample with 50% Co had a maximum M_s of 500 Oe (Fig. 8). Probably two competitive processes are occurring: (1) the increase in crystal size increases the M_s ; (2) the transformation of γ -Fe₂O₃ to nonmagnetic α -Fe₂O₃ decrease M_s . Since M_s increases, this means that the particle agglomeration has a higher impact on M_s than the transformation of γ -Fe₂O₃ to α -Fe₂O₃. Samples treated at 600°C have good correlation between M_s and the value of x . Thus, samples with $x = 1; 0.5; 0.2; 0.1$ and 0 have 100%, 59%, 15%, 7% and 1% of the M_s value of the sample with $x = 1$. After annealing at 600°C, particles with $x < 0.2$ particles show paramagnetic behavior while for $x \geq 0.2$, they show ferromagnetic behavior.

The properties of MNPs can change in biological fluids. Since Fe–Co ferrites could potentially be used for the magnetic hyperthermia treatment, we dispersed the Fe–Co ferrite MNPs in a saline solution (1 wt.%) and measured their magnetic properties before and afterwards. As shown in Fig. 9, neither the M_s nor the coercivity of the Fe–Co ferrite MNPs changed with after immersion in saline solution for 1 h, which is usually enough time to inject nanoparticles and perform hyperthermia.

3.4. SAR

The SAR was measured for the Co_{*x*}Fe_{1-*x*}Fe₂O₄ MNPs both as-produced and after various heat treatments, see Fig. 10. The data shows that the SAR depends on both the annealing temperature and the applied field strength. In most cases, increasing the field strength results in an increase in the SAR value: the dependence of the SAR on the Co content is not linear. A small addition of Co increases SAR significantly, but the effect of Co diminishes with increasing amounts. The highest SAR was observed for MNPs with $x = 0.05$ annealed at 400°C and at an AMF of 400 Oe. Further increases in Co content results in particles becoming large and having a wide hysteresis curve (Fig. 7). More detailed explanation of these phenomena is provided in Sec. 4.

4. Modeling

To understand the magnetic properties of Fe–Co ferrites for different compositions, first, we develop an empirical model to describe the H_c of the iron/cobalt MNPs as a function of cobalt content, i.e.,

$$H_{\text{Co}_x\text{Fe}_{1-x}\text{Fe}_2\text{O}_4}^c = H_{\text{Fe}_3\text{O}_4}^c + H_{\text{Co}_1}^c \left(1 - e^{-\frac{1}{\beta} \left(\frac{x}{\gamma} \right)^\alpha} \right), \quad (1)$$

where $H_{\text{Fe}_3\text{O}_4}^c$ and $H_{\text{Co}_1}^c$ are the values of H_c for Fe_3O_4 and Co, respectively, which can be obtained from the limited measured data; $x \in [0,1]$, is the amount of Co; and; α , β and γ are model fit parameters that are determined by fitting equation 1 to the measured data. To demonstrate the applicability of the model, Eq. (1) was fitted to the actual data collected in our lab, as depicted in Fig. 11. The comparison between observed H_c (red dots) and the empirical modeled (blue lines) data for $T = 100^\circ\text{C}$ and $T = 300^\circ\text{C}$ are shown in Figs. 11 and 12, respectively. The results show that the empirical model presented in Eq. (1) matches the observed data (red dots) well, and thus provides the ability to estimate the coercivity H_c for Fe–Co ferrites at any x value.

H_c depends not only on the amount of Co, but also on the mNP size.¹¹ Hergt *et al.*¹⁰ proposed the following empirical model for relating the experimentally observed H_c to the particle size,

$$H_c(D) = H_m \left(\frac{D_1}{D} \right)^{0.6} \left(1 - e^{-\left(\frac{D}{D_1} \right)^5} \right), \quad (2)$$

where D is particle diameter in (nm), and H_m and D_1 are model parameters that are obtained from measured data. In the current work, we assume $H_m = H_{\text{Co}_x\text{Fe}_{1-x}\text{Fe}_2\text{O}_4}^c$.

The H_c of the MNPs is then related to magnetic field loss, i.e., the amount of magnetic field energy converted into heat during magnetization reversal. Finally the specific loss is calculated to be (for more details see Ref. 11,

$$w(H, D) = \begin{cases} \xi D H^3 & \text{for } H \leq H_c, \\ \frac{4B_r H_c(D)}{\rho} \left(1 - \left(\frac{H_c(D)}{H} \right)^5 \right) & \text{for } H > H_c, \end{cases} \quad (3)$$

where H is the applied magnetic field; B_r is remanent magnetic flux density, which can be found experimentally; ρ is the mass density of the magnetic material; and the parameter ξ is a measure of losses of the Rayleigh type that are due to a small fraction of multi-domain particles.

The SAR (in watts per gram of magnetic material) can then be calculated from

$$\text{SAR}(H, D) = w(H, D) f_r, \quad (4)$$

where f_r is the frequency of the exciting field.

Since, the mNP sizes vary significantly, to take into account the size distribution and correctly calculate the SAR, the calculated SAR should be weighed by the particle size distribution $f(D)$,¹¹ i.e.,

$$W(H) = \int w(H, D) f(D) dD. \quad (5)$$

The $f(D)$ mNP distribution function is normalized on a volumetric basis as determined from TEM images see Fig. 1. This is approximated with the gamma distribution function¹¹

$$f(D) = \frac{\pi f_n(D) D^3}{6V} \quad (6)$$

and

$$f_n(D) = \frac{1}{D_o} \left(\frac{D}{D_o} \right)^{\chi-1} e^{-\frac{D}{D_o}} \frac{1}{\Gamma(\chi)}, \quad \chi = \frac{\langle D \rangle}{D_o}, \quad (7)$$

where $\langle D \rangle$ is the mean size, and $\sqrt{\langle D \rangle D_o}$ is the size variance.

To understand the Fe–Co ferrites magnetic heating properties, we measured the SAR for different compositions, see Fig. 10, and also calculated it using Eqs. (2)–(7). Comparisons between the model and the experimentally-measured SARs, for $\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$, $x = 2\%$, MNPs, are shown in Fig. 13. The modeled parameters are as follows: $f_r = 155$ kHz, $H_m = 94$ kA/m, $D_1 = 12$ nm, and $\langle D \rangle = 8.7$ nm, $D_o = 0.2$ nm, $B_r = 0.033$ as determined from the TEM data (Fig. 1) and hysteresis curves (see Fig. 7). The comparison clearly shows that there is a good agreement between the model and actual data, indicating that the model, which takes into account the particle size distribution, H_c and the remanent magnetization, accurately predicts the SAR for Fe–Co ferrites for up to 600 Oe magnetic fields. In addition, the model data predicts SAR saturation above 1000 Oe magnetic fields. However, due to limitations of our AMF system maximum output power, we could not validate the model data experimentally above the 600 Oe magnetic fields. From Eq. (3), it follows that the SAR is directly proportional to the material's H_c . Our experimental data show that as the amount of cobalt increases the H_c of the material increases, see Fig. 9. To illustrate this, we calculated the SAR for different Co additions. The calculated SARs, which are shown in Fig. 14 illustrates that as the amount of cobalt increases the SAR increases, particularly for high AMFs.

These simulations show that while the Co addition increases the SAR of the MNPs, they require high AMF values to achieve desirable therapeutic effects. This makes Fe–Co ferrites less attractive for magnetic field hyperthermia applications.

5. Conclusions

In this paper, the structure, size, magnetic properties and heating behavior under an AMF of Fe–Co ferrite NMPs were studied for different annealing temperatures and different Co concentrations. The studies showed that as the annealing temperature increased from room temperature to 600°C the MNPs sizes increase, and the magnetic behavior changes correspondingly. The MNP size and magnetic saturation have their highest values after annealing at 400°C and behave as superparamagnetic materials below this temperature. This shows a direct link between the MNP size and the magnetic properties. After annealing above 400°C, the NPs are nonmagnetic. It was shown that the Fe–Co MNPs coercivity, and as a consequence the SAR, depends nonlinearly on the Co addition. An empirical model was developed to describe this nonlinear dependence. For an AMF at 160 kHz with the field strength varied from 100–600 Oe, the highest SAR was observed for ferrite with 0.05 Co. The Fe–Co MNP SAR was calculated for higher Co concentrations and high fields using the empirical model. The modeled results illustrated that higher SARs can be achieved for higher Co amounts but only at higher AMF strengths.

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Fig. 1. BF TEM images of $\text{Co}_x\text{Fe}^{\text{II}}_{1-x}\text{Fe}^{\text{III}}_2\text{O}_4$ nanoparticles: (a) $x = 0$, as-synthesized; (b) $x = 0$, 600°C for 1 h; (c) $x = 0.5$, as-synthesized; (d) $x = 0.5$, 600°C for 1 h; (e) $x = 1$, as-synthesized; (f) $x = 1$, 600°C for 1 h.

(a)

(b)

(c)

Fig. 2. Size distribution of MNPs $\text{Co}_x\text{Fe}_{1-x}^{\text{II}}\text{Fe}^{\text{III}}_2\text{O}_4$ for different values of x after various heat treatments: (a) as-synthesized; (b) 400°C for 1 h; (c) 600°C for 1 h (color online).

Fig. 3. X-ray diffractograms of as-synthesized iron-cobalt ferrites $\text{Co}_x\text{Fe}^{\text{II}}_{1-x}\text{Fe}^{\text{III}}_2\text{O}_4$ and standard patterns for Fe_2O_3 (ICDD card 01-075-1609) and CoFe_2O_4 (ICDD card 00-0003-0864) (color online).

Fig. 4. XRD patterns of $\text{Fe}^{\text{II}}_{0.5}\text{Co}_{0.5}\text{Fe}^{\text{III}}_2\text{O}_4$ ($x = 0.5$) nanoparticles showing increase in particle size and greater completion of the phase transformation from $\gamma\text{-Fe}_2\text{O}_3$ to $\alpha\text{-Fe}_2\text{O}_3$ with increasing annealing temperature (1h) (color online).

Fig. 5.

XRD patterns of Fe^{II}Fe^{III}₂O₄ ($x = 0$) nanoparticles as-synthesized and after heat treatment at temperatures of 400°C and 600°C for 1 h. The α -Fe₂O₃ and γ -Fe₂O₃ patterns (ICCD cards 00-024-072 and 01-075-1609, respectively) are also shown (color online).

Fig. 6.

XRD patterns of $\text{CoFe}^{\text{III}}_2\text{O}_4$ ($x = 1$) after heat treatment at various temperatures for 1 h. The graph labeled A is the pattern for CoFe_3O_4 ICDD pdf card No00-0003-0864 (color online).

Fig. 7.
Room temperature magnetization curves for $\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}_2\text{O}_4$ nanoparticles heat treated at temperatures from 100–600°C for 1 h (color online).

Fig. 8. Room temperature saturation magnetization, M_s , of $\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$ nanoparticles as a function of annealing temperature for different amounts of Co (color online).

(a)

(b)

Fig. 9.

Influence of saline treatment for 1 h on the magnetic properties of as-synthesized Fe–Co ferrites with various Co:Fe (II) molar ratios from 0–100%: (a) saturation magnetization, M_S , and (b) coercivity, H_C (color online).

Fig. 10. Measured SAR values for various $\text{Co}_x\text{Fe}_{1-x}\text{Fe}_2\text{O}_4$ MNPs annealed at different temperatures (color online).

Fig. 11.

Comparisons between modeled (solid blue line) and measured (red dots) data for iron/cobalt ferrites versus cobalt concentration at 100°C. The model fit parameters are: $H_{\text{Fe}_3\text{O}_4}^c$ 4 [A/m], H_{Co}^c = 241 [A/m]; α = 3; β = 4; γ = 10 (color online).

Fig. 12.

Comparisons between modeled (solid blue line) and measured (red dots) data for iron/cobalt material versus cobalt concentration at 300°C. The model fit parameters are:

$$H_{\text{Fe}_3\text{O}_4}^c = 4 \text{ [A/m]}, H_{\text{Co}}^c = 241 \text{ [A/m]}; \alpha = 4; \beta = 0.4; \gamma = 100 \text{ (color online).}$$

Fig. 13.
Modeled and measured SAR for $\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$, $x = 2\%$ (color online).

Fig. 14. Modeled SAR versus applied AMF field strength for different $\text{Fe}^{\text{II}}_{1-x}\text{Co}_x\text{Fe}^{\text{III}}_2\text{O}_4$ compositions (color online).