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Research articles

The influence of synthesis parameters on one-step synthesized superparamagnetic cobalt ferrite nanoparticles with high saturation magnetization

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ABSTRACT

In this study, superparamagnetic cobalt ferrite nanoparticles (SCFNs) were synthesized by co-precipitation in one-step. The synthesis parameters; reaction time and stirring rate, were varied separately while the other parameters were fixed constant to investigate the effect of the parameters on the properties of SCFNs. X-ray diffraction analysis and Fourier transform infrared spectroscopy confirmed that synthesized samples are cobalt ferrite. The magnetization consistently increased with the particle size as the reaction time increased and the stirring rate decreased. While the reaction time was effective on the size of the cobalt ferrite nanoparticles, the stirring rate was also found to have influence on the particle size and thus the magnetization of the nanoparticles. The critical size of cobalt ferrite nanoparticles for superparamagnetic limit with zero coercivity and remanence was found to be around 7 nm and its maximum magnetization value was 41.0 emu/g. When the size of the SCFNs went over 7 nm, the magnetization increased with a small coercivity of 2–5 Oe, which may offer a potential usage in magnetic hyperthermia applications. It was seen that structural properties, especially the particle sizes and corresponding magnetic properties of SCFNs were considerably affected by the parameters of stirring rate and especially reaction time. Therefore, it is seen that SCFNs with desirable properties can be tailored by changing the synthesis parameters and therefore may have the potential to use in biomedical applications.

1. Introduction

Cobalt ferrite is an inverse spinel ferrite which has Co^{2+} and Fe^{3+} ions at tetrahedral and octahedral sites. It has a moderate saturation magnetization and high coercivity with good physical and chemical stability [1]. Bulk cobalt ferrite is ferrimagnetic and the saturation magnetization is 80 emu/g at 20 °C [2]. However, nanoscale cobalt ferrite may show superparamagnetic behaviour. The superparamagnetic effect allows changing the coercivity of the material by controlling the particle size. The size limit of superparamagnetic behavior for cobalt ferrite nanoparticles is around 6 nm [3]. Below the size limit, cobalt ferrite nanoparticles show zero coercivity. Due to its different properties, cobalt ferrite can be used in variable areas like high density data storage [4], sensors and actuators [5], magnetic resonance imaging (MRI) [6] and magnetic hyperthermia [7,8]. Cobalt ferrite is an interesting material with high specific absorption rates (SAR) for magnetic hyperthermia and high relaxation time ratios for MRI. Sharifi et al. [9] indicates that cobalt ferrite has higher SAR values than the SAR values of magnetite and manganese ferrite. Superparamagnetic cobalt ferrite nanoparticles with particle size of 8 nm show better relaxation rates (r_1/r_2) than the magnetite and manganese ferrite with similar sizes [10].

A large number of synthesis methods have been used to obtain cobalt ferrite nanoparticles, such as; hydrothermal synthesis [11], microwave assisted hydrothermal method [12], thermal decomposition [13], microemulsion [14] and sol-gel method [15]. However, these methods require long reaction times, multiple steps, surfactants or high pressure and temperature. On the other hand co-precipitation is simpler and takes place at ambient conditions in shorter times. It is also available to tailor the properties of magnetic nanoparticles by controlling the parameters of co-precipitation. The effects of some parameters were investigated [16–22]. Prabhakaran et al. [16] synthesized cobalt ferrite nanoparticles at 60, 70 and 80 °C by co-precipitation and presented the effect of reaction temperature on the properties of the

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nanoparticles. The nanoparticles obtained in the study shows saturation magnetizations from 6.41 to 20.0 emu/g and coercivities between 345 and 700 Oe. Chinnasamy et al. [17] investigated the effects of reaction temperature, NaOH concentration and feeding rate of metal ions into the alkali solutions on the properties of cobalt ferrite nanoparticles. The authors obtained cobalt ferrite nanoparticles with high coercivity values (301–2290 Oe). Kuruva et al. [18] studied the effect of the pH of the solution as well as the sintering temperature. The sintered nanoparticles have saturation magnetizations in the range of 47.9–87.9 emu/g and coercivity in the range of 69–400 Oe. In [19], cobalt ferrite nanoparticles were synthesized at different reaction times (0.5–6.5 h) and the effect of reaction time was presented. Cobalt ferrite nanoparticles with coercivities were obtained. In some studies [20–22], co-precipitation was followed by heat treatment and the effects of post-synthesis parameters were also investigated. As seen in above studies [16–22], the effects of reaction temperature, pH and reaction time on cobalt ferrite nanoparticles with coercivity were studied. The influence of the parameters on superparamagnetic cobalt ferrite nanoparticles (SCFNs) with no coercivity and remanence needs to be investigated. Thus, in this study, the SCFNs were synthesized by co-precipitation in one-step, and the effect of reaction time and stirring rate on the properties of the SCFNs were studied. The SCFNs with high magnetizations were obtained. Maximum magnetization value for cobalt ferrite nanoparticles with zero coercivity and remanence is 41.0 emu/g. The critical size limit of cobalt ferrite nanoparticles for superparamagnetism with zero coercivity was found to be around 7 nm. Also, higher magnetization values of superparamagnetic nanoparticles were obtained with small coercivities of 2–5 Oe.

2. Experimental

2.1. Materials and synthesis

Cobalt ferrite nanoparticles were synthesized by co-precipitation. Cobalt chloride hexahydrate ($CoCl_2{\cdot}6H_2O$, Fluka > 98%) and ferric chloride hexahydrate ($FeCl_3{\cdot}6H_2O$ Sigma Aldrich > 99%) were used for the synthesis of the nanoparticles. 0.07 mol $CoCl_2{\cdot}6H_2O$ and 0.14 mol $FeCl_3{\cdot}6H_2O$ were dissolved in 100 ml distilled water to obtain ion solution. Then, 100 ml of ammonium hydroxide solution (NH_4OH, Merck %25) was added to the ion solution under vigorous stirring. The reaction was performed at 80 °C by using water bath. The synthesis took place in air atmosphere. In order to investigate the effect of reaction time, time was changed between 5 and 210 min while the other parameters were fixed. The sample obtained at 5 min was named as T5 and the others were named similarly. The effect of stirring rate was also studied by changing the stirring rate from 100 to 1850 rpm. The samples obtained from the synthesis were labeled as R100, R500, R1000, R1500 and R1850 due to the stirring rates used for the process, see Table 1. After the reaction, the precipitate was washed three times with distilled water and dried in an oven at 60 °C to obtain powders.

2.2. Characterizations

X-ray diffraction (XRD) technique was used to analyze the crystal structure of cobalt ferrite nanoparticles and to calculate the crystal sizes of them. CuK_α radiation ($\lambda = 1.54056$ Å) was used between the angles (2θ) 20° and 80° using PANanalyticals X'PertPRO X-ray diffractometer. Fourier transform infrared spectroscopy (FT-IR) was used to obtain transmission spectrums. Scan was performed between 400 and 4000 cm^{-1}. The region between 400 and 1500 cm^{-1} was given in the spectrum since the characteristic metal-oxygen (M − O) vibration bands appear in this region. FT-IR analysis was performed with Perkin Elmer spectrometer by using KBr pellets. Transmission electron microscope (TEM, FEI TECHNAI G2 F30) was also used for the structural characterization. Particle sizes were calculated by using Image J software. Magnetic characterization was made by using vibrating sample magnetometer (VSM, ADE EV9). Measurements were made at room temperature at ± 20 kOe (1 Oe intervals). Magnetic particle sizes were also calculated by using the equation mentioned in [23]. Maximum magnetizations of cobalt ferrite nanoparticles were denoted as M_{max} in the text at the highest field of ± 20 kOe with under laboratory conditions.

Table 1
Synthesis conditions, particles sizes and magnetic properties of cobalt ferrite nanoparticles.

	Synthesis Conditions		Particle Size (nm)			Magnetic properties	
	Reaction time (min)	Stirring rate (rpm)	d_{TEM}	d_{XRD}	d_{VSM}	M_{max}* (emu/g)	H_c (Oe)
T5	5		3.0 ± 0.9	4.7	3.7 ± 0.3	12.7	–
T10	10		–	–	4.4 ± 0.2	18.3	–
T20	20		–	–	5.0 ± 0.4	23.0	–
T30	30		–	–	5.5 ± 0.5	26.9	–
T60	60	1000	–	–	5.7 ± 0.6	33.0	–
T90	90		6.0 ± 1.7	6.7	5.8 ± 0.6	35.5	–
T120	120		–	–	–	40.7	3
T150	150		–	–	–	39.9	2
T180	180		–	–	–	43.3	5
T210	210		7.2 ± 2.5	7.1	–	44.3	2
R100		100	6.2 ± 1.7	6.7	6.1 ± 0.7	41.0	–
R500		500	–	–	6.0 ± 0.7	38.8	–
R1000 (T-90)	90	1000	6.0 ± 1.7	6.7	5.8 ± 0.6	35.5	–
R1500		1500	–	–	5.5 ± 0.6	35.3	–
R1850		1850	4.6 ± 0.9	6.0	5.5 ± 0.7	35.6	–

* Magnetization value at maximum applied field.

3. Results and discussion

3.1. Reaction time effect

The SCFNs and corresponding synthesis conditions were given in Table 1. The XRD patterns of the samples T5, T90 and T120 were presented in Fig. 1. In the XRD pattern of sample T5 synthesized at 5 min, only (3 1 1) peak at 2θ ≈ 35.3° is observed. The peak corresponds to cobalt ferrite according to the JCPDS card no. 22-1086. In the patterns of the other samples in Fig. 1, (2 2 0), (4 0 0), (5 1 1) and (4 4 0) peaks of cobalt ferrite are also observed at 2θ ≈ 30.3, 42.9, 56.9 and 62.9° besides the (3 1 1) peak. The intensity of the peaks increases with the increase of reaction time. The particle size of cobalt ferrite nanoparticles, d_{XRD} were calculated according to the Scherrer equation [24] and listed in Table 1. The most intense peak (3 1 1) was used for the

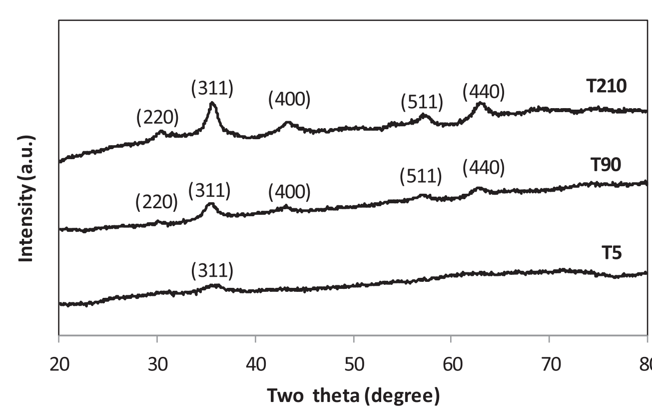

Fig. 1. XRD patterns of the samples synthesized at 5 min. (T5), 90 min. (T90) and 210 min (T210).

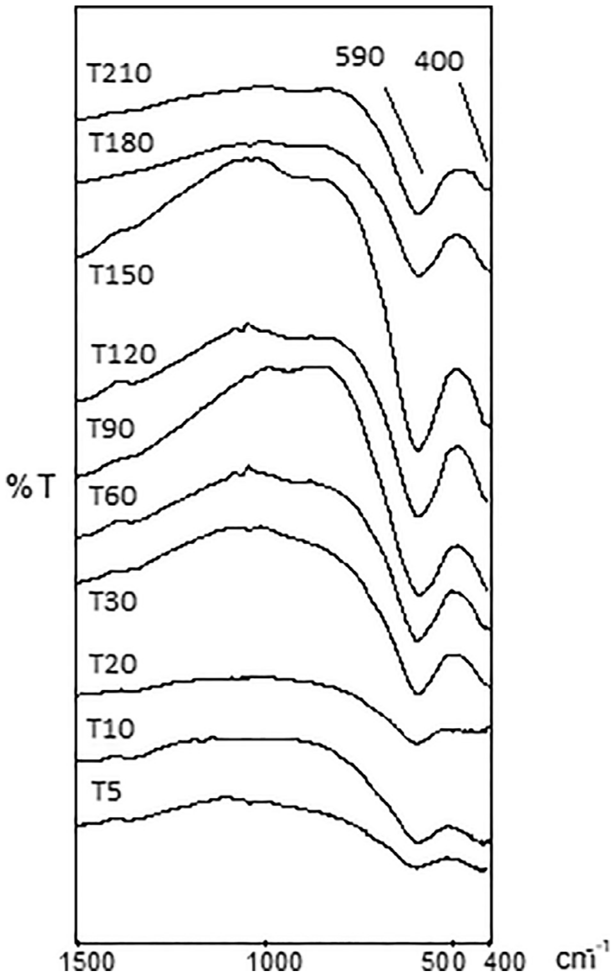

Fig. 2. FTIR spectrum of the samples synthesized at different times.

calculation. d_{XRD} of the nanoparticles for samples T5, T90 and T210 are 4.7, 6.7 and 7.1 nm, respectively. Reaction time is found to be effective on the particle size since the particle size of cobalt ferrite nanoparticles increases with the increase of reaction time.

In order to confirm and identify the cobalt ferrite structure, FTIR spectroscopy was used and the spectra were given in Fig. 2 in the range of 400–1500 cm^{-1}. In the spectrum of all samples, two transmittance peaks can be observed at around 400 cm^{-1} and 590 cm^{-1} which corresponds to the M_o-O and M_t-O vibrations, respectively. M_o indicates the metal ion at the octahedral site and M_t indicates the metal ion at the tetrahedral site [25]. Two peaks at 400 cm^{-1} and 590 cm^{-1} confirm the cobalt ferrite structure. The peaks observed in the spectrum of samples T5, T10 and T20 are weaker than the peaks seen in the spectrum of the other samples. Peaks intensify for the nanoparticles synthesized at longer times than 20 min.

Transmission electron microscope (TEM) images of the samples synthesized at 5 min (T5), 90 min (T90) and 210 min (T210) were given in Fig. 3a, b and c, respectively. Physical particle sizes of the samples, d_{TEM} were calculated from the images by using ImageJ and given in Table 1. Also the particle size histograms were given with the related TEM images. In Fig. 3a, a few cobalt ferrite nanoparticles can be seen. The size of these nanoparticles is 3.0 ± 0.9 nm. In both XRD and FT-IR analysis, sample T5 has only weak peaks that indicate the nucleation is low and the nanoparticles are small in size and these results are consistent with the TEM analysis. The sizes of samples T90 and T210 are calculated to be 6.0 ± 1.7 nm and 7.2 ± 2.5 nm, respectively. The size of cobalt ferrite nanoparticles increases as the synthesis time increases (see Table 1).

Magnetic characterizations of cobalt ferrite nanoparticles were made by VSM and the magnetization curves of the samples were given in Fig. 4a and 4b with the insets at ± 30 Oe. The results display that all samples are superparamagnetic with zero or small coercivities. Maximum magnetizations, M_{max} of cobalt ferrite nanoparticles and the coercivity, H_c values can be seen in Table 1. The M_{max} is the magnetization value measured at the maximum applied field (20 kOe). The M_{max} of the sample increases from 12.7 to 35.5 emu/g with the increase of synthesis time from 5 to 90 min. The samples T5-T90 show zero H_c. With the increase of synthesis time above 90 min, the samples start to show small H_c (2–5 Oe). The M_{max} values of the samples with H_c (T120-T210) reach up to 44.3 emu/g. Phong et al. [8] indicates that 13.5 nm cobalt ferrite nanoparticles with H_c (43 Oe) shows higher SAR than bigger nanoparticles (17.8 and 24.2 nm) with higher H_c (378 and 850 Oe). The SCFNs with little H_c values (2–5 Oe) and high magnetizations from 39.9 to 44.3 emu/g in our study may also offer a potential in magnetic hyperthermia applications.

Initial susceptibilities, χ_i of SCFNs were also calculated. The χ_i is calculated from the magnetization curves within the linear response regime in low fields as [26];

$$\chi_i = (dM|dH)_{H\to 0} \quad (1)$$

M is the magnetization and H is the applied field. The χ_i values for samples T5, T10, T20, T30, T60 and T90 are 0.003, 0.006, 0.011, 0.016, 0.022 and 0.025 emu/gOe, respectively. χ_i and M_{max} of SCFNs increase as the reaction time increases. It can be concluded that the magnetic response of the nanoparticles at low fields improves with the increase of χ_i caused by the increase of the reaction time.

The magnetic particle size, d_{VSM} and standard deviation, σ_{VSM} were derived from the following equations [23,27–29];

$$d_{VSM} = \left[\frac{18kT}{\pi m_s}\sqrt{\frac{\chi_i}{3M_sH_o}}\right]^{1/3} \quad (2)$$

$$\sigma_{VSM} = \frac{1}{3}\left[ln\left(\frac{3\chi_i H_o}{M_s}\right)\right]^{1/2} \quad (3)$$

where m_s and M_s are saturation magnetization of the bulk phase (400 emu/cm^3) [30] and magnetic nanoparticles, respectively. The M_s was calculated by considering the density of cobalt ferrite 5.29 g/cm^3 [31]. χ_i is the initial susceptibility $\chi_i = (dM/dH)_{H\to 0}$ and H_o is calculated by extrapolating M to zero at high fields where the relationship between M and 1/H is linear. k is Boltzman constant and T is 298.15 K. The calculation is appropriate for the nanoparticles with zero H_c, thus d_{VSM} of the samples with zero H_c were calculated and listed in Table 1. The magnetic particle size increases from 3.7 ± 0.3 to 5.8 ± 0.6 nm as the synthesis time increases from 5 to 90 min. The increase of the particle size is compatible with the increase of crystal and physical sizes of cobalt ferrite nanoparticles (Table 1). The biggest particle size of cobalt ferrite nanoparticles with zero H_c is 6.0 ± 1.7 nm and 6.7 nm calculated from TEM and XRD data, respectively whereas the particle size of cobalt ferrite nanoparticles with H_c was seen over 7.2 ± 2.5 nm (7.1 nm), see Table 1. Thus it can be concluded that the particle size limit for superparamagnetic cobalt ferrite nanoparticles with zero H_c is around 7 nm. It is compatible with the superparamagnetic size limit presented in [3].

3.2. Stirring rate effect

XRD analysis of the samples synthesized at different stirring rates was performed. The patterns of two samples synthesized at the stirring rates of 100 rpm and 1850 rpm were given in Fig. 5. In the patterns of both samples, (2 2 0), (3 1 1), (4 0 0), (5 1 1) and (4 4 0) peaks are observed at 2θ ≈ 31°, 35°, 43°, 57° and 63°, respectively. According to the JCPDS card no. 22-1086 the samples are cobalt ferrite. The particles sizes, which are calculated from the most intense peak (3 1 1), are 6.7,

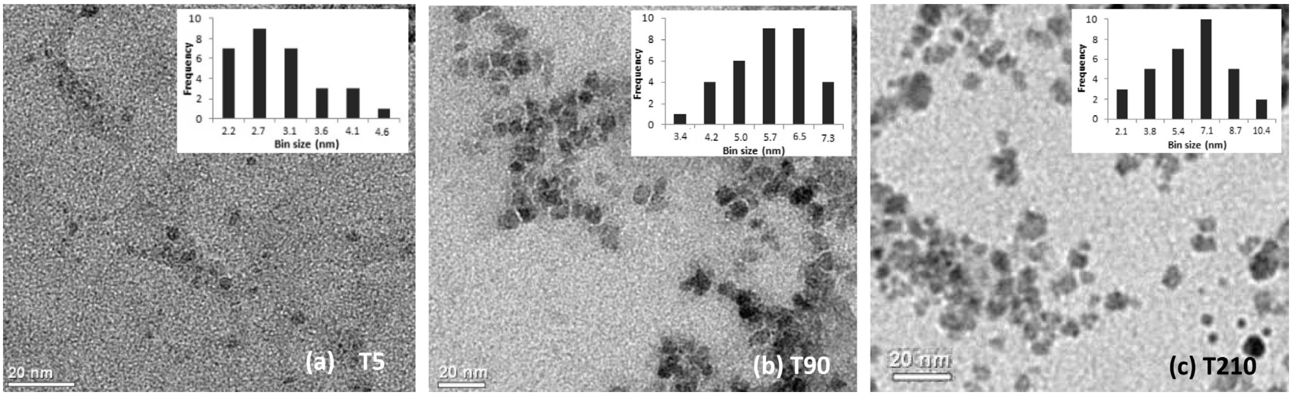

Fig. 3. TEM images of cobalt ferrite nanoparticles synthesized at different reaction times a) 5 min, b) 90 min and c) 210 min.

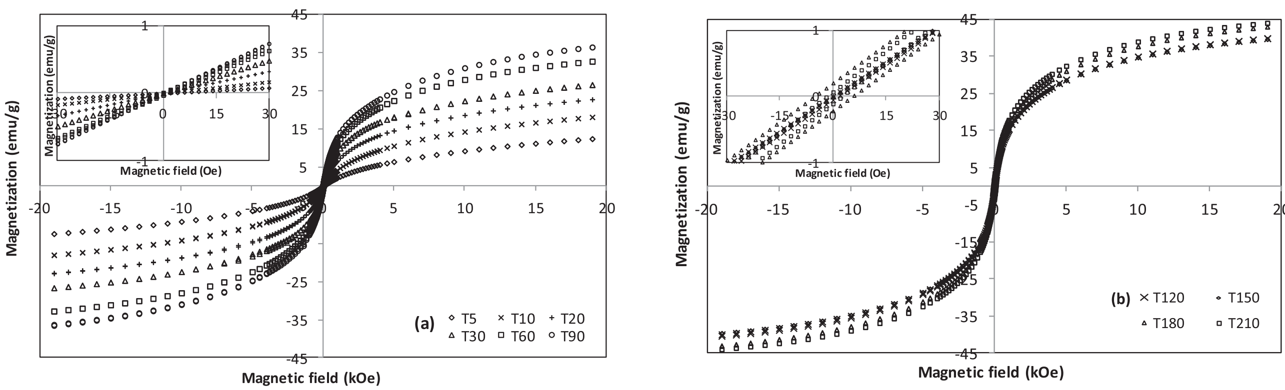

Fig. 4. Magnetization curves of the samples synthesized at different reaction times a) 5, 10, 20, 30, 60 and 90 min, b) 120, 150, 180 and 210 min. (Insets show the magnetization curves at ± 30 Oe.)

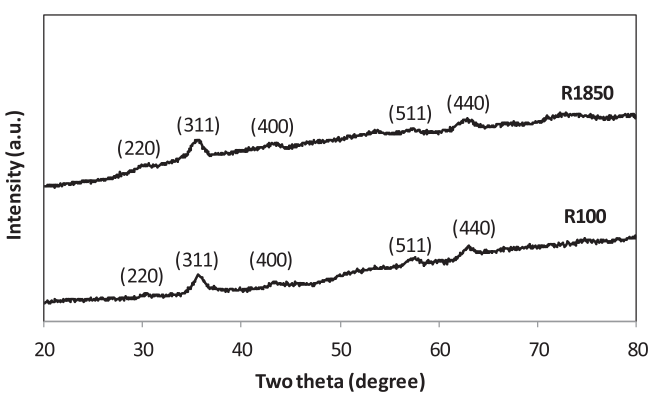

Fig. 5. XRD patterns of the samples synthesized at 100 rpm (R100) and 1850 rpm (R1850).

6.7 and 6.0 nm for the samples R100, R1000 and R1850, respectively. It is seen that the particles size slightly decreases with the increase of stirring rate. FT-IR spectrum of the nanoparticles in Fig. 6 shows the characteristic bands of cobalt ferrite at around 400 cm^{-1} and 590 cm^{-1}.

Particle sizes of cobalt ferrite nanoparticles were calculated from the TEM images and given in Table 1. The particle sizes of the nanoparticles synthesized at 100 rpm (R100), 1000 rpm (R1000) and 1850 rpm (R1850) are 6.2 ± 1.7, 6.0 ± 1.7 and 4.6 ± 0.9 nm, respectively. TEM images of two samples R100 and R1850 are also given in Fig. 7 with the histograms. The trend of the decrease in particle size with the increase of stirring rate is consistent with other studies [32]. The decrease is more remarkable in high stirring rates (1850 rpm).

Magnetization curves of the nanoparticles synthesized at various

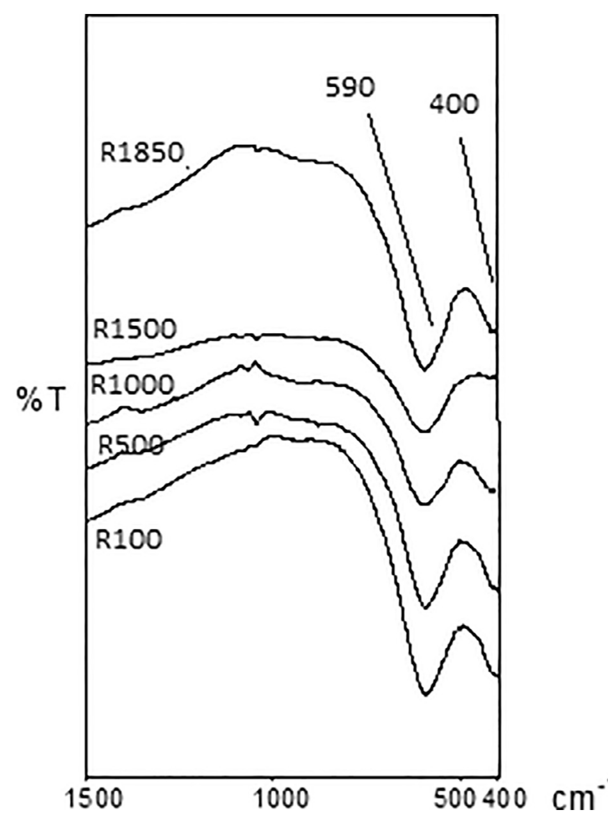

Fig. 6. FTIR spectrum of the samples synthesized at different stirring rates.

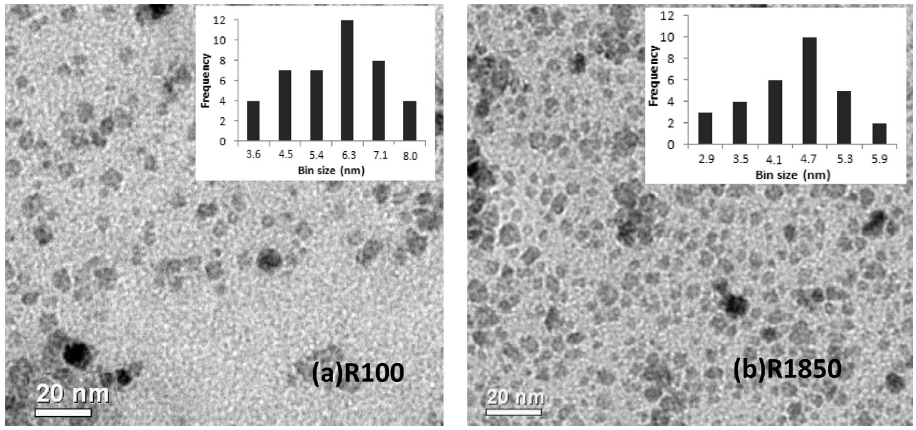

Fig. 7. TEM images of cobalt ferrite nanoparticles synthesized at different stirring rates a) 100 rpm and b) 1850 rpm. Fig. 3b also shows the TEM image of sample synthesized at 1000 rpm (R1000 or T90)).

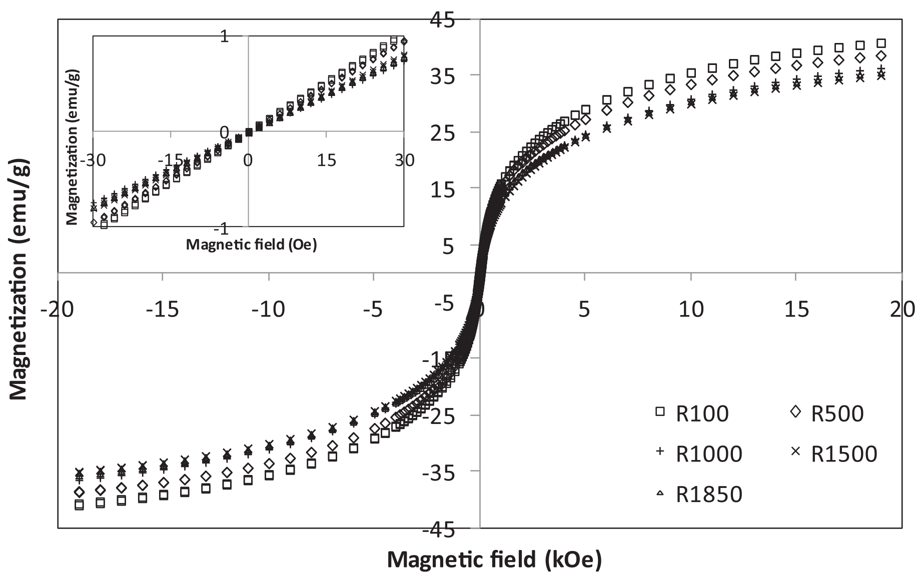

Fig. 8. Magnetization curves of the samples synthesized at different stirring rates (Inset shows the magnetization curves at ± 30 Oe.)

stirring rates were given in Fig. 8. All samples are superparamagnetic according to the magnetic measurements at room temperature. The M_{max} changes between 35.3 and 41.0 emu/g. It is observed that the M_{max} decreases with the increase of stirring rate. The d_{TEM}, d_{XRD}, and d_{VSM} of the nanoparticles were presented in Table 1. The change in the particle sizes is consistent with each other. As seen from the table, the decrease of M_{max} is also consistent with the decrease of particle size as the stirring rate increases. The M_{max} value is smaller than the saturation magnetization of bulk cobalt ferrite (80 emu/g) due to the small size and surface effects [33]. The maximum M_{max} for SCFNs (sample S100) is 41.0 emu/g. In the studies [33,34], SCFNs has much smaller magnetization. In the study [33], superparamagnetic nanoparticles with little H_c, has a magnetization value of 10.9 emu/g. In [34], magnetization of SCFNs at 10 kOe is 4.2 emu/g. Under the effect of stirring rate, much higher magnetization value of 41.0 emu/g for SCFNs with zero H_c and remanence was obtained in this study. Using Eq. (1), the χ_i values for samples R100, R500, R1000, R1500 and R1850 were calculated to be 0.035, 0.032, 0.025, 0.027 and 0.026 emu/gOe, respectively. The χi value decreases from 0.035 to 0.025 emu/gOe as the stirring rate increases from 100 to 1000 rpm and stays almost constant with further increase of stirring rate. It is observed that sample R100 has the highest M_{max} and χ_i values and is superparamagnetic with zero H_c.

4. Conclusions

Superparamagnetic cobalt ferrite nanoparticles (SCFNs) with high magnetization values were synthesized by co-precipitation at 80 °C in one step in air atmosphere. The effects of reaction time and stirring rate on the structural and corresponding magnetic properties of the nanoparticles were investigated. X-ray diffraction analysis (XRD), Fourier transform infrared (FT-IR) spectroscopy and transmission electron microscopy (TEM) were used to identify and structurally characterize the nanoparticles. The XRD and FT-IR analysis showed that the particles are cobalt ferrite. And, the particle sizes were calculated by using the XRD, TEM and vibrating sample magnetometer (VSM) data. Changing the degree of each parameter, the effect on the particle size and thus the magnetization of the nanoparticles were observed. Magnetic measurements performed by VSM at room temperature showed that all cobalt ferrite nanoparticles are superparamagnetic in nature. For reaction time effect, the particle size limit for the SCFNs with zero coercivity is found to be 7 nm. With the increase of particle size above 7 nm, magnetization increases as well as small coercivities of 2–5 Oe appear. In the case of stirring rate effect, a maximum magnetization value of 41.0 emu/g was obtained. The results proved that SCFNs can easily be obtained in one-step synthesis, and their structural properties especially the particle sizes and corresponding magnetic properties are affected by the synthesis parameters of reaction time and stirring rate. As a result, this study displayed that the SCFNs can easily be tailored by the synthesis parameters for potential applications in biomedical areas i.e. magnetic hyperthermia and MRI.

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