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Synthesis of nano-sized magnetic iron oxide by a simple and facile co-precipitation method

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Abstract

Magnetic iron oxide (Fe_3O_4) in nano form has been synthesized by a simple wet chemical method involving co-precipitation using iron salt and ammonium hydroxide as precipitating agents. Characterization of the synthesized nanoparticles were carried out by using XRD, FTIR, FESEM and VSM techniques. Crystallite sizes determined from XRD data were found to be between 6.8 and 12.6 nm with face center cubic crystal structures. The lattice parameter was determined to be 8.34 Å. FESEM microstructure revealed a spherical like nanoparticles with average particle size of ~26 nm. The saturation magnetization of the synthesized magnetite nanoparticles was measured to be 49.88 emu/gm. The Fe_3O_4 nanoparticles synthesized by co-precipitation method is considered to be potentiality good magnetic material with diverse applications.

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Introduction

Nanotechnology, in this 21st century has emerged as an indispensable area of research offering huge opportunities for the researchers to come up with new technological advances aiming wide-ranging applications. Particularly, owing to exclusive size-dependent properties coupled with large surface area, nanoparticles have received substantial attention to the scientists around the world (Ajinkya *et al.,* 2020; Rashid *et al.,* 2020; Schwaminger *et al.,* 2020; Shibaev *et al.,* 2020). Nevertheless, depending on the application, choice of nanoparticles is a vital issue to be considered. Indeed, nanoparticles with high impact in medical science have now become a lucrative area of research and in this context both oxide and hydroxide forms of iron having excellent magnetization competency but low toxicity scores top ranking. Indeed, iron being an associate of transition element family exists naturally in different phases including oxide and hydroxide and in particular, hitherto a variation of sixteen different phases of iron oxide has been recorded (Rashid *et al.,* 2020). Among the members of iron family, iron oxide predominantly in nano form has found widespread applications as mentioned in Fig. 1.

Nevertheless, iron oxide at the nano scale level is fairly superparamagnetic which depends on the synthesis temperature (Shibaev *et al.,* 2020) and according to the strength of the magnetism, hierarchy of different phases of iron oxide are classified where being positioned in the top, magnetite is followed by maghemite and hematite respectively (Shibaev *et al.,* 2020).

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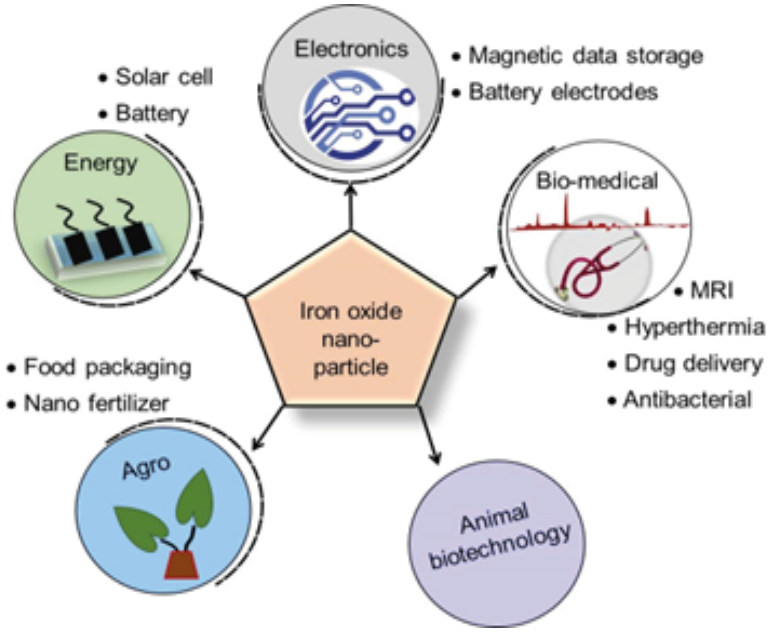

Fig. 1. Application of iron oxide nanoparticle

Concerning the importance of iron oxide magnetic nanoparticles, recently, researchers have paid substantial attention to enrich this field. Magnetite is an ideal oxide which has a cubic inverse spinel crystal structure and it exhibits unique electric and magnetic properties based on the transfer of electrons between the ferrous ion and ferric ion in the octahedral sites (Gawande *et al.,* 2013; Xu and Wang, 2012). Compared to magnetite in a bulk form, magnetite nanoparticles have been intensively investigated because of their superparamagnetism, high coercivity and low Curie temperature (Kim and Kim, 2003; Suicide and Event, 2000). Accordingly, development of new synthetic routes as well as field of application is of significant concern now. Hitherto, several different methods have been developed for the synthesis of iron oxide nanoparticles such as co-precipitation, micro emulsion, laser pyrolysis, microwave and ultra sound assisted synthesis, gas phase flame synthesis, electrochemical synthesis, hydrothermal and solvothermal synthesis (Hong *et al.,* 2007; Iwasaki *et al.,* 2010; Rashid *et al.,* 2020; Tabares *et al.,* 2009; YAO *et al.,* 2009; Yazdani and Seddigh, 2016; Yu *et al.,* 2007; Zheng *et al.,* 2020). Among these developed methods, the co-precipitation is categorically selected as the simplest but efficient chemical procedure to synthesize the nanoparticles (YAO *et al.,* 2009). The positive features of co-precipitation method include high degree of crystallinity, significant yield, short reaction time and relatively low temperature etc. Keeping these views in mind, in this research work we have attempted to synthesize magnetite nanoparticles by simple co-precipitation method which will be a step forward in various field. But due to sensitivity to oxidation, it is quite difficult to synthesize and stabilize pure magnetite. In most of the cases, a mixture of magnetite and maghemite is obtained, and the quantification of each phase in such mixtures is quite difficult (Stoia *et al.,* 2016). This work presents the synthesis of magnetite nanoparticles by simple co precipitation method using ferric and ferrous salts with ammonium hydroxide as a precipitating agent and the synthesized nano magnetite was characterized by XRD, FTIR, FESEM and VSM.

Materials and methods

Analytical grade chemicals ($FeCl_3.6H_2O$, $FeSO_4.7H_2O$, NH_4OH) obtained from Sigma Aldrich (USA) and Merck

(Germany) were used for this study without further purification. Deionized water was used in all experiment.

Synthesis of magnetite (Fe_3O_4) nano particles

A simple co-precipitation method was adopted to synthesize magnetite nanoparticles where Ferric and Ferrous salts were used as the starting materials. A homogenous mixture of both salts was prepared by using 0.6 M Ferric chloride and 0.3 M Ferrous sulfate (Molar ratio of Fe^{3+} : Fe^{2+} = 2:1) solution. After complete dissolution by stirring at 300 rpm for 30 minutes the solution was transferred into a two necked glass round bottom flask. One neck of the round bottom flask was equipped with the nitrogen gas balloons to ensure inert environment while Ammonium hydroxide (25%) was added dropwise through the second neck. The entire experimental arrangement was run at 80° C with constant stirring at 400 rpm using a hotplate with magnetic stirrer. A highly alkaline pH (9 to 14) medium was maintained. After the completion of the reaction, both heating and stirring were stopped and the solution was allowed to be cool without any disturbance. Such an arrangement prompted the black precipitate to settle down. The precipitate was then filtered carefully and washed with sufficient amount of deionized water ensuring highest purity. The washed precipitate was dried at 50°C for 10 h in an oven. The experimental set-up is shown in Fig. 2.

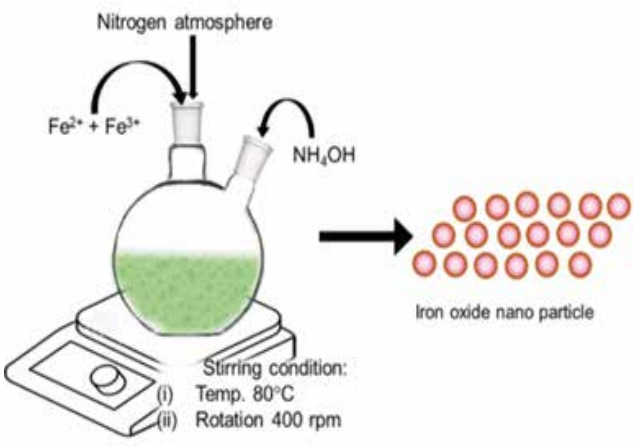

Fig. 2. Experimental setup

The possible reaction scheme for the formation of Fe_3O_4 is considered as follows:

$$Fe^{3+} + 3OH \quad Fe(OH)_3$$

$$Fe(OH)_3 \longrightarrow FeOOH + H_2O$$

$$Fe^{2+} + 2OH \longrightarrow Fe(OH)_2$$

$$FeOOH \longrightarrow Fe(OH)_2 \longrightarrow Fe_3O_4 + H_2O$$

Characterization

The crystal structure of the synthesized nano magnetite was characterized by different techniques. BRUKER AXS-D8 Advance (Germany) diffractometer having Bragg-Brentano geometry with Cu-Kα X-ray radiation source (λ=1.5406 Å) was used to analyze the desired phases while functional groups were identified by Fourier Transform Infrared (FT-IR) Spectroscopy (Model: Shimadzu Prestige 21). The morphology of the Fe_3O_4 nano material was examined by Field Emission Scanning Electron Microscope (Model: JEOL-JSM7610F). The magnetic property of the Fe_3O_4 materials was measured using vibrating sample magnetometer (VSM) in the Physical Property Measurement System (PPMS) DYNACOOL Quantum Design, at room temperature with a magnetic field in the range of -2000 to 2000 Oe.

Results and discussion

X-ray diffraction (XRD) analysis

The crystallographic structure of the synthesized Fe_3O_4 was evaluated by X-ray diffraction (XRD) technique. The XRD measurements of Fe_3O_4 were performed from 2θ=10° to 80° at room temperature. Fig. 3 displays the X-ray diffraction patterns of the studied sample. The diffraction peaks corresponding to (220), (311), (222), (400), (422), (511) and (440) are similar to the characteristic diffractions of the Magnetite (Fe_3O_4) depicted by PCPDFWIN v.2.02, PDF No. 89-0691 (Zhuang *et al.,* 2015). Therefore, inverse spinel cubic structure of the $Fd\bar{3}m$ space group is confirmed in the synthesized sample. The lattice parameters were obtained by refining XRD data with $Fd\bar{3}m$ space group by an X'Pert High Score Plus software. The

obtained lattice parameter (a=b=c) 8.3442 Å, is closer to a reported data of 8.394 Å (Mamani *et al.,* 2013) and the difference is attributed to the synthetic conditions. Moreover, absence of any sharper peak in the diffraction patterns may be due to smaller crystallites, which might be associated with strain. The comparatively sharper and separate diffraction peaks at (220), (311), (511) and (440) were utilized to calculate crystallite size for different peaks of

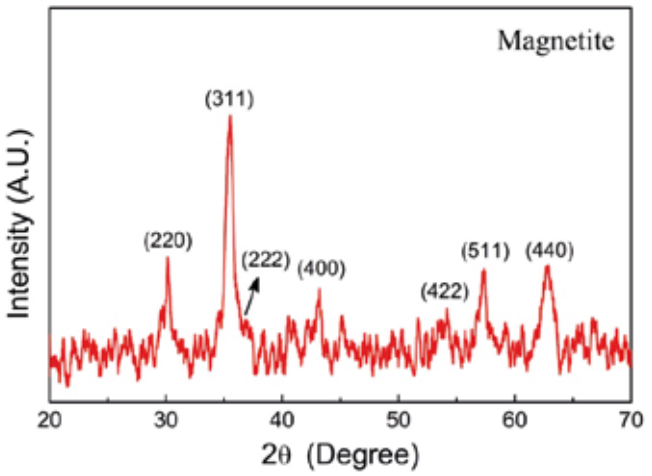

Fig. 3. XRD patterns of magnetite (Fe_3O_4) nanoparticles

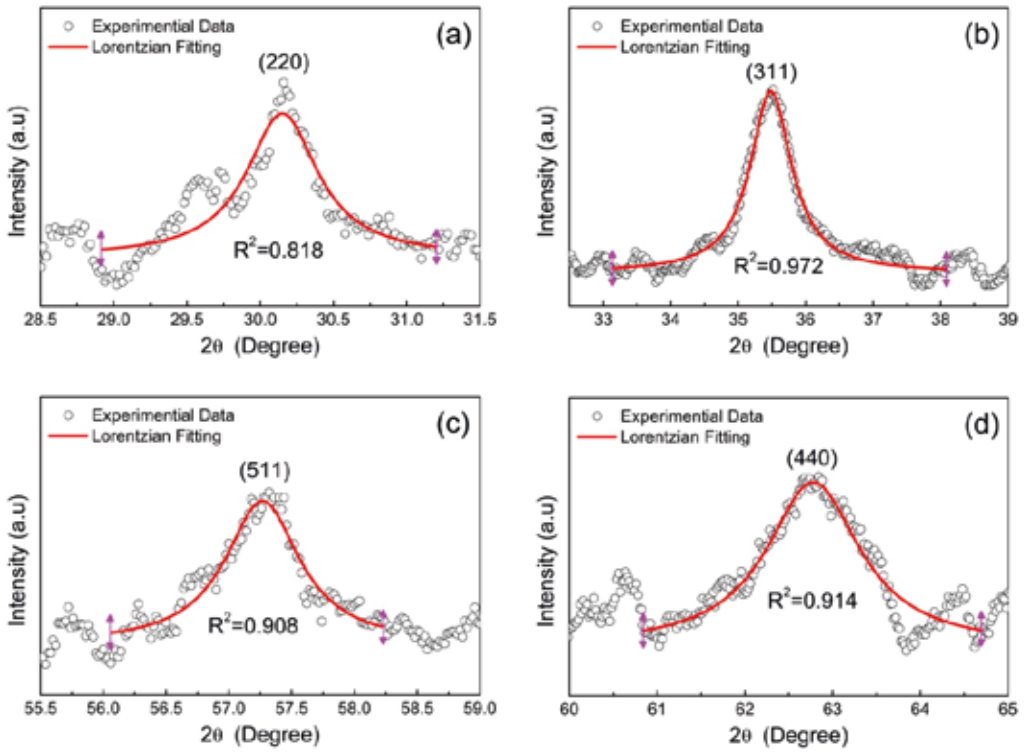

Fig. 4. Lorentzian Fitting of XRD peaks at (220), (311), (511) and (440) measured for Fe_3O_4

the sample by using Debye-Scherrer formula (Keskenler *et al.,* 2013):

$$d = \frac{k\,\lambda}{\beta\cos\theta} \qquad (1)$$

Where typically k~9 (a dimensionless constant), λ =1.5406 Å (wavelength of Cu k_α radiation). Moreover, the value of θ was estimated from Bragg's position of the diffraction peaks and the parameter, β (FWHM) has been calculated by Lorentzian fitting of the peaks at (220), (311), (511) and (440) respectively and displayed in Fig. 4(a-d). The obtained parameters and sizes of the crystallites are reported in Table I. The crystallite sizes (X_C) vary from 6.82 to 12.61 nm. However, the average size of crystallites is 10.91 nm, which may be reported for the synthesized sample, Fe_3O_4.

Table I. Structural Parameters with Crystallite Size (X_C) and Lattice Strain (ε) obtained from different peaks and their positions and R^2 is the fitting parameter where for the best fit R^2=1. Apart from this, average particle size (X_A) for different magnifications are also included

Peaks	Peak Position (2θ)	FWHM	X_c (nm)	ε	(R^2)	X_A (50 kX)	X_A (100 kX)	X_A (150 kX)	X_A (200 kX)
220	30.14	0.6815	12.61	0.0110	0.818				
311	35.48	0.7434	11.72	0.0101	0.972	25.12	27.45	26.52	26.54
511	57.27	0.7580	12.48	0.0061	0.908	±0.54	±0.42	±0.16	±0.36
440	62.77	1.4260	6.82	0.0102	0.914				

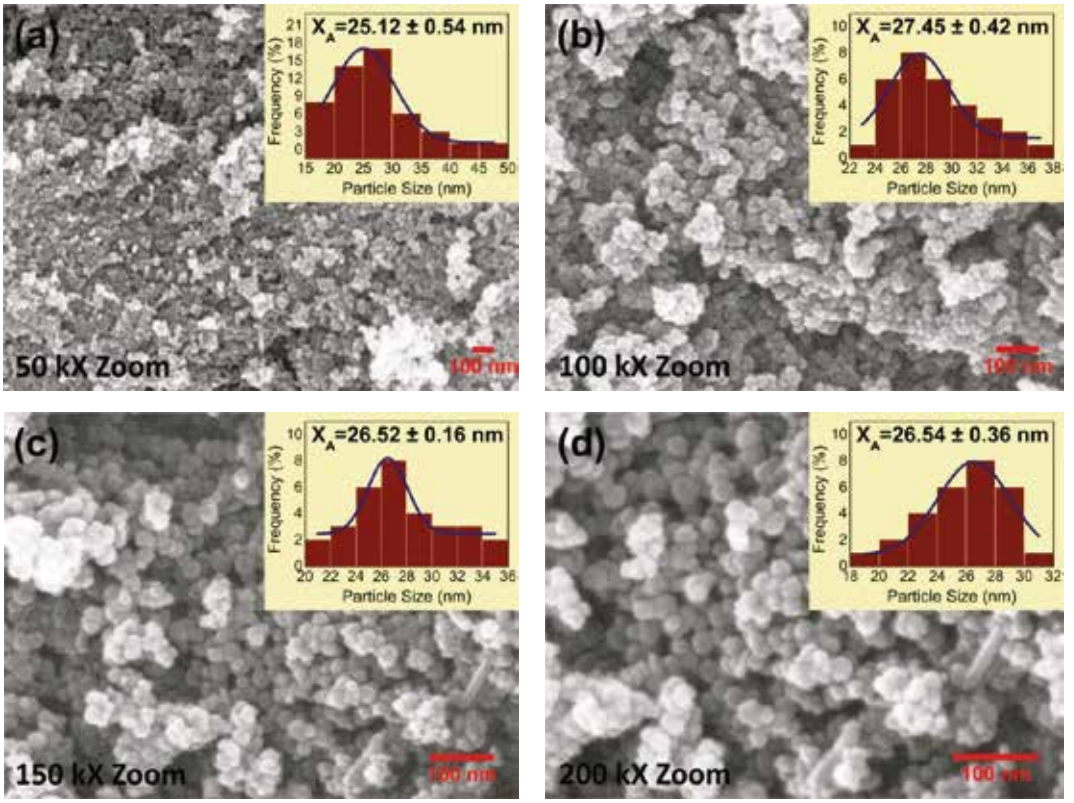

Fig. 5. Micrograph of synthesized magnetite (Fe_3O_4) with different magnifications showing the particle shape and size distribution and inset is displaying particle size distribution

Microstructural study

Field Emission Scanning Electrom Microscopye (FESEM) was used to evaluate the microstructure and particle size of ferrite samples. The surface morphology of the synthesized Fe_3O_4 nanoparticles were examined by the excitation of 15 keV electron beam. Fig. 5 (a-d) shows the microstructure of the studied samples in different magnification starting from 50 kX to 200 kX. In all magnification it is manifested that all particles are of quasi-spherical shape and the sizes of

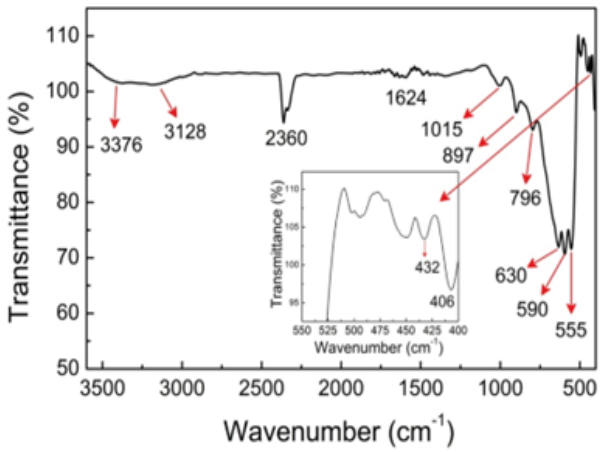

Fig. 6. FTIR spectra of synthesized nano magnetite (Fe_3O_4)

particles varies from 15 nm to 50 nm. Moreover, the particles are distinct except negligible agglomeration. However, the appropriate size of the particles was obtained from the calibrating and line interpolation method using Image J 1.50i software. In this regards several particle sizes were obtained and with this size histogram was plotted. Thereafter, average particle sizes were obtained by the Gaussian fitting of particle size distribution shown in inset of Fig. 5(a-d). For all magnification the average particle size, X_A, is nearly same (~26 nm) and the values are presented in Table I. It is noteworthy that the average crystallite size, X_c obtained from XRD is 10.91 nm, which is less compared to the average particle size, X_A (~26 nm). This result clearly implies that 2/3 crystallites are added together to form a particle.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Fourier Transform Infrared Spectroscopy (FTIR) is a powerful tool that can identify the chemical bonds present inside any material. The FTIR spectra were recorded for Fe_3O_4 sample in the range 400-3600 cm^{-1} and shown in Fig. 6. From the spectra, several absorption peaks are observed which correspond to different stretching modes. In general, two strong infrared absorption bands at 390 and 570 cm^{-1} are detected for an ideal magnetite (Ercuta and Chirita, 2013; Ishii *et al.,* 1972). The absorption band at 390 cm^{-1} is assigned to the Fe-O stretching mode at octahedral site while the mode at 570 cm^{-1} corresponds to the same mode at tetrahedral site (Siva Ram Prasad *et al.,* 2011). Maghemite, Fe_2O_3, γ-Fe_2O_3, is a defective phase of magnetite and displays absorption bands at 430, 590 and 630 cm^{-1} (Ercuta and Chirita, 2013). Goethite, FeO (OH), has ν-OH, δ-OH, γ-OH stretching vibrations and absorption bands are observed at ~3125, ~890 and ~800 cm^{-1} respectively (Cambier, 1986).

Table II. Absorption peaks and their corresponding modes extracted from FTIR spectra of the synthesized Fe_3O_4

Peak position (cm^{-1})	Bands
406	Fe-O stretching mode at octahedral site
432	Fe_2O_3, γ-Fe_2O_3
555	Fe-O stretching mode at octahedral site
590	Fe_2O_3, γ-Fe_2O_3
630	Fe_2O_3, γ-Fe_2O_3
796	γ OH
897	δ OH
1015	symmetric Si-O-Si stretching vibration
1624	H_2O molecule stretching
2360	CO_2 asymmetric stretching
3128	ν OH
3376	H_2O molecule stretching

The peak at 1,070 cm^{-1} corresponds to symmetric Si-O-Si stretching vibration (Repo *et al.,* 2011). The peak at 2,349 cm^{-1} is an asymmetric stretching peak of CO_2 in the air (Gong and Tang, 2020). From these literature reviews we may conclude that the synthesized sample is not a pure magnetite while it contains a small amount of goethite and maghemite with some environmental impurity. All of the absorption peaks and their corresponding modes are presented in Table II.

Magnetic Measurement by Vibrating Sample Magnetometer (VSM)

M-H hysteresis curve of magnetite nanoparticles recorded at room temperature are illustrated in Fig. 7. The saturation magnetization (M_s), retentivity (M_r) and coercivity (H_c) of the synthesized sample are largely controlled by domain state, which in turn depends upon grain size (Kahani and Yagini, 2014). Saturation magnetization (M_s), retentivity (M_r) and coercivity (H_c) of the sample are listed in Table III. The saturation magnetization of magnetite nano particles was found to be 49.88 emu/gm which is lower than the saturation magnetization value of the bulk Fe_3O_4 (92emu/gm) (Lu *et al.,* 2007). Several explanations have been provided that the saturation magnetization of ferrite nanoparticles sharply decreases with decreasing particle size (Daoush, 2017; Khatiri *et al.,* 2013). Magnetic property is clearly size-dependent and superparamagnetism occurs in nanoparticles which are single domain and it is possible when their diameter is below 3-50 nm, depending on the materials (Marghussian, 2015; Wallyn *et al.,* 2019). It is also reported that when the diameter of the magnetite nanoparticles is reduced to below a certain critical value (usually less than 35 nm); they lose their bulk ferromagnetism and are said to become superparamagnetic (Khatiri *et al.,* 2013). In the present study the synthesized magnetite nanoparticles of average diameter is ~26 nm which is lower than upper limit for superparamagnetic behavior which indicates that the synthesized nanoparticles in this study may be assumed as superparamagnetic though small amount of remnant magnetization and coercivity are found in the measurement. It is also observed that there was a small rententivity 12.10 emu/gm. One possible mechanism for this unique form is the independent thermal fluctuation of small ferromagnetic domain inside the nano-

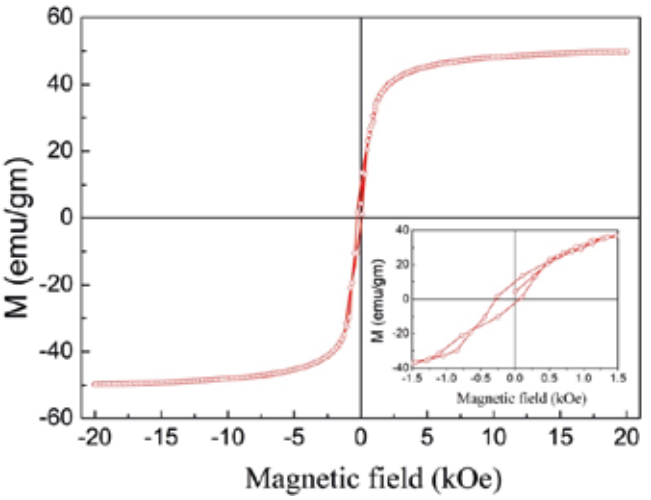

Fig. 7. M-H hysteresis curve of Fe_3O_4 nanoparticles

Fig. 8. Magnetic response of Fe_3O_4 nanoparticles

Table III. Magnetic properties values of synthesized Fe_3O_4 nano particles

Magnetic properties	Values
Saturation magnetization (M_s), emu/gm	49.88
Retentivity (M_r), emu/gm	12.10
Coercivity (H_c), Oe	328

particles (Daoush, 2017). On the other hand, the magnetite nanoparticles displayed a strong magnetization in the presence of a magnetic field which is shown in Fig. 8.

Conclusion

The synthetic method for preparing magnetic iron oxide (Fe_3O_4) nano particles was achieved by a simple and facile co-precipitation method in aqueous solution using iron salts as a precursor. Large quantities of magnetite nano particles with small amount of maghemite has been synthesized by using the above mentioned synthetic method. The present study shows that the magnetite nano particles have a spinel crystal structure with average particle size of ~26 nm and nearly spherical in shape. The properties of superparamagnetic nanoparticles depend on the size and the method of synthesis. The magnetite nanoparticles with low saturation magnetization (49.88 emu/gm) may be correlated with reduction of the particle size and small amount of maghemite phase compared with the bulk magnetite (92 emu/gm). So it is concluded that Fe_3O_4 nanoparticles synthesized by co-precipitation method can be promising as potentiality good magnetic material having good properties.

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Authorship contribution statement

Juliya Khanam: Conceptualization, Methodology, Supervision, Project Administration, Visualization, Software, Writing – original draft, Writing – review and editing. Md. Farid Ahmed: Methodology, Data curation, Validation, Writing – review and editing, SK. Methela Zaman: Methodology, formal analysis, Writing – review and editing. Nahid Sharmin: Project Administration, Supervision, Writing – review and editing, Fund acquisition, Samina Ahmed: Supervision, Visualization, Writing – review and editing.

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