

Synthesis, Structural and Magnetic properties of $Mg_{0.25}Co_{0.75}Fe_2O_4$ Nanoparticles Ferrites using Wet chemical routes

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Abstract

Ferrite nanoparticles are interesting materials due to their rich and unique physical and chemical properties. They find applications in catalysis, bio-processing, medicine, magnetic recording, adsorption, devices, etc. Nanoparticles of $Mg_{0.25}Co_{0.75}Fe_2O_4$ were synthesized by co-precipitation method. Using stable ferric, magnesium and cobalt salts with oleic acid as the surfactant Structural studies are carried out using X-ray diffraction (XRD). The XRD pattern of the sample provides information about single – phase formation of spinel structure with cubic symmetry and space group (SG Fd3m) and 0.834 nm lattice constant, which is confirmed by micro Raman measurements at room temperature. From the analysis of powder X-ray diffraction patterns, the nanocrystallite size was calculated from the most intense peak (3 1 1) using Scherrer formula. Thus, the size of the particles is found to be $\sim 16 \pm 5$ nm. The lattice constant is obtained using XRD data. The structural morphology of the nanoparticles is studied using Scanning Electron Microscopy (SEM). Formation of spinel structure is confirmed using Fourier transform infrared spectroscopy (FTIR). Vibration frequency and force constant are discussed with the help of FTIR data. The M–H loop of $Mg_{0.25}Co_{0.75}Fe_2O_4$ has been traced using the Vibrating Sample Magnetometer (VSM) and magnetic parameters such as saturation magnetization (MS), coercivity (HC) and retentivity (MR) are obtained from VSM data. Mössbauer spectroscopy measurements revealed that the sample was ferromagnetic material.

Key words: Ferrites Nanoparticles; Wet chemical routes; VSM; FTIR; micro –Raman spectroscopy;

الخصائص التحضيرية والتركيبية والمغناطيسية لمركب $Mg_{0.25}Co_{0.75}Fe_2O_4$ الحديدي النانوي باستخدام طريقة الجذور الكيميائية الرطبة

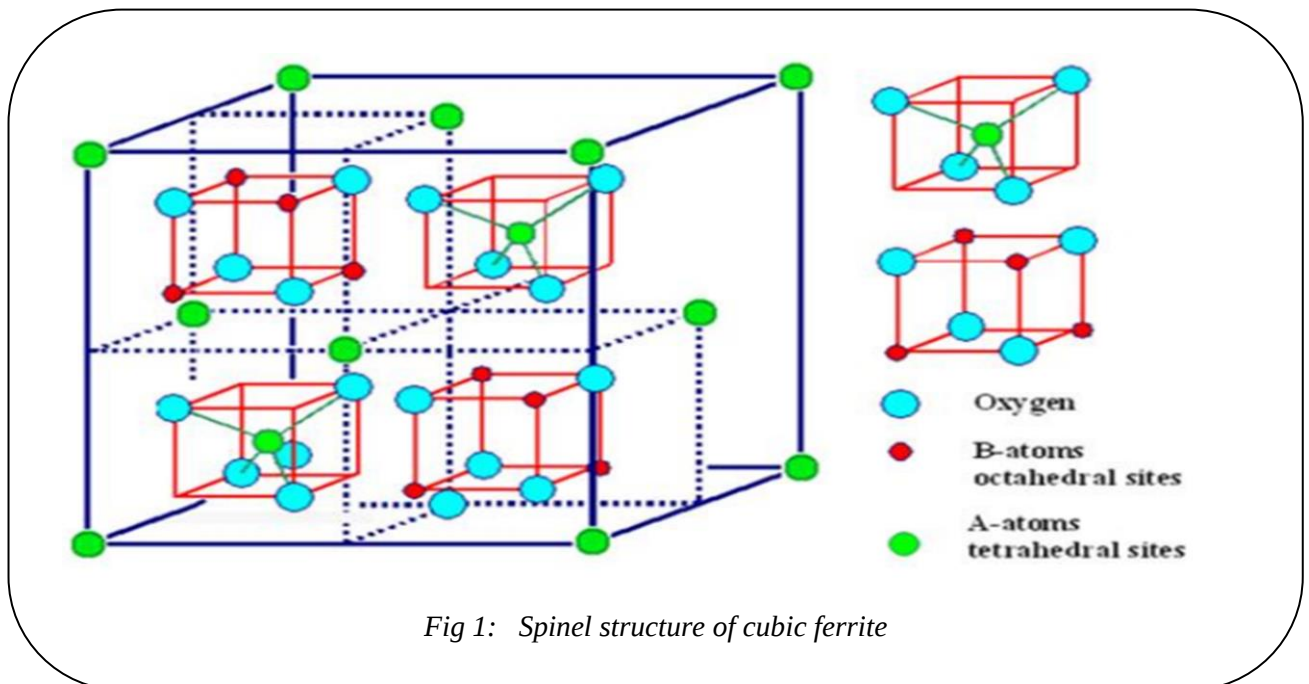
المستخلص

جزيئات الحديد النانوية تعد مواد جاذبة من خلال صفاتها الفيزيائية والكيميائية الفريدة. حيث يوجد استخدامها في المحفزات والعمليات الحيوية والطب والتسجيل المغناطيسي والادماص والأجهزة وغيرها. حُضِرَ مركب $Mg_{0.25}Co_{0.75}Fe_2O_4$ النانوي بطريقة البادرات المترافقة. باستخدام حديد وأملاح المغنيسيوم والكوبالت المستقرة مع حمض الأوليك كمحفز. تُرِست الخصائص التركيبية عن طريق جهاز حيود الأشعة السينية (XRD) والتي أظهرت المعلومات المأخوذة من الطيف أن الطور أحادي ويأخذ التركيب البلوري المكعبي بشبكة فراغية (Fd3m) و ثابت شبكة 0.834 nm، ودُعِمَ ذلك من خلال قياسات رامان في درجة حرارة الغرفة. من خلال تحليل طيف حيود الأشعة السينية حُسِبَ حجم الشبكة النانوية عبر أعلى قمة (3 1 1) من خلال صيغة شيرار حيث وُجِدَ أن حجم الجسيمات حوالي 16 ± 5 nm. إذ وُجِدَ ثابت الشبكة من خلال بيانات جهاز حيود الأشعة السينية (XRD) والتركيب التضاريسي تُرِست عبر مطياف المساح الألكتروني (SEM). الصيغة التركيبية للأُسْبُلُ أُكِدَت باستخدام مطياف فورير للأشعة تحت الحمراء. والذي بمساعدة بياناته نوقِشَ التردد الاهتزازي وثابت القوة. منحنى M–H أُخِذَ بمغناط اهتزاز العينة (VSM) وكذلك المعاملات المغناطيسية مثل التشبع المغناطيسي (MS) و القسرية (HC) و التخلفية (MR). قياسات مطياف المسبار أظهرت أن العينة من المغناطيسيات الحديدية

Introduction

Spinel ferrite nanoparticles of chemical formula (MFe_2O_4) where ($M = Zn, Co, Mn, Mg, Gd$, etc) have been intensively investigated in recent years not only due to usual properties different from their corresponding bulk materials but also for their promising technological applications (Liu et al., 2020). Infamies, the magnetic ions occupy the tetrahedral (A) and octahedral (B) sites of the spaniel structure. The exchange integrals (J_{AB} , J_{BB} and J_{AA}) are generally ergative and the anti-Ferro magnetiteinteraction A-B is stronger than B-B and A-A interaction (Parida, Karati, Guruvidyathri, Murty, & Markandeyulu, 2020). This leads to ferromagnetism: B-B and A-A interaction being frustrated. The spin of the same ions in the same sublattices are parallel and those of the two sublattices are ant parallel. A certain amount of site exchange of cations is sufficient to change J_{AB} and J_{BB} interactions and has shown drastically different magnetic behaviour in nanoparticles

chemical properties of spinel ferrite nanoparticles (Nps) are different from their corresponding bulk material due to their higher surface to volume ratio (Naseri, Saion, Ahangar, Hashim, & Shaari, 2011). The unit cell of spinel ferrite belongs to the cubic structure, Ferrites continue to be remarkable magnetic materials due to their potential applications in Ferro-fluids, magnetic resonance, imaging, bio-medical diagnostics, drug delivery, sensors, permanent magnets, magnetic refrigeration systems, etc. (Berry, 2009; Xia et al., 2003). Experiment has shown that the properties of ferrites are strongly influenced by the material composition and microstructure, which are sensitive to the preparation methodology used in the synthesis, where several methods such as ball milling, sol-gel, co-precipitation, hydrothermal technique auto-combustion route, ultrasonic cavitation, etc. have been used to synthesize zinc ferrite nanoparticles. Among these methods, co-precipitation is promising technique to synthesis the ferrites at low temperatures (Cannas, Gatteschi, Musinu, Piccaluga, & Sangregorio, 1998; Yue, Zhou, Li, Zhang, & Gui, 2000). In this



spinel in comparison with the bulk (Brabers, 1995; Chinnsamy et al., 2001). It has been wildly found that in the bulk ferrites, Mg actions have a strong preference for occupying A-sites and conation for occupying B-sites. The physical and

work, we have investigated the possibility of synthesizing nanocrystalline magnisuim ferrite via co-precipitation technique at room temperature without any subsequent calcinations using chloride precursors and NaOH as precipitating

agent. The properties of prepared nanoparticles samples have been studied after annealing at 450°C. Choosing an optimal method is the key to getting a quality ferrite (Costa, Tortella, Morelli, & Kiminami, 2003). Oxygen anions formed the close face-centered cubic (FCC) packing consisting of 64 tetrahedral (A) and 32 octahedral (B) metal ions. As in **Figure (1)**, The present work deals with the synthesis of cobalt doped magnesium ferrite nanoparticles ($\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$) using chemical co-precipitation and study of their structural and magnetic properties. The present work deals with the synthesis of cobalt doped magnesium ferrite nanoparticles ($\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$) using chemical co-precipitation and study of their structural and magnetic properties.

2-Material & Methods

Ferric chloride [$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$], magnesium chloride [$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$] and cobalt chloride (98% purely) [$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$] and sodium hydroxide were used. Oleic acid of HpCl grade was used as a surfactant. All the materials were reagent grade and used without further purification. Double distilled de-ionized water was used as a solvent.

2-1 Procedure:

For $\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$ synthesized, we used 0.4M (25 ml) solution of iron chloride and a 0.2M (25 ml) of cobalt and magnesium solutions were mixed in double distilled water deionized distilled water was used as a solvent to avoid the production of impurities in the final product. 3M (25 ml) solution of sodium hydroxide was prepared and slowly added to the salt solution dropwise. The pH of the solution was constantly monitored as the NaOH solution was added. The reactant was constantly stirred using the magnetic stirrer unit a pH level of 11 – 12 was added to the solution as a surfactant and coating material (Pillai & Shah, 1996). The liquid precipitate was then brought to a reaction temperature of 80°C and stirred for one hour. The product was then cooled to room temperature to get free particle from sodium and chlorine compounds, the precipitate was then washed twice with distilled water and

then with ethanol to remove the excess surfactant from solution to isolate the supernatant liquid. The supernatant liquid was then decanted. The supernatant liquid was then decanted, and then centrifuged until any thick black precipitate remained. The precipitate was then dried overnight at 100°C. The acquired substance was then grounded into a fine powder. At this stage, the product contains some associated water, which was removed by heating at 450°C for 10 hours (**figure 2**). The final product obtained was then confirmed by X-ray diffraction, etc to be magnetic nanoparticles of cobalt, magnesium ferrite with inverse spinel structure.

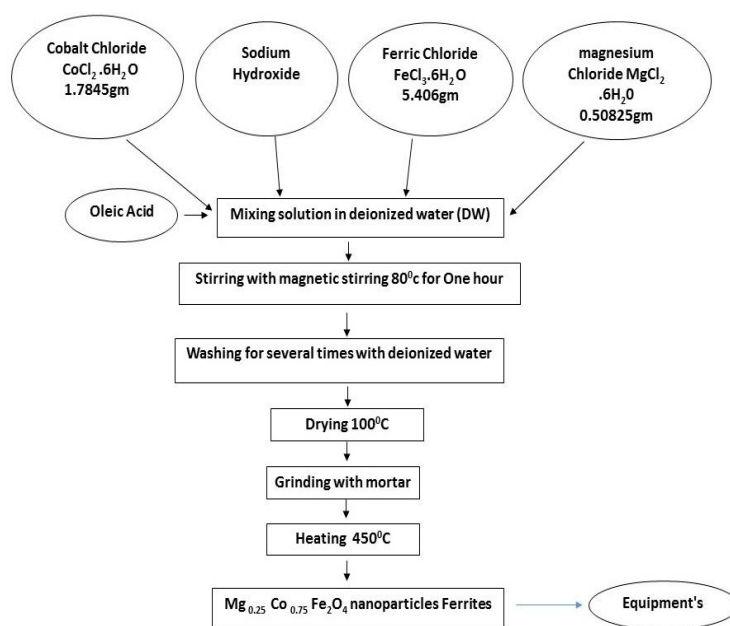


Fig 2: Schematic representation of the procedure for the preparation of $\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$ nanoparticles using a chemical co-precipitation method

2-2 Characterization technique

The phase purity of the samples was verified using XRD with a Shimadzu 6000 X-ray diffractometer equipped with Cu- α radiation of a wavelength of $\lambda=1.5406 \text{ \AA}$. The data were collected in a 2θ range from 20° to 80° at a step size of 0.02. The phase purity and the lattice parameters were determined using the FullProf suite (Omer, Elbadawi, & Yassin, 2013). To measure the Raman spectrum, a powder sample was pressed into a pellet (without further heating)

to get rid of any air cavities that may lead to heat the sample. The particle size was determined by scanning electron microscope (SEM). Furthermore, Raman spectra of the sample were collected at room temperature using the BrukerSentera Raman microscope spectrometer equipped with 532-nm excitation lasers. The laser beam spot was focused on the sample using a 50x lens and an aperture of 25 μm . The accumulation time was set to 40 sec with 10 co-additions making the duration of a scan run to about 12 minutes. The laser power was set at 20 mW. Magnetic measurements were conducted using vibrating sample magnetometer (VSM) and Mossbauer spectra were collected at room temperature 27°C (300 K) at a constant acceleration. The spectrometer was calibrated with $\alpha\text{-Fe}$ foil at room temperature.

3. Result and discussion

3.1 Structural Analysis

3.1.1 XRD analysis

X-ray diffraction pattern of cobalt doped magnesium ferrite nanoparticles $\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$ annealed at 450°C is shown in Figure 3. The data are collected in a 2θ range from 20° to 80° at a step size of 0.02 using Shimadzu 6000 X-ray diffractometer equipped with Cu- $\text{k}\alpha$ radiation of a wavelength of $\lambda=1.5406 \text{ \AA}$. All the main peaks are indexed concerning the standard pattern and found to be (220), (311), (400), (442), (511) and (440) (Lal, Sharma, & Singh, 2005). The well-defined (311) peak appears to be more intense. The crystalline nature of the preparations Mg - doped with cobalt ferrite was analyzed by powder X-ray diffraction. The average crystallite size was calculated using the Scherrer equation (Ali et al., 2020) and was found to be $16 \pm 5 \text{ nm}$.

$$D(\text{crystalline size}) = \frac{0.94 \lambda}{\beta \cos \theta} \quad (1)$$

where D is crystallite size, k is the shape factor, λ is the wavelength of the radiation, β is the full width at half maximum of the diffraction peak, and θ is the corresponding diffraction angle. The

lattice parameter of the sample, the X-ray diffraction patterns of the cobalt doped magnesium ferrite combined with the Rietveld analysis method showed that the sample is cubic spinel ferrites particles with (SG: Fd-3m) and lattice constant $\sim 0.834 \text{ nm}$. The estimated average ferrite particle sizes of Mg ferrites using the Debye-Scherrer (DS) equation are found to be $\sim 16 \pm 5 \text{ nm}$ (Joint Committee on Powder Diffraction Standards [JCPDS] (Hashim et al., 2020).

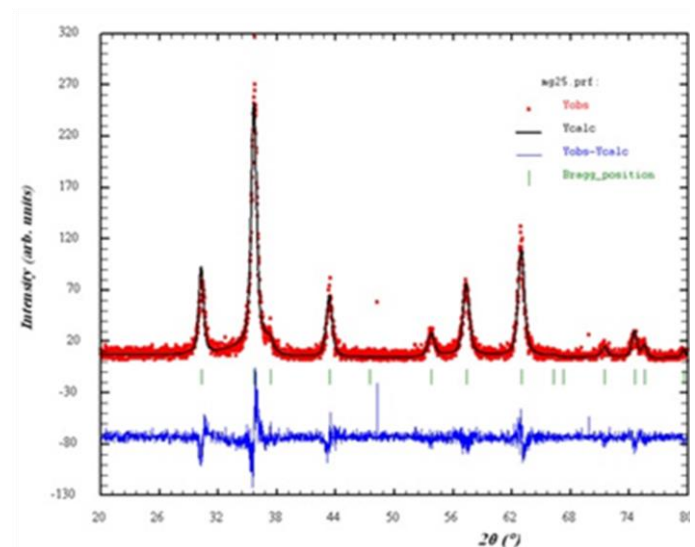


Fig 3: XRD spectrum of Cobalt doped magnesium ferrite of $(\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4)$ nanoparticles annealed at 450°C

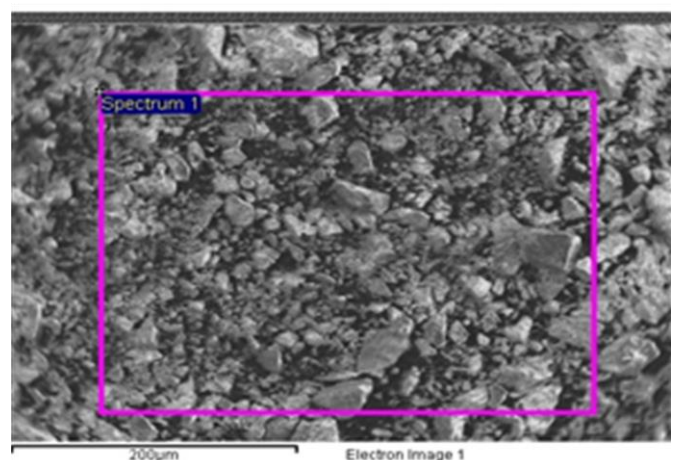


Fig 4: SEM micrographs of $(\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4)$ nanoparticles annealed at 450°C

3.1.2 SEM Analysis

Figure 4 shows the surface morphology of cobalt doped magnesium ferrite nanoparticles ($\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$) annealed at 450°C at different magnifications. From the scanning electron micrographs, it is clear that the method of synthesis of cobalt doped magnesium ferrite has resulted in a uniformly distributed granular like ferrite nanoparticles.

3.2 Molecular optical study

3.2.1 FT-IR spectral analysis

The infrared absorption spectrum in the range of $4000\text{--}400\text{ cm}^{-1}$ is recorded at room temperature by using Fourier Transform (FT) using a KBr pellet method. The spectrum transmittance (%) against wavenumber is used for interpretation of the results. The FT-IR spectrum of the investigated cobalt doped magnesium ferrite nanoparticles ($\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$) is shown in Figure 5. FTIR spectral analysis helps to confirm the formation of spinel structure with ferrite sample. In the wavenumber range of $4000\text{--}400\text{ cm}^{-1}$, two main broad metal-oxygen bands are seen in the infrared spectra of all spinels, especially ferrites. The higher one (ν_1) generally observed in the range $600\text{--}550\text{ cm}^{-1}$, is caused by the stretching vibrations of the tetrahedral metal-oxygen bond. The lowest band (ν_2) usually observed in the range $450\text{--}440\text{ cm}^{-1}$, is caused by the metal-oxygen vibrations in the octahedral sites. This difference in the spectral positions is expected because of the difference in the $\text{Fe}_3^+ - \text{O}_2$ distance for the octahedral and tetrahedral compounds. It is confirmed from Fourier transform Infrared spectroscopy (FTIR) that the structure remains cubic spinel after magnesium substitution in zinc nano ferrite (Rosnan et al., 2016; Cullity, 1967; Shinde Gadkari, & Vasambekar, 2013). The vibrational frequencies of IR bands ν_1 and ν_2 of samples prepared by sol-gel are shown in figure 5. The spectra show prominent bands near 3400 and 1600 cm^{-1} , which are attributed to the stretching modes and H-O-H bending vibrations of the free or absorbed water. The peaks are around 3429.27 cm^{-1} , 2921 cm^{-1} , corresponding to the stretching

and bending ferrite nanoparticles calcined at 450°C for 2 hours.

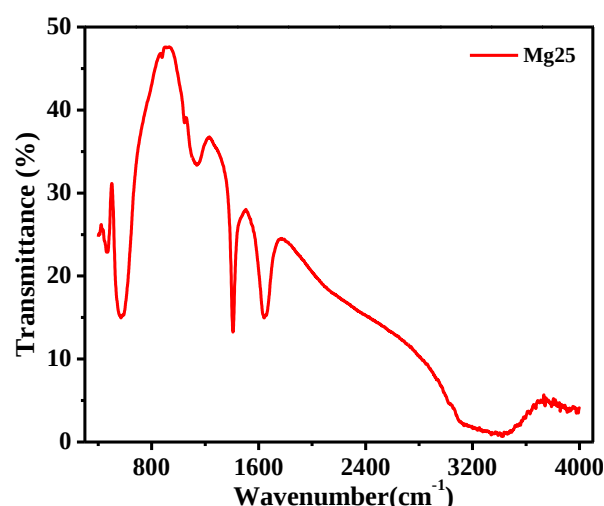


Fig 5: FTIR spectrographs of $\text{Mg}_{0.25}\text{Co}_{0.75}\text{Fe}_2\text{O}_4$ ferrites nanoparticles calcined at 450°C

3.2.2 Raman Spectroscopy analysis

Micro- Raman spectroscopy is used for further investigation of the phase formation and the effects of size reduction on the vibrational modes. The symmetry is checked by micro-Raman measurements. The micro-Raman spectra collected at room temperature is shown in figure 6 which reveals five broad peaks. These positions can be assigned to the symmetric modes A_{1g} , E_g and $3T_{2g}$ well known to be for the cubic symmetry (Darbandi et al., 2012) because the $I41/amd$ symmetry has 10 Raman active modes (Jacintho, Brolo, Corio, Suarez, & Rubim, 2009). The positions of the symmetric modes as well as their line width are analyzed by fitting them to the Breit-Wigner-Fano (BWF) equation (Pimenta et al., 2001) given by

$$I(\omega) = I_o \frac{[1 + (\omega - \omega_o)/q\Gamma]^2}{[1 + (\omega - \omega_o)/\Gamma]^2} \quad (2)$$

where I represents the Raman intensity, I_o is the background, ω is the Raman frequency shift, ω_o is the natural Raman frequency shift, q is the asymmetry parameter and Γ is the line width. The results of the analysis of these peaks showed

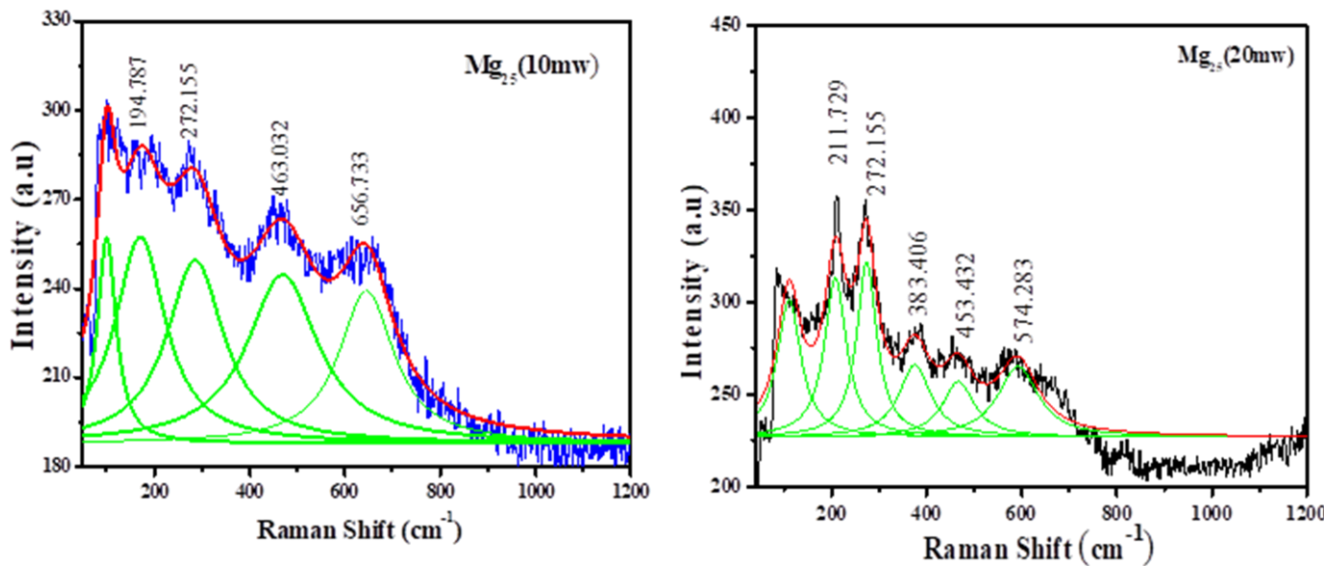


Fig 6 : Raman shift for $(Mg_{0.25}Co_{0.75}Fe_2O_4)$ nanoparticles annealed at $450^{\circ}C$ (a) for 10 mW, (b) for 20 mw

Table 1. Raman spectroscopy measurements of $(Mg_{0.25}Co_{0.75}Fe_2O_4)$ nanoparticles annealed at $450^{\circ}C$

Compound	Laser power (mW)	A_{1g} (cm^{-1})	E_g (cm^{-1})	$T_{2g(1)}$ (cm^{-1})	$T_{2g(2)}$ (cm^{-1})	$T_{2g(3)}$ (cm^{-1})
$Mg_{0.25}Co_{0.75}Fe_2O_4$	10	194.787	272.155	463.032		656.733
	20	211.729	272.155	383.406	453.432	574.283

that their positions are at 686.94 cm^{-1} , 593.05 cm^{-1} , 458.78 cm^{-1} , 282.45 cm^{-1} and 217.14 cm^{-1} which correspond to the symmetry modes A_{1g} , E_g and $3T_{2g}$, respectively.

3.3 Magnetic Analysis

3.3.1 VSM Analysis

The magnetic measurements of cobalt doped magnesium ferrite nanoparticles $(Mg_{0.25}Co_{0.75}Fe_2O_4)$ are performed using the VSM technique and the results of magnetic hysteresis at room temperature are shown in Figure 7. The M–H measurement exhibits a clear hysteresis loop (Cherepanov et al., 2020). This proves that the Mg-doped with cobalt ferrite magnetic nanoparticles was ferromagnetic.

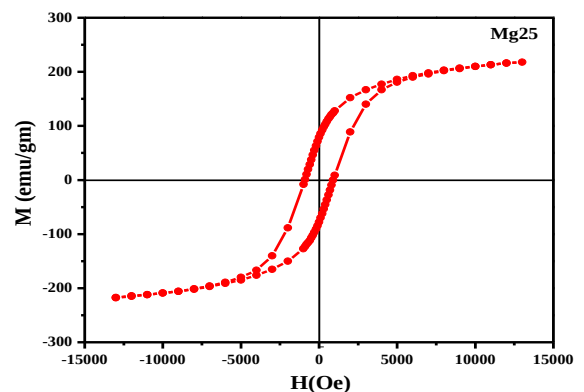


Fig 7: VSM measurements of $(Mg_{0.25}Co_{0.75}Fe_2O_4)$ nanoparticles annealed at $450^{\circ}C$

Table 2. VSM measurements of $(Mg_{0.25}Co_{0.75}Fe_2O_4)$ nanoparticles annealed at $450^{\circ}C$

The Sample	Magnetization emu/g Ms	Coercivity Hc	Retentivity emu/g Mr
$Mg_{0.25}Co_{0.75}Fe_2O_4$	119.016	200.688	15.534

3.3.2 Mossbauer Spectroscopy analysis

The Mossbauer spectra of the nanoparticles at room temperature are typically a superposition of magnetic (six lines) and quadrupole patterns (two lines). These two components are due to particles with super magnetic relaxation time that is long or short compared to the time scale of Mossbauer spectroscopy respectively (Cherepanov et al., 2020). The relative weight of the doublet increases with temperature and the Mossbauer absorption lines become broader. This type of line broadening and the presence of central peak are ascribed to Ferromagnetic. The experimental data were fitted with and table 3 shows the result of fitting (figure 8).

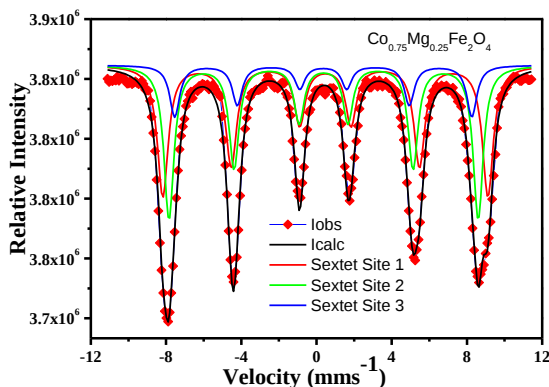


Fig 8: Mossbauer measurements of $(Mg_{0.25}Co_{0.75}Fe_2O_4)$ nanoparticles annealed at $450^\circ C$

Table 3. The analysis of Mossbauer spectroscopy results of $Mg_{0.25}Co_{0.75}Fe_2O_4$, at room temperature 300 K, I.S the isomer (chemical) shift, QS is the quadruple shift and HF is the hyperfine interaction.

The Sample	Subspectra	IS (mm/s)	QS (mm/s)	HF (T)
$Mg_{0.25}Co_{0.75}Fe_2O_4$	Sextet 1	0.4700		53.607
	Sextet 2	0.3755		50.991
	Sextet 3	0.3590		49.000

Conclusion

Cobalt doped magnesium ferrite nanoparticles $(Mg_{0.25}Co_{0.75}Fe_2O_4)$ annealed at $450^\circ C$ are prepared by the co-precipitation method. The FT-

IR spectra show main absorption bands around 578 cm^{-1} and 406 cm^{-1} corresponding to the vibration modes of the tetrahedral and octahedral sites respectively. XRD pattern reveals that the synthesized ferrites consist of Nanocrystalline particles with a size in the range $\approx 16 \pm 5\text{ nm}$. The SEM micrographs show uniformly distributed granular like structure. The magnetic properties of the synthesized nanoparticles are found to be improved because of cobalt doping. The synthesized materials can be tested for gas sensing properties.

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