Effect of temperature on structural and magnetic properties of $MgFe_2O_4$ nanoparticles prepared via co-precipitation method

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ABSTRACT

Magnesium ferrite nanoparticles were prepared by co-precipitation method. The prepared samples were calcinated at different temperatures such as 600 °C, 700 °C and 800 °C and characterized by using XRD, FTIR and VSM. Magnesium ferrite nanoparticles calcinated at 700 °C were further characterized by using FESEM with EDAX, HRTEM with SAED pattern. TG/DT analysis shows that the prepared sample was thermally stable above 570 °C. XRD analysis has shown that the formation of $MgFe_2O_4$ nanoparticles, also the crystallite size increase with increase of temperature. The functional group present in the sample was identified by using FTIR analysis, which indicates the presence of absorption bands at 564 cm^{-1} and 436 cm^{-1} are attributed to the vibrations of tetrahedral and octahedral complexes of magnesium ferrite nanoparticles. The surface morphology of $MgFe_2O_4$ studied by FESEM was cubic spinel structure and it was confirmed by HRTEM. The element identified in the $MgFe_2O_4$ nanoparticles from EDAX analysis were Fe, Mg and O. From the VSM study, the enhanced saturation magnetization value is observed from 20 to 32 $emug^{-1}$ for the different calcination temperature which shows that the soft ferrimagnetic behaviour and it is suitable for magnetic recording media devices and microwave applications.

Keywords: magnesium ferrite, XRD, FTIR, FESEM, HRTEM and VSM

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1 INTRODUCTION

Magnetic particles in the nanometer scale are attracted in recent days because of their technological applications and unique magnetic properties [1]. Ferrites belong to the family of magnetic materials which exhibit remarkable magnetic properties [2]. The spinel ferrites with a general formula of MFe_2O_4 (M= Ni, Co, Mg, Mn, Zn) have attracted enormous attention in recent days. Among the spinel ferrites, $MgFe_2O_4$ is a soft magnetic n-type semiconducting material which finds numerous applications in various fields such as microwave devices, computer memory chips and high density recording media [3, 4]. The magnesium ferrite has high chemical stability at room temperature and is a partially inverse cubic spinel type structure, belonging to the space group Fd3m [5, 6]. The cationic distribution between the tetrahedral (A) and octahedral [B] sites is often represented by $(Mg^{2+}_{1-x} Fe^{3+}_{x})_A[Mg^{2+}_{x} Fe^{3+}_{2-x}]_B O_4$. Where x represents the degree of cation inversion in the structure, which is related to the fractions of

Mg^{2+} and Fe^{3+} ions occupying the (A) or [B] sites [7]. The magnetic properties of the spinel ferrites depend upon the choice of the cation and their distribution between A and B sites of the spinel lattice which is highly sensitive to preparation methods, annealing temperature, chemical composition and nature of dopants [8]. Magnesium ferrite nanoparticles can be prepared by different methods such as chemical co-precipitation [9], sol-gel [10], electrospinning [11], hydrothermal [12], and ball milling [13]. Apart from the other methods, chemical co-precipitation method can produce ultrafine, high purity, crystalline and high yield nanoparticles in shortest duration [14, 15]. In the present work, chemical co-precipitation technique is utilized to synthesize the magnesium ferrite nanoparticles and the synthesized particles are characterized by XRD, FTIR, FESEM with EDAX, HRTEM, and VSM.

2 Materials and methods

Magnesium ferrite nanoparticles were synthesized by using co-precipitation method [16]. To synthesize $MgFe_2O_4$ nanoparticles, 0.2M of ferric nitrate nonahydrate $[Fe(NO_3)_3.9H_2O]$ and 0.1M of magnesium nitrate hexahydrate $[Mg(NO_3)_2.6H_2O]$ were dissolved in 20 ml of de-ionized water respectively. The pH value of the nitrate solution was maintained at 9, by adding the required amount of sodium hydroxide (NaOH) solution drop wise as a precipitate agent. After the addition of NaOH solution a brown color solution was observed, then the solution was stirred continuously for 2 hours at 80 °C stirred at 600 rpm in order to obtain the precipitate. Then the final precipitate was filtered and washed a number of times by de-ionized water and acetone to remove the impurities. Finally, the synthesized sample was dried in hot air oven at 100 °C for two hours and further it was calcinated for 2 hours at 600 °C, 700 °C and 800 °C.

3. RESULTS AND DISCUSSION

3.1. Thermo gravimetric analysis (TG/DTA)

Simultaneous thermo gravimetric and differential thermal analysis of the as-synthesized magnesium ferrite nanoparticles is carried out in the temperature range from 35 °C to 1000 °C at the rate of 20 °C / minute. The TG/DTA curve of the as-prepared product is shown in Fig 1.

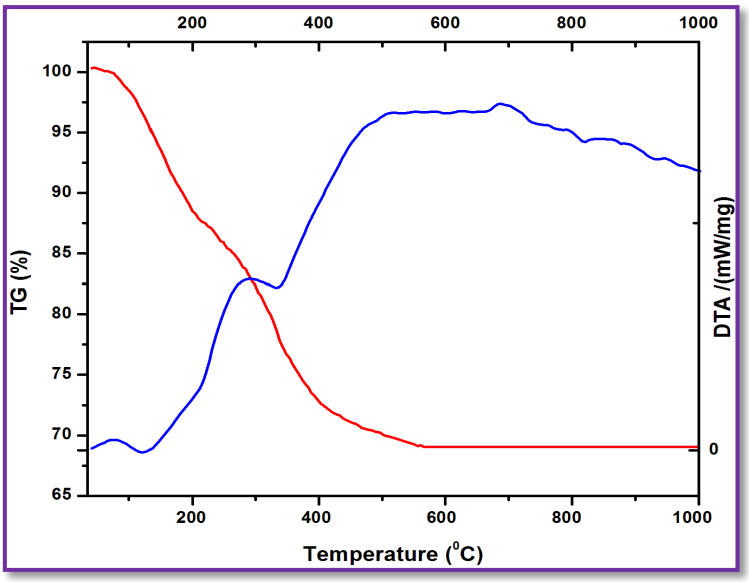

Fig.1. TG/DT analysis of as-prepared $MgFe_2O_4$ nanoparticles

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From Fig.1, it is found that there are three weight losses from the TG analysis. The first weight loss has been observed in the range of 35 to 113 °C (12 %) is due to elimination of water. The second major weight loss of 15 % is observed in the range 114 °C to 416 °C owing to the decomposition of nitrate and organic templates. The third weight loss of 3 % has been observed in the range of 417 °C to 567 °C owing to the crystallization of the final product. No weight losses occur beyond 570 °C, which indicates the formation of magnesium ferrite nanoparticles. This tendency is in line with Somvanshi *et al.,* for the synthesis of $MgFe_2O_4$ nanoparticles by auto-combustion assisted sol-gel route [17]. DTA analysis showed three endothermic peaks at 113 °C, 334 °C and 546 °C which are shown in Fig.1. The first endothermic peak observed at 113 °C due to desorption of water, second endothermic peak observed at 334 °C due to thermal decomposition of organic templates and peak occurred at 546 °C which corresponds to continuous dehydroxylation and formation of final product. Similar result was reported by Aliyan *et al.,* for the synthesis of magnesium ferrite nanoparticles using chemical coprecipitation method [18]. From this observation, it may confirmed that the annealing process is essential to stabilize the magnesium ferrite nanoparticles.

3.2 X-ray diffraction analysis

X-ray diffraction of $MgFe_2O_4$ nanoparticles as-prepared and calcinated samples at 600 °C, 700 °C and 800 °C is shown in Fig.2.

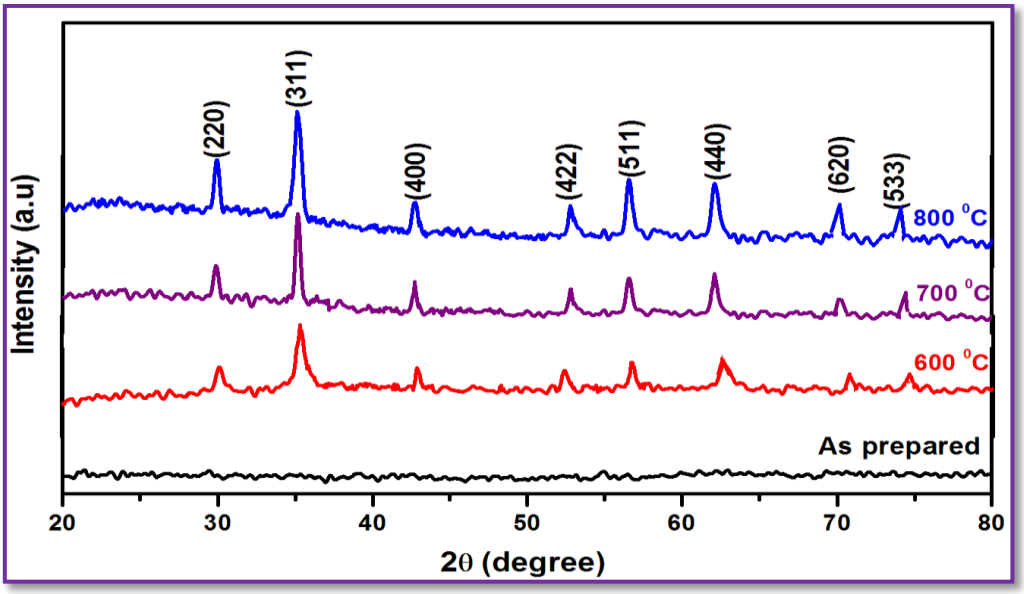

Fig.2. XRD spectra of as-prepared and calcinated $MgFe_2O_4$ nanoparticles at 600 °C, 700 °C, 800 °C

The prepared $MgFe_2O_4$ nanoparticles indicate the amorphous nature and confirm that calcination is essential to stabilize the crystallinity and prevent their agglomeration [19]. The XRD pattern of $MgFe_2O_4$ nanoparticles for all the calcinated samples, exhibits the reflections with h, k, l values of (220), (311), (400), (422), (511), (440), (620) and (533) planes specify the cubic phase with space group Fd3m [20]. The obtained XRD peaks are well matched with the standard characteristic peaks of the cubic phase of $MgFe_2O_4$ reported in JCPDS, file No #73-1720 [21]. The observed peaks in this work obviously designate the formation of magnesium ferrite nanoparticles without any impurities. The XRD peaks are gradually getting narrowed with increase of calcination

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temperature from 600 °C to 700 °C and 700 °C to 800 °C. This observation clearly demonstrate that the increasing temperature solely responsible for enhancing the crystallite size of the nanoparticles. Further the sharp and intense peaks observed at 800 °C indicate a high degree of crystallinity of the synthesized nanoparticles. Similar results were observed by Prajapat *et al.,* for magnesium ferrite nanoparticles prepared by sol-gel auto-combustion method [22]. From the present study it may be conclude that magnesium ferrite nanoparticles transforms from a low degree of crystallization to the high degree of crystallization with an increase in calcination temperatures. The average crystallite size has been calculated using Debye Scherer's formula.

$$D = K\lambda/\beta \cos\theta \quad (1)$$

Where D is the crystallite size, K=0.9, is the Scherer's constant, 'λ' is the incident wavelength of X-Ray (1.5406 Å), β is the full width of the diffraction line at half maximum (FWHM) intensity measured in radians, and θ is the Bragg's angle are observed. The Lattice Parameter ''a'' can be estimated by using the relation $a = d_{hkl}\sqrt{h^2 + k^2 + l^2}$ (2)

The crystallite size and lattice parameter of magnesium ferrite nanoparticles calcinated at different temperature are provided in Table 1.

Table 1. Structural parameter of $MgFe_2O_4$ nanoparticles calcinated at 600 °C, 700 °C and 800 °C

Temperature (°C)	$MgFe_2O_4$	
	Crystallite size (nm)	Lattice constant (nm)
600	18	0.8341
700	24	0.8360
800	28	0.8387

The observed values of crystallite size 18 nm, 24 nm and 28 nm for 600 °C, 700 °C and 800 °C respectively. The particle size increment with increase of calcination temperatures observed in the present study clearly demonstrates the growth of the $MgFe_2O_4$ nanoparticles. This observation is in line with the results of Khanna *et al.,* for prepared magnesium ferrite nanoparticles by the sol gel method [23].

The lattice parameter "a" value is 0.8341 nm, 0.8360 nm, 0.8387 nm for $MgFe_2O_4$ nanoparticles calcinated at 600 °C, 700 °C and 800 °C respectively. Similar observation was carried out by Singh *et al.,* have observed that the lattice parameter of (a) values 0.8371 nm, 0.8376 nm, 0.8389 nm for calcination temperature 500 °C, 600 °C and 800 °C respectively [24]. The value of lattice constant increases with calcination temperatures are observed in the present study reveals that the changes in ionic radii and cation redistribution between the tetrahedral and octahedral sites.

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3.3. FTIR analysis

The FTIR spectrum of $MgFe_2O_4$ nanoparticles calcinated at different temperatures are recorded in the range of 4000-400 cm^{-1} through the solid phase as shown in Fig.3.

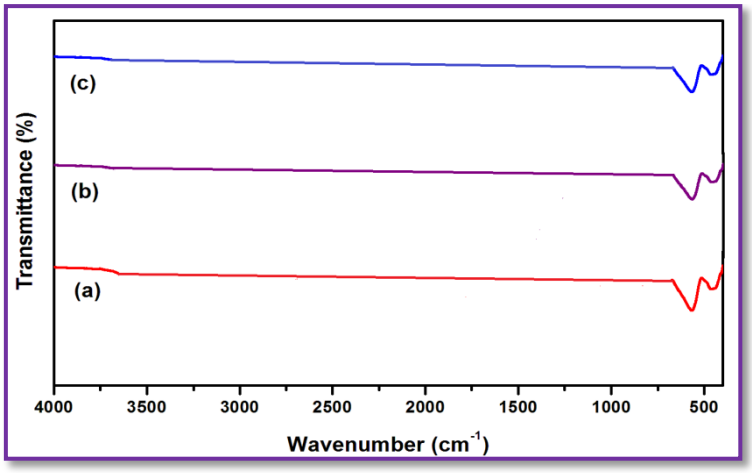

Fig.3. (a), (b) and (c) FTIR spectra of $MgFe_2O_4$ nanoparticles calcinated at 600 °C, 700 °C, 800 °C

The present work exposes that there are two absorption bands between the region 600-400 cm^{-1}, which corresponds to the tetrahedral and octahedral sites of $MgFe_2O_4$ nanoparticles. The higher absorption band around v_1 is around at 562 cm^{-1} corresponds to the stretching vibrations of tetrahedral complexes of Mg-O. The lower absorption band around v_2 is around at 434 cm^{-1} is accredited to the vibrations of octahedral groups Fe-O is shown in Table 2.

Table 2. The absorption band of $MgFe_2O_4$ nanoparticles calcinated at 600 °C, 700 °C and 800 °C

Temperature (°C)	$MgFe_2O_4$	
	v_1 (cm^{-1})	v_2 (cm^{-1})
600 °C	562	434
700 °C	564	436
800 °C	566	438

These absorption bands v_1 and v_2 observed in the present work represents the characteristic features of spinel ferrites in single phase. Erdawati *et al.,* have made similar observation for the synthesis of magnesium ferrite nanoparticles at 600 °C and 800 °C by employing sol gel method [25]. Saravanan *et al.,* have observed similar behavior for the preparation of magnesium ferrite nanoparticles at 500 °C, 700 °C and 900 °C by combustion method [26].

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3.4. FESEM with EDAX analysis

The morphology of the $MgFe_2O_4$ nanoparticles calcinated at 700 °C has been examined through FESEM, as shown in Fig.4a-b.

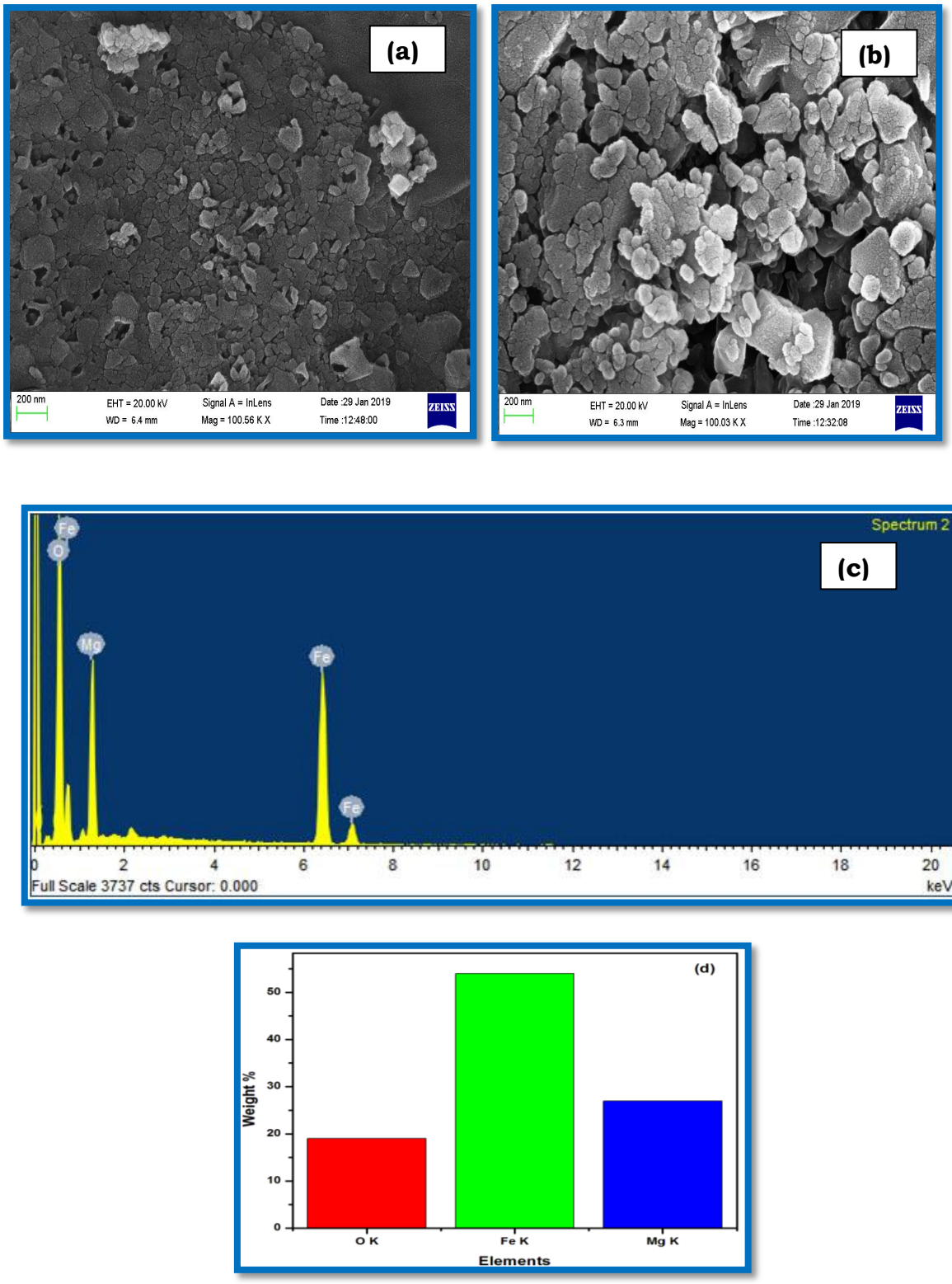

Fig. 4 (a), (b), FESEM micrographs of $MgFe_2O_4$ nanoparticles calcinated at 700 °C for different magnification (c) EDAX, and (d) Corresponding weight percentage

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The $MgFe_2O_4$ nanoparticles show the spherical shape with small agglomeration at few places. This agglomeration is observed due to the naturally happening interaction between magnetic nanoparticles. Similar conclusion was drawn by Nguyen *et al.,* for $MgFe_2O_4$ nanoparticles calcinated at 600 °C was synthesized by solution combustion method [27]. The elemental composition of the synthesized $MgFe_2O_4$ nanoparticles is studied by using EDAX spectrum. From the EDAX pattern, the spectra contain the occurrence of Fe, Mg, and O as the elements in the synthesized material with no impurities and its elemental mapping is shown in Fig. 4c. The corresponding histogram of weight percentages is shown in Fig.4d. Similar observation was observed by Zhao *et al.,* for the preparation of $MgFe_2O_4$ nanoparticles calcinated at 600 °C by co-precipitation route [28].

3.5 High-resolution Transmission Electron Microscopy

HRTEM micrographs helps to visualize the inherent matrix morphology and structure of the synthesized $MgFe_2O_4$ nanoparticles are shown in Fig. 5 a, b.

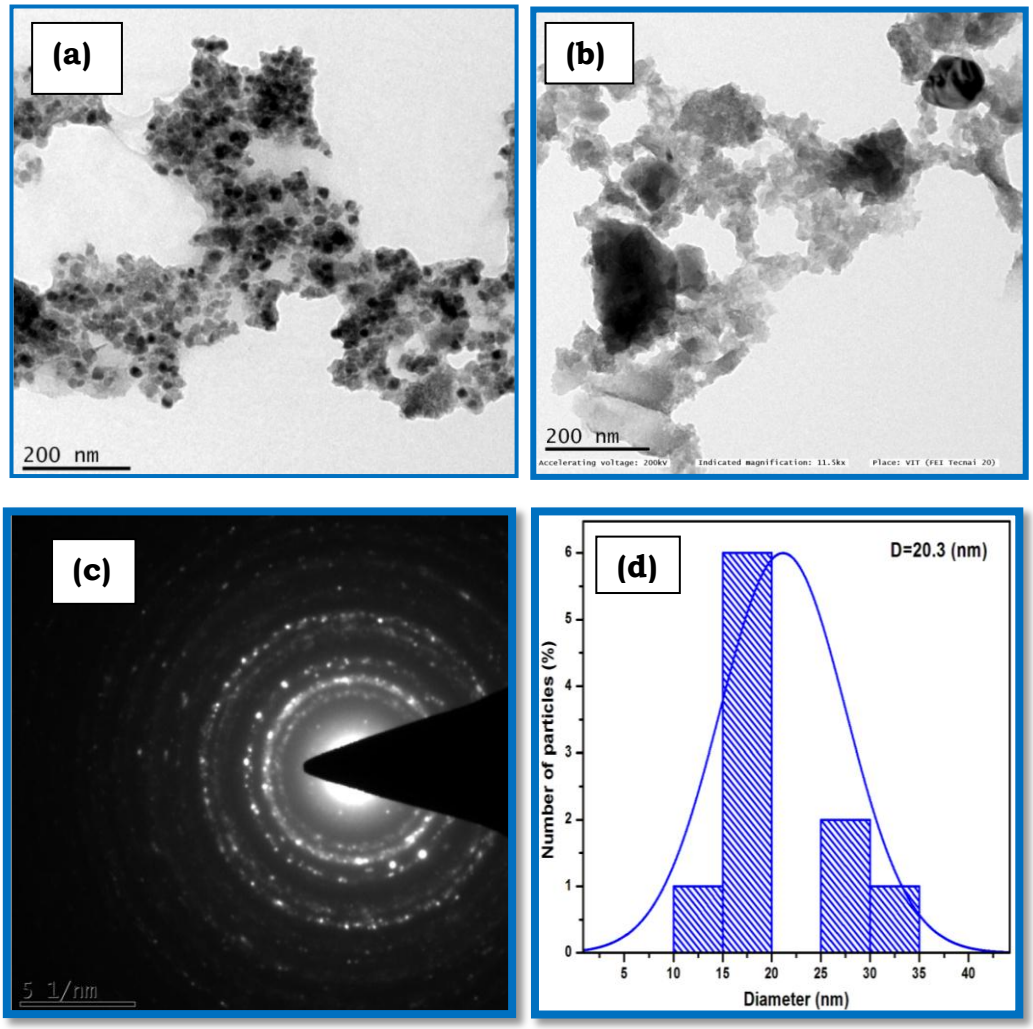

Fig.5 (a), (b), HRTEM micrographs of $MgFe_2O_4$ nanoparticles calcinated at 700 °C with various magnifications (c) SAED and (d) Corresponding histogram size

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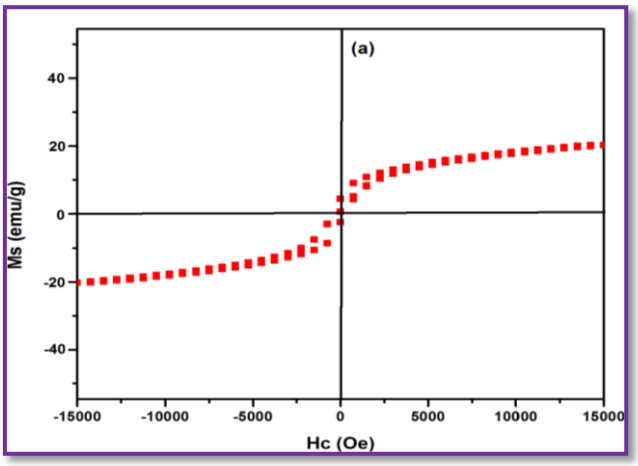
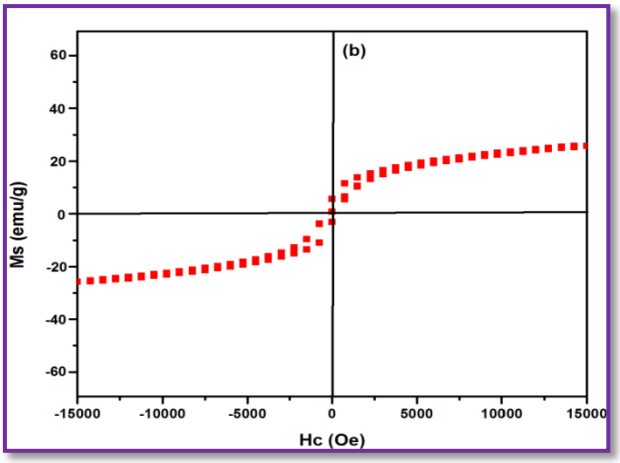
The HRTEM micrographs of $MgFe_2O_4$ nanoparticles are calcinated at 700 °C with various magnifications, which revealed the clear morphology, and spherical shape with some agglomeration. The observed result in the present work agrees well with Hussein *et al.,* for synthesis of $MgFe_2O_4$ by sol gel auto combustion method calcinated at 600 °C [29]. Fig. 5c indicates the selected area electron diffraction (SAED) analysis representing sharp rings, which reveal the good crystallinity of the nanoparticles. The size distribution histogram presented in Fig. 5(d) is obtained by counting from the micrographs. The length of the individual selected particles using the "Image J viewer software" and its mean particle size of 20 nm observed in HRTEM which is in good agreement with the XRD result. Similar result was observed by Kaur *et al.,* for the preparation of $MgFe_2O_4$ nanoparticles calcinated at 500 °C by co-precipitation method [30]. Further they concluded that agglomeration phenomenon was observed as small particles aggregated in order to achieve lower free energy state.

3.6 MAGNETIC PROPERTIES

The magnetic characterization of $MgFe_2O_4$ nanoparticles calcinated at 600 °C, 700 °C and 800 °C was done by VSM at room temperature with an applied magnetic field from -15 kOe to 15 kOe and is shown in Fig. 6 (a), (b), (c).





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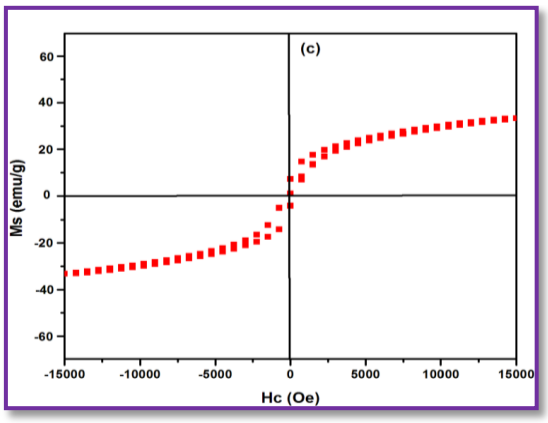

Fig.6. (a), (b) and (c) Room temperature M-H curves for $MgFe_2O_4$ nanoparticles calcinated at 600 °C, 700 °C and 800 °C

It is clear from Fig.6 (a), (b) and (c) that the saturation magnetization (Ms) could not be achieved even for an applied field of ± 15 kOe. The observed behaviour in the present study reveals that the synthesized $MgFe_2O_4$ nanoparticles are ferrimagnetic nature. Kaur *et al.,* have observed the similar pattern by employing sol–gel auto-combustion method and concluded that the $MgFe_2O_4$ nanoparticles are ferrimagnetic [31]. The non-saturation of magnetization is observed in the present study is due to the existence of surface spin disorder of the nanoparticles. Similar conclusion was reported by Kanagesan *et al.*, for the preparation of $MgFe_2O_4$ nanoparticles by microemulsion technique [32]. In spinel ferrites, there are three kinds of magnetic interactions which are possible between the metallic ions through the intermediate O^{2-} ions, namely A-A interactions, B-B interactions, and A-B interactions. Among these, A-B interaction is predominant than A-A and B-B interaction. The effective magnetization M is a result of the difference of the magnetization $\sum M_B$ and $\sum M_A$ of the [B] and (A) sites (i.e.) M=$\sum M_B$ - $\sum M_A$. The magnetic moment of Mg^{2+} is zero, so in $MgFe_2O_4$ magnetic couplings originate from the magnetic moment of Fe^{3+} cations [33]. The saturation magnetization value was determined by the extrapolation of inverse of the field vs. magnetization (M) plot to 1/H=0. The magnetic parameters such as saturation magnetization and coercivity values of $MgFe_2O_4$ nanoparticles with different annealing temperatures namely 600, 700 and 800 °C are shown in Table 3.

Table 3. Magnetic parameters of $MgFe_2O_4$ nanoparticles calcinated temperature at 600 °C, 700 °C and 800 °C

Temperature (°C)	Ms (emu/g)	Hc (Oe)
600	20	39
700	26	56
800	32	94

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The calculated values of saturations magnetizations of the prepared $MgFe_2O_4$ samples are found to be 20, 26, 32 emu/g for calcinated temperature of 600 °C, 700 °C, 800 °C respectively which is smaller than that of the bulk magnesium ferrite 35 emu/g [34]. The reduction of saturation magnetization may be due to the surface structural distortions and spin canting existing in the whole volume of the nanoparticles. In addition, the different cation distributions in magnesium ferrite may have also reduced its saturation magnetization value compared to their bulk counterpart [35]. The coercivity value of $MgFe_2O_4$ nanoparticles are 39 Oe, 56 Oe and 94 Oe for calcinated temperature is 600 °C, 700 °C and 800 °C respectively. The observed coercivity values in the present work clearly indicate that the prepared $MgFe_2O_4$ nanoparticles are soft ferrimagnetic nature. Similar observation was made by Bergmann *et al.,* for the $MgFe_2O_4$ nanoparticles calcined at 650 °C by combustion method [36]. The increase of saturation magnetization and coercivity with increase of temperature is also observed in the present study is due to the part of magnetic Fe^{3+} ions present in A- site migrate to B-site and push some non-magnetic Mg^{2+} ions to A-site which enhances the magnetic ions in octahedral site. Further the increase of saturation magnetization proves that the average magnetic domain size of the nanoparticle gets increased and the spins become more aligned with the direction of the applied magnetic field. Similar observation was observed by Amer *et al.,* for the synthesized magnesium ferrite nanoparticles using coprecipitation method [37]. The saturation magnetization may correlate with the increase of crystallite size and surface/volume ratio of the particles as well as with the ordering of cations at the octahedral and tetrahedral sites with the increase of temperature [38]. Huang *et al*., was observed similar result and they concluded that the improvement of crystallinity and the decrease of the concentration of oxygen vacancies have been considered as being responsible for the increase of the saturation magnetization [39]. Sangmanee *et al*., reported that crystallite size 27 nm and corresponding saturation magnetization was 31 emu/g for $MgFe_2O_4$ nanoparticles annealed at 800 °C which is lower than that of the present work with crystallite size 28 nm with saturation magnetization 32 emu/g [40]. The superexchange interaction, magnetocrystalline anisotropy, and spin-canting effect, are more prevalent in the nanoparticles of ferrites, and hence the magnetic property observed in the system of nanoferrites is a cumulative effect of these interactions. The temperature and coercivity values are direct relation between them are observed in the present study and reveals that the prepared magnesium ferrite nanoparticles are single domain nature. Similar trend was observed by Pradeep *et al*., for preparation of $MgFe_2O_4$ nanoparticles by sol–gel route [41]. The present work a pure $MgFe_2O_4$ nanoparticles are synthesized which have high saturation magnetization, and coercivity values are suitable for fabricating new magnetic materials.

4. CONCLUSION

$MgFe_2O_4$ nanoparticles are prepared by a simple, high yield and low cost co-precipitation method. No weight loss was noticed from 570 °C in TGA and exothermic peak at 546 °C in DTA study confirmed the formation of $MgFe_2O_4$ nanoparticles. The variations of the structural factors with increase of calcination temperature were linearly connected with cation distribution between tetrahedral (A) and octahedral [B] sites of the $MgFe_2O_4$ nanoparticles. XRD

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results revealed the phase pure with cubic structure of $MgFe_2O_4$ nanoparticles. The crystallite size increased with the calcination temperatures. Metal Fe-O and Mg-O stretching vibrations observed by FTIR spectrum revealed that the synthesized product of magnesium ferrite nanoparticles. Morphological study of FESEM and HRTEM images showed that the particles are spherical shape. EDAX spectra confirmed the elemental composition of Fe, Mg and O in the synthesized $MgFe_2O_4$ nanoparticles. The observed saturation magnetization and coercivity values linearly vary with temperatures shows that the synthesized materials are soft magnetic material in nature, and it is suitable for sensor elements in technological devices, magnetic recording media devices and microwave applications.

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