

Synthesis, Structural and Optical Properties of Mg-Cd Nano Particles

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Abstract: The nano ferrite particles of $Mg_xCd_{1-x}Fe_2O_4$ ($x=0.1, 0.3, 0.5, 0.7, 0.9$, and 1.0) were synthesized by Citrate gel auto combustion method. The synthesized samples were characterized by XRD, FTIR and UV-Vis spectroscopy. A single-phase cubic spinel structure was detected by XRD analysis. The average crystalline size increased up to $x=0.9$ and it decreased suddenly for $x=1.0$. Variation in lattice constant obeys Vigard's law. FTIR characteristic peaks were observed which indicates the spinel nano ferrite structure of synthesized ferrites. Band gap energy of the samples were measured by using optical absorption properties were studied by UV-Visible spectral technique.

Keywords: Citrate gel auto combustion method, Mg-Cd nano ferrites, XRD, FTIR and UV-Visible

1.Introduction

The study of nano ferrites attracted significance due to wide range applications and properties shown by the nano ferrites in various fields. Nano ferrites have variety of applications in the fields of chemistry, physics, biology, medical and engineering. Investigating the structure of nano ferrites and their properties is an important part of material science. Properties and applications of nano ferrites are influenced by their microstructure. Synthesis of nano ferrites has been focused by the researchers because of their significant optical, electrical and magnetic properties than that of their bulk material [1]. The materials properties could be depended on method of synthesis, chemical composition, sintering temperature and cation distribution. The cations can either be in a tetrahedral or an octahedral site. Some of the methods for synthesizing ceramic materials are sol-gel, coprecipitation, hydrothermal method, etc. [2-4,]. Nano ferrites that exhibit magnetic behaviour have various technological applications and can be used in the fabrication of magnetic, high frequency transformers, storage devices and various microwave and radar devices [5]. Spinel ferrites are also applied as magnetic adsorbents in wastewater treatment technique. The properties of these materials are strongly influenced by the size distribution, morphology, shape, density of particles which in turn is sensitive to the method of preparation. Spinel ferrites, MFe_2O_4 ($M=Cd, Co, Zn, Mg$ etc.), are a family of nano ferrites in which M represent divalent cation occupies tetrahedral void and Fe^{3+} is a trivalent cation occupies octahedral voids. The physical and chemical properties of the spinel ferrites depend on the distribution of cations in tetrahedral and octahedral voids. The properties like

saturation magnetization, Curie temperature are strongly dependent on the distribution of cations and the type of doping cation. Spinel ferrites can be formed as a normal spinel $[M^{2+}]_A [Fe^{3+} Fe^{3+}]_B$ or inverse spinel $(Fe^{3+})_A [M^{2+} Fe^{3+}]_B$ depending on the distribution of cations in the tetrahedral (A) and octahedral voids (B) with the oxide ions. Doping of ferrite can enhance its physical properties and different types of divalent cations with considerable ionic size can be substituted in the interstitial sites of spinel ferrite [6].

In this work, characterization by XRD, FTIR and optical properties are reported for spinel ferrite system $Mg_x Cd_{(1-x)} Fe_2O_4$ ($x=0.1, 0.3, 0.5, 0.7, 0.9, 1.0$) has been prepared by citrate gel auto combustion method.

2.Materials and Experimental Method

Nano ferrite particles Ferrite of chemical formula $Mg_x Cd_{1-x} Fe_2O_4$ ($x=0.1, 0.3, 0.5, 0.7, 0.9, 1.0$) synthesized by using Magnesium Nitrate, Cadmium Nitrate, Ferric Nitrate, Citric acid and Ammonia of 99% purity as raw materials the citrate-gel auto combustion technique at room temperature. Calculated and amount of metal nitrates and citric acid as per the composition of the sample were dissolved in double distilled water separately and was stirred to form homogenous solution, mixed together and heated up to $80^\circ C$. Later the pH was maintained to 7 by adding ammonia solution drop wise. The final mixture of solutions was heated at about $200^\circ C$ with help of magnetic stirrer, auto combustion occurred and produced powder which was grinded with Agate Mortar and calcinated at $500^\circ C$ for 4 hours and cooled to normal temperature [7].

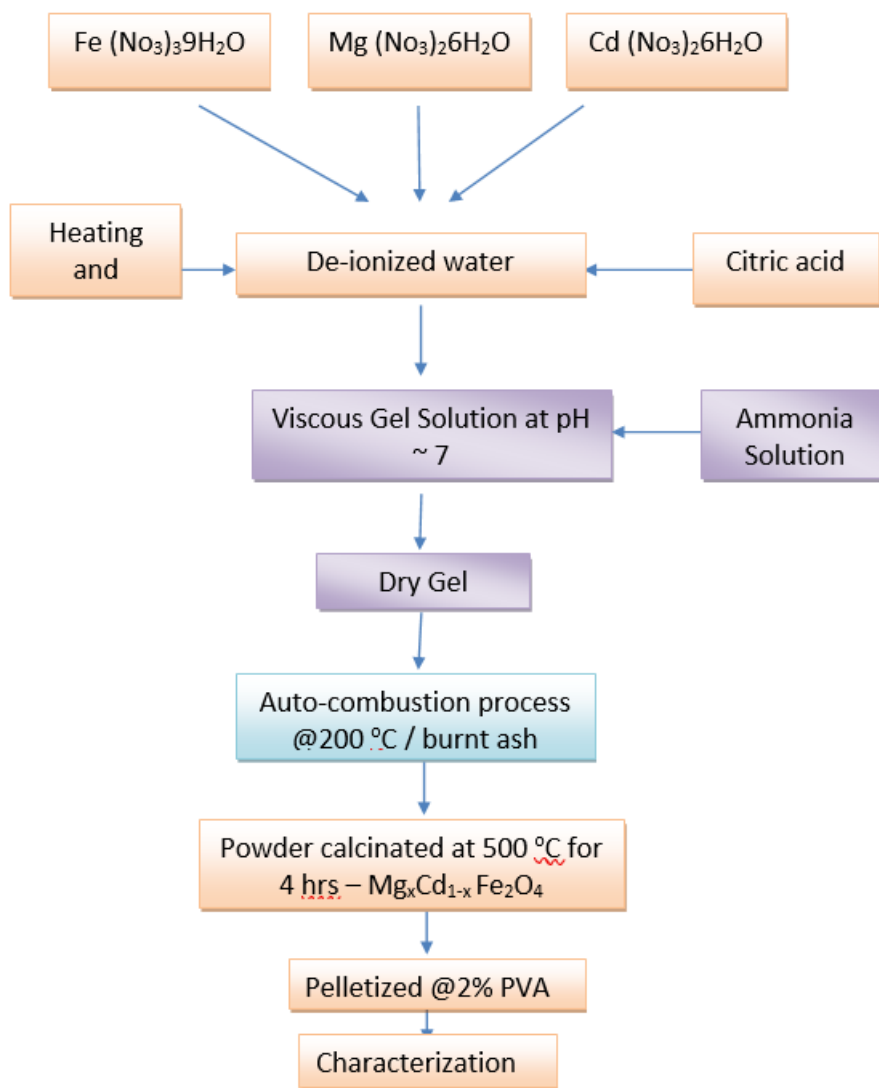


Figure 1: Flow chart of synthesis of Mg-Cd nano ferrites

Figure 1 shows the flow chart for synthesis of Mg-Cd spinel nano ferrites. Structural characterization of prepared samples investigated with X-ray diffractometer (Philips) using Cu K_{α} radiation ($\lambda=1.5405 \text{ \AA}$). The spinel structure of ferrites was confirmed by FTIR spectral analysis. The optical properties of synthesized nano ferrites was studied by UV-DRS (diffuse reflectance spectroscopy). The UV-DRS spectroscopy absorption and reflection in the UV region, barium sulphate (BaSO_4) is used as reference with absorbance verses wavelength.

3.Results and Discussion

3.1 XRD analysis of Mg-Cd nano ferrites

Figure 2 depicts the XRD pattern of low temperature synthesized $\text{Mg}_x\text{Co}_{1-x}\text{Fe}_2\text{O}_4$ (where $X=0.1$ to 0.9 with 0.2 variation) spinel nano ferrites. From the XRD pattern revealed that the samples consist of cubic spinel with single phase structure, there is no presence of other impurity peaks in composition of samples. Samples cubic spinel structure and belongs to Fd_3m space group [8]. The presented peak positions of XRD were also confirmed by existence of the JCPDS data. The average crystalline size of the synthesized nano ferrites were calculated from

Debye Scherrer's equation [9]. $l = \frac{0.94\lambda}{\beta \cos \theta}$ Where λ =wavelength, β =FWHM, and θ =Bragg angle.

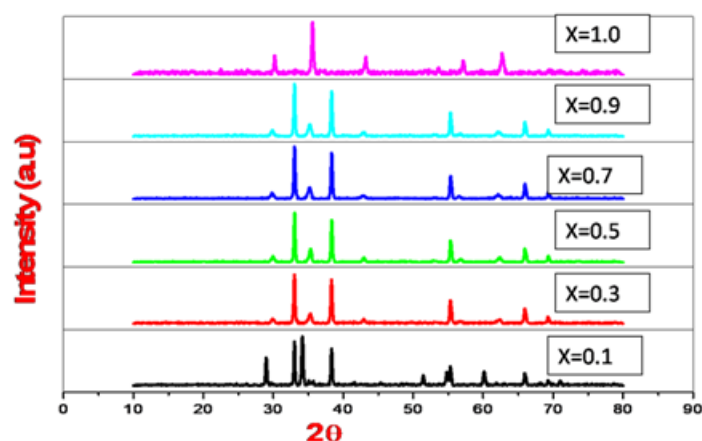


Figure 2: XRD patterns of Mg-Cd nano ferrites

The average crystalline size of pure sample (CdFe_2O_4) is 39 nm . The average crystalline size increased with increasing the doping concentration on pure sample from 39 to 44 nm , it could be due to smaller ionic radii of Mg^{2+} was substituted on larger ionic radii of Cd^{2+} ions.

Lattice constant of fabricated nano ferrites were calculated by the following equation. $a = d\sqrt{h^2 + k^2 + l^2}$, Where hkl indicates miller indices and, d indicates lattice constant. Mg-Cd nanoferrites lattice constant range from

8.9903 to 8.3669 [10]. Lattice constant was increased up to 0.8 molar ratio later it was decreased (8.3678 Å⁰), lattice constant varies it indicates obeys Vegard's law.

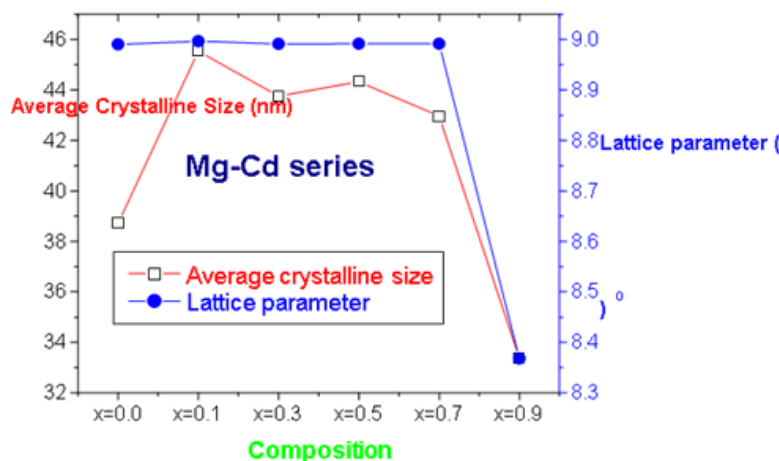


Figure 3: Composition Vs average crystalline size and lattice parameter

Volume of unit cell determined from following equation $v = a^3$ where a lattice constant Volume of unit cell varied with dopant concentration [11]. Volume of unit cell depends on lattice parameter of the material. The synthesized ferrite samples x-ray density were calculated from the following equation $d_x = \frac{8M}{Na^3}$, Where M is molecular wt. of the sample, N is Avogadro number, a is

lattice parameter. Pure Cd ferrite x-ray density was found to be 5.25 gm/cm³. X-ray density of the nano ferrites were increased from 5.56 to 4.32 gm/cm³. Mainly x-ray density was depends on the molecular wt. of the sample, lattice constant, dopant concentration and sintering temperature etc,

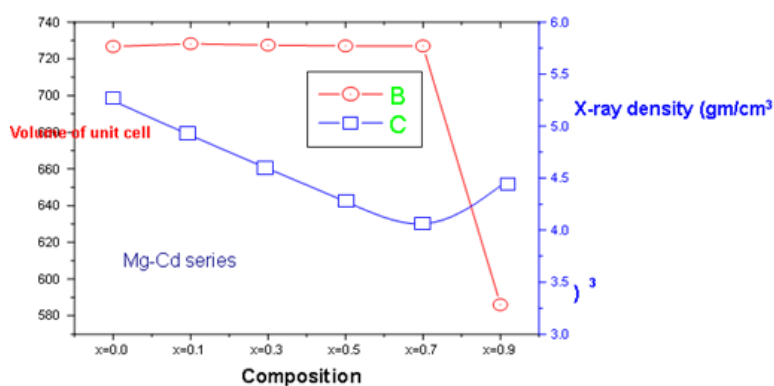


Figure 4: Composition Vs Volume of unit cell and X-ray density

All these parameters such as average crystalline size, lattice parameter, volume of unit cell (v), and x-ray density of the values were tabulated in Table 1. all the above-mentioned parameters variation depends on the

molar ratio of the sample, sintering temperature, method of fabrication, ionic radius of the elements and sintering condition etc.

Table 1: Average crystalline size, lattice parameter, volume of unit cell, x-ray density

Name of the composition	Average crystalline size (nm)	Lattice Parameter (Å ⁰)	Volume of unit cell	X-ray density (gm/cm ³)
Mg _{0.1} Cd _{0.9} Fe ₂ O ₄	37.72	8.9902	726.621	5.265
Mg _{0.3} Cd _{0.7} Fe ₂ O ₄	44.53	8.9963	728.101	4.932
Mg _{0.5} Cd _{0.5} Fe ₂ O ₄	43.66	8.9912	727.345	4.615
Mg _{0.7} Cd _{0.3} Fe ₂ O ₄	44.22	8.9913	726.88	4.301
Mg _{0.9} Cd _{0.1} Fe ₂ O ₄	41.96	8.9914	726.912	3.975
MgFe ₂ O ₄	34.35	8.3679	585.928	4.531

3.2 FTIR spectra of Mg-Cd nano ferrites

To know the structural characterization FTIR spectrum acts as a powerful technique. Figure 7 showed the FTIR of synthesized Mg-Cd nano ferrites. The FTIR graphs plotted transmittance versus wave number. In this spectra two main absorption bands were observed, that is higher frequency at 600 cm^{-1} and lower frequency at 400 cm^{-1} , these two absorption peaks suggest the spinel ferrite

character of the sample [12]. Vibrational spectra of synthesized nanoferrites were observed by Waldron and Hafner and attributed the high frequency band (ν_1) at about 600 cm^{-1} to tetrahedral (A) site and low frequency band (ν_2) at about 400 cm^{-1} to octahedral (B) site. The band positions difference it could be due to the differences in the Fe^{3+} and O^{2-} ions distance in octahedral and tetrahedral sites, it was observed in the present ferrite system [13].

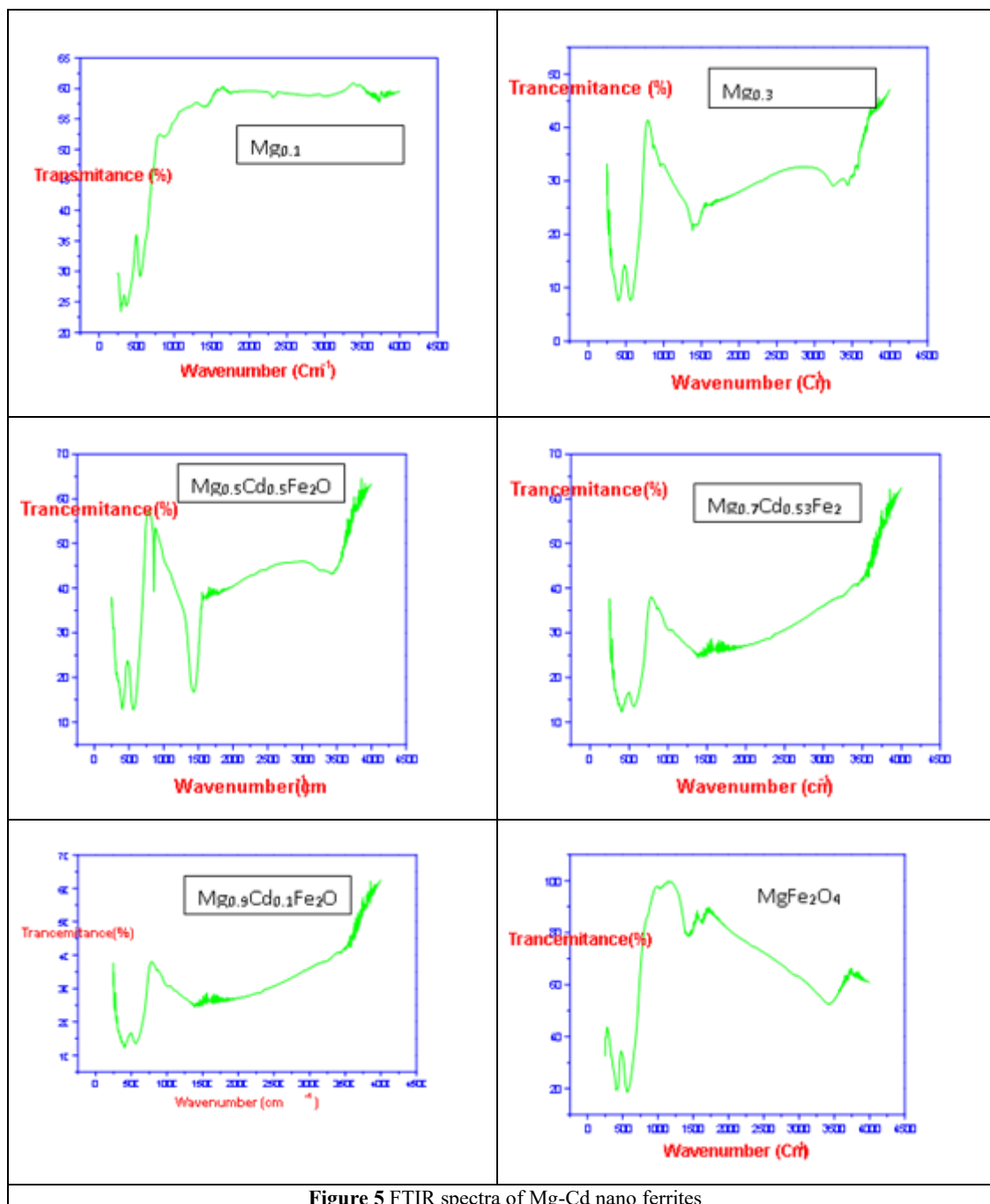


Figure 5 FTIR spectra of Mg-Cd nano ferrites

3.3 Optical Studies of Mg-Cd nano ferrites

To identify optical properties of nano ferrite samples, UV-Vis spectroscopy a powerful technique. Figure 8 depicts the UV-Vis spectra of synthesized Mg-Cd nano ferrites. UV-Vis spectra were recorded by UV-Vis Spectro photo meter with an integrating sphere between 200-800 nm

wavelength region [14]. in the field of optoelectronic applications band gap energy plays important role for semiconductor materials. From this UV-Vis spectra band gap energy of the sample were calculated by following equation $E = 1240/\lambda$, where λ is cut off wavelength [15].

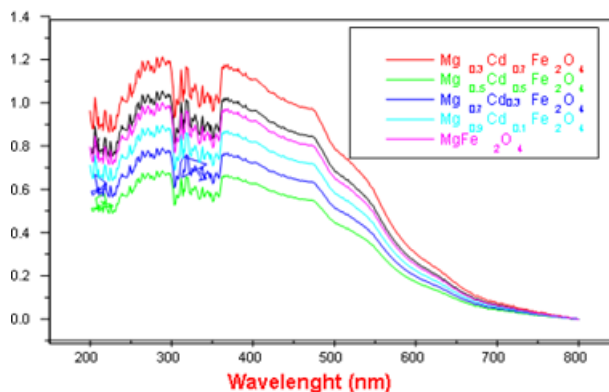


Figure 8 UV-Vis spectra of Mg-Cd nano ferrites

From the above equation band gap energy calculated, band gap energy for pure CdFe_2O_4 was 3.36 eV. Band gap energy increased from 3.36 to 3.56 eV with dopant

concentration Mg content it may be due to quantum confinement of samples [16]. These band gap energy values exhibit the semiconducting behaviour.

Table 3: Cut off wavelength and band gap energy of $\text{Mg}_x\text{Cd}_{1-x}\text{Fe}_2\text{O}_4$ $x=0.1$ to 1.0

Composition name	Cut off wavelength (nm)	Band gap energy (eV)
$\text{Mg}_{0.1}\text{Cd}_{0.9}\text{Fe}_2\text{O}_4$	368	3.35
$\text{Mg}_{0.3}\text{Cd}_{0.7}\text{Fe}_2\text{O}_4$	365	3.37
$\text{Mg}_{0.5}\text{Cd}_{0.5}\text{Fe}_2\text{O}_4$	359	3.43
$\text{Mg}_{0.7}\text{Cd}_{0.3}\text{Fe}_2\text{O}_4$	356	3.47
$\text{Mg}_{0.9}\text{Cd}_{0.1}\text{Fe}_2\text{O}_4$	352	3.51
MgFe_2O_4	347	3.57

4. Conclusions

Series of spinel nano ferrites with chemical formula $\text{Cd}_{1-x}\text{Mg}_x\text{Fe}_2\text{O}_4$ (where $x=0.1, 0.3, 0.5, 0.7, 0.9$ and 1.0) were fabricated from citrate gel auto combustion method. Phase conformation was confirmed by X-ray diffraction analysis, synthesized samples belong to cubic spinel structure, without any other impurity peaks. From x-ray diffraction analysis lattice parameter of the samples were observed, the variation of lattice parameter represents it obey s Vigard's law. From X-ray diffraction analysis samples average crystalline size, lattice parameter, volume of unit cell, x-ray density was calculated, all these parameters could be depended on the material synthesis method, sintering temperature, sintering condition, ionic radii of the elements etc. FTIR spectra of samples revealed that two IR peaks were observed which are stretching frequency of M-O bonds in tetrahedral sites and octahedral sites, it could be belongs spinel structure of the ferrites. From UV -Vis spectra samples band gap energy was observed it is near to 3.35 to 3.57 eV. The synthesized samples were exhibiting florescence nature; it could be uses in optoelectronic application of the materials.

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