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RESEARCH ARTICLE

Synthesis and Structural Investigation of Nano-Sized Cadmium Ferrite

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ABSTRACT

This report presents the synthesis of cadmium ferrite ($CdFe_2O_4$) by Oxalate co-precipitation and its subsequent characterization by using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) techniques. XRD results confirm the single cubic spinel phase formation with lattice parameter 8.7561A°. An infrared spectroscopy study shows the presence of main two absorption bands indicating the presence of tetrahedral and octahedral group complexes, respectively, within the spinel lattice. We also report strain (ψ), hopping length (L_A and L_B) and dislocation density (ρ_D) of ferrite sample.

Keyword: Cadmium ferrite, $CdFe_2O_4$, XRD, Dislocation density, Strain, SEM.

1. Introduction

Ferrites are ferromagnetic substances, with spinel crystalline structure of commercial importance due to their high frequency applications in different devices such as radio frequency coils, transformer cores etc. and thus many magnetic materials are replaced by the conventional magnetic materials [1]. Soft magnetic ferrites are used as low frequency inductors, antenna rods and wide-band and for high frequency electronic devices such as electromagnetic wave absorbers or inductor devices, due to initial permittivity in high frequency. Ferrimagnets having low RF loss are used in passive microwave components such as isolators, circulators, phase shifters, and miniature antennas operating in a wide range of frequencies (1–100 GHz) and as magnetic recording media owing to their novel physical properties [1-8].Ferrite powder, one of the very important ferrite materials has been considered for many applications such as high density magnetic storage media, MRI contrast agent, colour imaging, ferro-fluids, high frequency devices, magnetic refrigerators, catalysts, humidity sensors, gas sensor, magnetic-fludies, photo-magnetic materials, site-specific drug delivery and microwave device[8-10].Due to the unparalleled physical and chemical properties, synthesis and applications of nano-particles are focused in the research. Materials are very attractive perspective of their scientific and technological importance. Several methods like solid state method [11], auto-combustion [10], sucrose precursor [12], organic gel-thermal decomposition [13], combustion method [14], self-propagating method [15], co-precipitation [16,17], hydrothermal [18], micro-emulsion [19], thermolysis [20], and wet chemical co precipitation technique [21] are employed to synthesis the materials. The present work aimed at the synthesis and detailed structural and morphological properties of cadmium ferrites ($CdFe_2O_4$) by Oxalate co-precipitation. We have

investigated the basic structural parameters like particle size, grain size, absorption bands, strain (ψ)and dislocation density (ρ_D) of ferrite sample.

2. Materials and Methods

All the starting materials $CdSO_4 7H_2O$ (purity 99.99%, Sd fine) and $FeSO_4 7H_2O$ (purity 99.5%, Thomas Baker) were of analytical grade is used to synthesis the nano $CdFe_2O_4$by standard oxalate co-precipitate method as shown in the Figure 1.

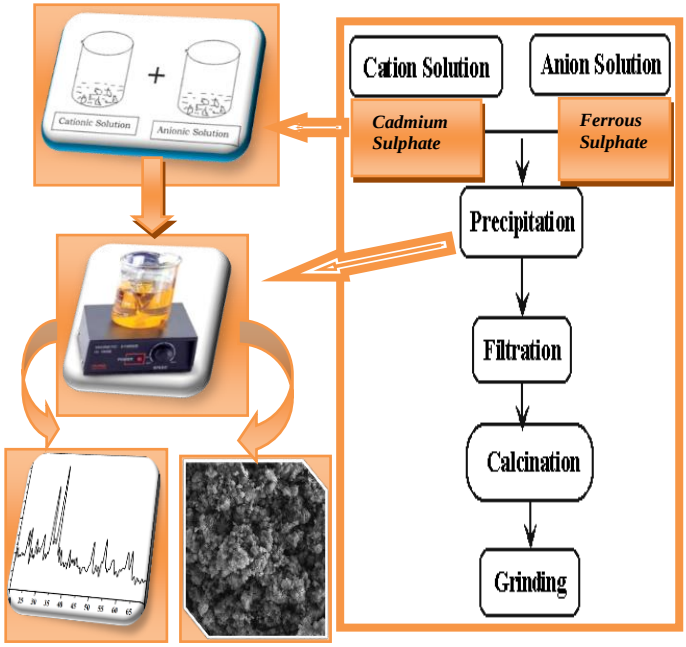

Figure 1: *Schematic representation of co-precipitation route.*

Filtered and dried powder is pre-sintered at 400^{0}C for 5 hours and final sintering is done at 500^{o}C for 4 hours. Structural characterization of the ferrite powders was carried out on Philips Diffractometer (XRD), (with Cu-K_α radiation, wavelength,λ= 1.54 Å). The scanning electron micrograph of the sample was taken on JEOL JSM 6360 SEM machine. FTIR spectra analysis carried out (using Perkin Elmer Model No. 783) in the wave numbers between 400 cm^{-1} and 4000 cm^{-1} to identify and understand the aspect of bonding in the present samples with KBr solvent.

3. Results and Discussions

3.1 XRD Studies

To evaluate the crystal structure of $CdFe_2O_4$ analysis were carried out and the XRD images of the samples are presented in Figure 2. The diffraction pattern analysis by using (220), (311), (222), (400), and (511) reflection planes confirms the cubic spinel structures.

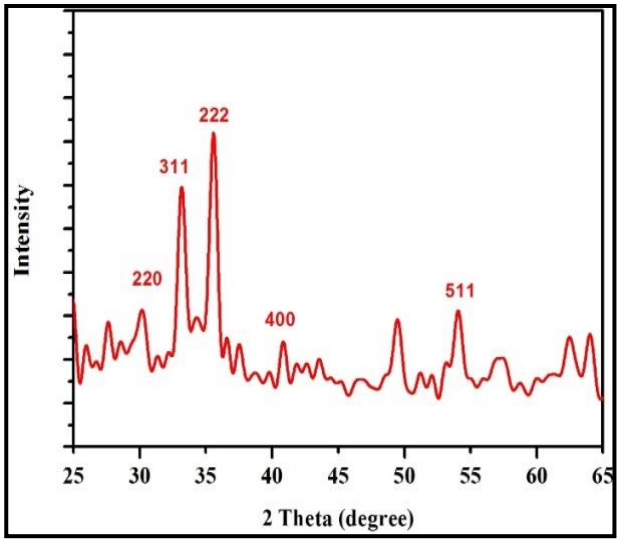

Figure 2: *XRD pattern of Cd-ferrite.*

Based upon Bragg's equation the condition for diffraction maxima is obtained if-

$$2d. \text{Sin}\,\theta = n\,\lambda \qquad (1)$$

where 'd' is interplanar spacing, 'θ' is the angle of diffraction, 'λ' is the wavelength of monochromatic x-rays and 'n' is the order of reflection (i.e. n = 1). The peaks position and relative intensity matches with the standard cadmium ferrite {$CdFe_2O_4$ (JCPDC card 00-022-1063)}. This shows that synthesized sample belongs to space group *Fd3m*cubic spinel structure with lattice constant 8.7561A^0. For cubic spinel structure the interplanar distance 'd', the lattice constant 'a' and the Miller indices (hkl) of reflecting planes are related by the equation.

$$d = \frac{a}{(h^2 + k^2 + l^2)^{1/2}} \qquad (2)$$

The calculated value of 'a', 'd', and 'hkl' are tabulated in Table 1. The observed and calculated d values are in good agreement with for all the samples. Crystallite size (D) is a measure of the size of a coherently diffracting domain. The averaged grain size estimation can also be estimated by measuring the peak width at half length of full maxima. The average crystallite size

for the different compositions was calculated by Debye -Scherrer's formula [16].

$$D = \frac{0.9 \cdot \lambda}{\beta . \cos\theta} \qquad (3)$$

Table1: *Miller indices and lattice parameter* a

Planes (hkl)	Angle (2Θ)	d cal (A^0)	Lattice constant (A^0)
220	30	2.9789	8.4256
311	33.19	2.6995	8.9533
222	35.53	2.5269	8.7535
400	40.85	2.2093	8.8372
511	54.09	1.6957	8.8109
Average lattice constant a=8.7561 A^0			

A dislocation is a crystallographic defect (irregularity) within a crystal structure, which strongly influences the properties of materials. The dislocation density (ρ_D) is a measure of the number of dislocations in a unit volume of a crystalline material. Dislocations are one-dimensional crystalline defects marking the boundary between a slipped and an unslipped region of a material [22]. This defect distorts the regular atomic array of a perfect crystal. The amount of the defects in the as deposited film was resolved by evaluating the dislocation density [21, 22] and the ferrite phase x-ray density ($\Delta x_{Ferrite}$) is calculated by the following relation calculated by-

$$\text{Dislocation density } (\rho_D) = 1/D^2 \qquad (4)$$

$$\text{micro-strain } (\psi) = \beta\cos\theta/4 \qquad (5)$$

where ρ_D is dislocation density and D is crystallite size.

$$\Delta x_{Ferrite} = \frac{8M}{Na^3} \qquad (6)$$

where 'M' is the molecular weight, 'N' is the Avogadro's number and 'a' is the lattice constant. The calculated value of lattice parameter (a=b=c), cell volume (V), crystallite size (D), Dislocation density (ρ_D) micro strain and x-ray density ($\Delta x_{Ferrite}$) of ferrite samples are tabulated in Table 2.

Table 2: *Crystallite size, Lattice constant, Grain size, Volume, Dislocation density and Strain*

Crystallite size (nm)	From Eqn. (3)	From W-H graph
	137	52.4
Lattice constant (A^o)	8.7561	
Grain Size (μm)	*0.17*	
Volume ($(A^0)^3$)	671.324	
Dislocation density(ρ_D)	5.293X 10^{15}	
Inhomogeneity	0.013	
Strain (ψ) (m^2)	From Eqn. (5)	From W-H graph
	0.0144	0.013

The distance between magnetic ions (hopping length) in A site (Tetrahedral) and B site (Octahedral) were calculated by using the following relations [14] (L_A and L_B)

$$L_A = \frac{a \times \sqrt{3}}{4} \qquad (7)$$

$$L_B = \frac{a \times \sqrt{2}}{4} \qquad (8)$$

where a is lattice constant.

The values of the Hopping length for tetrahedral site (L_A) and octahedral site (L_B) were arranged in Table 3.

Table 3: *FTIR vibration bands, x-ray density, bond length and hopping lengths*

Absorption Bands (Cm^{-1})	υ_1	υ_2
	565.1	425.7
Ionic radii (A^o)	r_A	r_B
	0.0144	0.013
Bond length (A^o)	A-O	B-O
	1.906	2.026
Hopping length (A^o)	L_A	L_B
	3.791	3.095
X-ray Density (gm/cm^3) (ϱ_x)	5.64	

Non-zero slope of the Williamson–Hall plots (Figure 3) are indicative [14] of inhomogeneous (i.e. strained) growth of the unit cell.

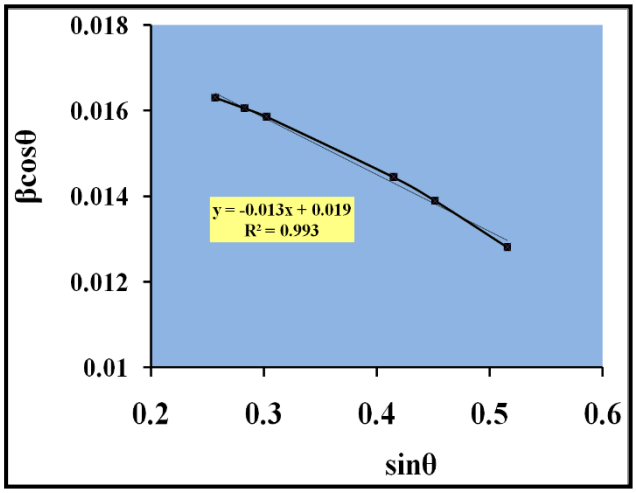

Figure 3: *Plot of βcosθ versus sinθ of ferrite sample*

The degree of such inhomogeneity is characterized in the last column of Table 2. The lattice strain also was calculated using Williamson - Hall equation

$$\frac{\beta \cos\theta}{\lambda} = \frac{1}{D} + \frac{\varepsilon \sin\theta}{\lambda} \qquad (9)$$

where ε is Lattice micro strain, D is average crystallite size, λ is the wavelength of X – Ray used, β is the Full Width Half Maximum and θ is the Bragg's angle. The above equation is in the format y= mx + c where m = η and c = 1/D and it is clear that by plotting the graph of βcosθ versus sinθ as gives the slope as lattice micro strain (ε) and intercept as 1/D. The graph of βcosθ versus sinθ obtained as shown in Figure3, and lattice strain ε value. From the equation (3) the plot of βcosθ versus sinθ will be in the form of straight line equation giving the slope as lattice strain and also from the intercept we can calculate the effective crystallite size which is in good agreement with the value obtained from the equation (3).

3.2 SEM Analysis

The grain size was calculated by linear intercept method [23]. The average grain diameter (G_a) was calculated by enumerating the number of grain boundaries intercepted by a measured length of a random straight line drawn on micrographs.

$$Ga = 1.5\frac{L}{MN} \qquad (10)$$

The scanning electron microscopy studies were undertaken for the samples images are shown in Figure 4 below.

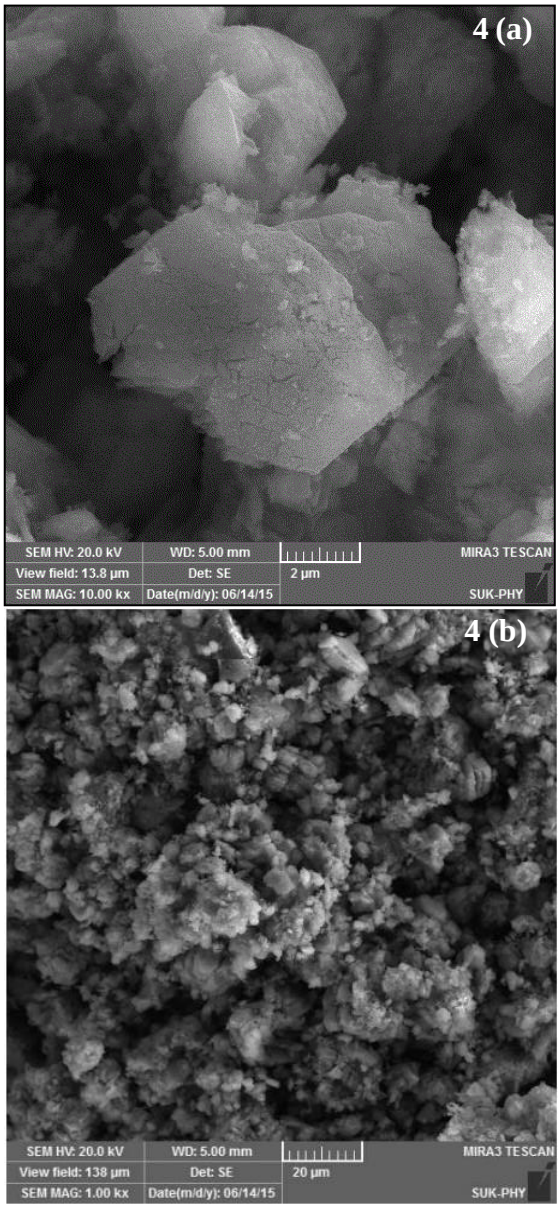

Figure 4: *SEM images of Cd-ferrite. (a) 2μm (10KX magnified) (b) 20μm*

The average grain size is about 0.12 μm with some agglomeration. It is evident from the SEM micrographs that; sample seems to be non-uniform with somewhat agglomeration in the

synthesized samples which is unavoidable with size less than 1μm. Agglomerated particle powder with inhomogeneous broader grain size distribution are Shown in Figure 4.

3.3 FTIR Studies

Infrared spectroscopy was used to determine the local symmetry of the solids and to study the ordering phenomenon in the ferrite samples and FTIR spectra is shown in Figure 5.

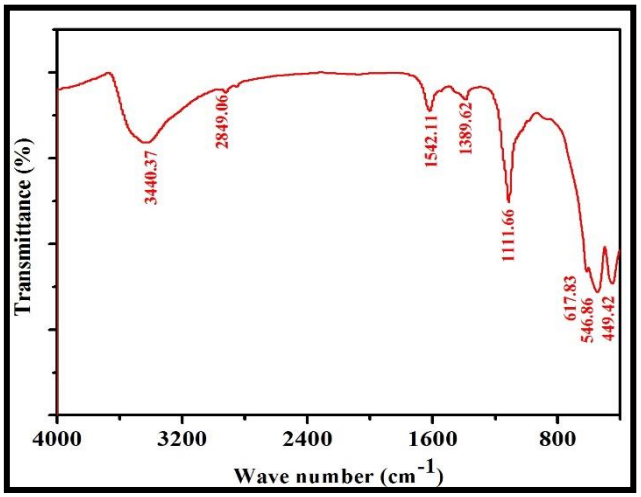

Figure 5: *FTIR spectra of Cd-Ferrite*

IR absorption bands mainly appear due to the vibrations of the oxygen ions with cations at various frequencies, but the spinel structure exhibits two IR vibration bands atν_1=546.86 cm^{-1} and atν_2= 449.42cm^{-1} correspond to the familiar intrinsic vibrations of tetrahedral complex site and octahedral sites. The value of$\nu_1$is more prominent than that ofν_2, indicating that the normal mode of vibration of the tetrahedral complex is higher than that of the corresponding octahedral site. This may be due to the shorter bond length of the tetrahedral site than that of the octahedral site. The$\nu_1$band is generated by stretching vibrations of the Fe^{+3} - O^{-2} bond in the tetrahedral complex and the $\nu_2$band is due to stretching vibrations of the Fe^{+3} -O^{-2} bond in the octahedral complex. Strong bands must be assigned to the vibration of the coordinated groups containing the highest valency cation [16,24]. The bands at 1542.11cm^{-1} could be attributed to the adsorbed water (or humidity) and around 3440 cm^{-1} due a small amount of hydroxyl ions (OH-), probably as a result of the humid atmosphere during the growth process [25-26]

4. Conclusions

The nano-crystalline $CdFe_2O_4$ successfully synthesized by Oxalate Co-Precipitation Method. The X-ray diffraction results for the samples of $CdFe_2O_4$ showed the formation of single phase cubic spinel structure with, having lattice constant and particle size 8.7561A^0 and 135nm respectively. spinel structure exhibits two IR vibration bands atν_1=546.86 cm^{-1} and atν_2= 449.42cm^{-1} correspond to the familiar intrinsic vibrations of tetrahedral complex site and octahedral sites. We have also discussed morphology, dislocation density (ρ_D), mechanical properties (strain), Hopping length {tetrahedral site (L_A) and octahedral site (L_B)} of Mg-ferrite also reported.

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