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Structural, Optical and Magnetic Studies of $CdTiCoFe_2O_4$ Nanoparticles by Sol-Gel Method

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ABSTRACT

Promising future applications of ferrite nanoparticles in medicine, making many devices like permanent magnets, memory storage devices etc. Ferrite nanoparticles have been the emerging focus of the recent scientific research. In the present investigation Cadmium Titanium Cobalt ferrites ($CdTiCoFe_2O_4$) is synthesized by Sol-Gel method. This approach is simple, faster, eco-friendly, cost effective and suitable for large scale production. These synthesized ferrites are characterized by X-ray diffraction (XRD), FTIR, UV–VIS spectroscopic techniques and Scanning Electron Microscopy (SEM). The crystalline nature and the structure of the synthesized nanoferrites are confirmed from X-Ray diffraction analysis. The ferrite powders showed XRD line broadening peaks and the average particle size of the materials is calculated as 8.7027 nm using Scherer formula. The strain (ε) and dislocation density (δ) of the materials are also calculated from XRD data. The optical band energy (Eg) at the edge of absorption band has been determined by the Tauc relation using UV-VIS spectroscopic data. The magnetic properties are studied using Vibrating Sample Magnetometer at room temperature and it is found that this study shows a ferromagnetic behavior of the synthesized ferrites. This significant property allows this type of ferrites can be used in fabrication of magnetic and energy storage media.

Introduction

The Recent interest in the study of several spinel type ferrites is in terms of the synthesis of their nanoparticles at low temperatures by different techniques, in view of the potential applications of these nanosized magnetic materials in different technological areas, as well as to study the intriguing magnetic properties of the nano-ferrite materials has been increased [1,2]. It is a versatile transition-metal oxide and a useful material in various present and future applications related to catalysis, electronics, photonics, sensing, medicine, and controlled drug release [3]. Wang et al. reported that titania has been extensively studied owing to its physical and chemical properties in photo-catalytic applications for environmental remediation. This method is useful to achieve the fabrication of magnetic nanoferrites at low annealing temperature [4]. In recent years very few studied based on different composition like Zn, Sr, Cd, Ba, Cu, Ti, Co etc have been carried out. So, the aim of this present work is to obtain Cadmium Titanium Cobalt ferrites ($CdTiCoFe_2O_4$) by Sol-Gel method and compare their characteristics [5]. The size and morphology of nano particles and their properties may be controlled by modifying the composition of the nano composites and by thermal treatment conditions [6]. Due to the small size of the nano crystals, an important part of the atoms are located at the surface this is the reason why the sol-gel synthesis method gone on intensive development [7]. Till date the research work on CdTiCo Ferrite is very limited. The sol-gel method is used for synthesis of these nanoferrite materials [8, 9].

In this paper, we present a study on the synthesis of CdTiCo nanocomposites of different composition. The composition, crystal structure, morphology, and size distribution of Cd, Ti, Co Ferrite nanocrystals can be controlled by adjusting the synthesis route and molar ratio of materials in the initial mixtures [10]. The synthesized nano crystals have been characterized by X-RD, SEM, and FT-IR, presented below are the details of investigation.

Experimental Details

Materials

The starting materials used in this work for the preparation of $CdTiCoFe_2O_4$ are Cadmium nitrate (Cd $(No_3)_2$ $4H_2O$), Titanium Oxide (Ti O_2), Cobalt nitrate (Co $(NO_3)_3$ $6H_2O$), Ferric nitrate (Fe $(NO_3)_3.9H_2O$), Ammonia.

Synthesis of $CdTiCoFe_2O_4$

The analytical grade Fe $(NO)_3 \cdot 9H_2O$, Cadmium nitrate Cd $(NO)_3 \cdot 4H_2O$, Titanium Oxide (Ti O_2), Cobalt nitrate (Co $(NO_3)_3$ $6H_2O$), liquid Ammonia were used as raw materials. The desired stoichiometry amounts of substance are first dissolved in 100 ml of distilled water by continuous stirring for 3 hours using magnetic stirrer to form a clear solution. The molar ratio of nanoferrites is 1: 2. Then liquid Ammonia is added drop wise to control the agglomeration of nanoparticles to the resulting clear solution, and then the stirring is continued for two hours. When these capping agents are added it hooks on to the metal nano particles of certain size, they reach solubility limit, as the net charge on the metal particle is now controlled by the hooked up capping agent. The solution was evaporated by intensive stirring and heating for 2 hours at heated to 80 °C and kept at this temperature until the sol turned into a transparent gel. The gel was then heated at the temperature 80°C for 24 hours, so that auto-combustion would takes place. The powder is crushed in an agate mortar to obtain the nanoferrites. This nanoferrites sample is annealed at 400°C for 2 hours. Finally Cadmium Titanium Cobalt Ferrite nanoparticles were obtained

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Results and Discussion:

The synthesized ferrite is subjected to following characterization, so that their properties can be discussed effectively.

X-Ray Diffraction Analysis

Careful analyses of the XRD patterns help to determine different parameters like crystalline nature, structure, size, strain, dislocation density etc. Figure 1 shows the XRD pattern of the $CdTiCoFe_2O_4$ powders. XRD pattern clearly indicates the crystalline nature and the presence of single-phase spinel structure. The well resolved peaks clearly signify the crystalline nature and the single phase of the sample.

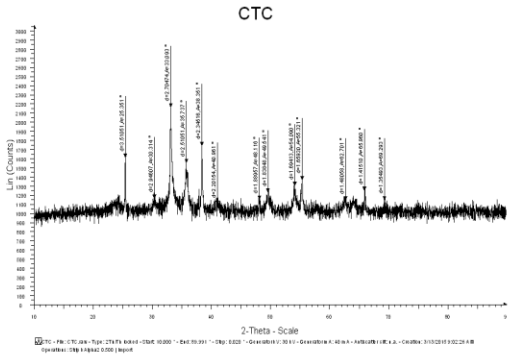

Figure 1. XRD patterns of the $CdTiCoFe_2O_4$ powder

The lattice parameter values of ferrite reveal that this ferrite posses tetragonal crystal structure. The values are a= *6.987888* A^0 *b= 6.987888* A^0 *c= 6.308339* A^0. The average particle size of crystal is evaluated from XRD, by using the Debye Scherer's formula [11],

$$D = K\lambda/\beta \cos\theta$$

The XRD pattern exhibits wide reflections, which indicates the narrow size nature of the synthesized crystallites. Moreover, from the graph as the base width of the 100% peak is broadened which indicates that the particle size is very less. The mean crystallite size of the sample is found to be 8.7072 nm. The dislocation density $\delta = 1.3204 \times 10^{-16} m^{-2}$ is calculated using crystallite size [12]. The strain is found to be $0.19921 \times 10^{-3} m^3$ from the figure 2 [13].

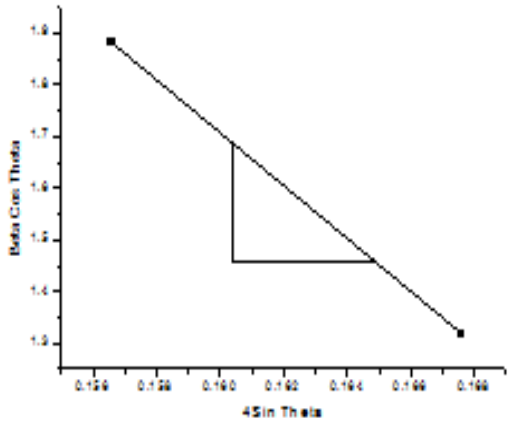

Figure 2. $\beta\cos\theta$ vs $4\sin\theta$ Strain plot form XRD

Fourier Transform Infra-Red Analysis

The FTIR (Fourier Transform Infrared Spectrometer) spectrograph of the synthesized material is as shown in figure 3. The spectrogram reveals the fact that bond formation and two main metals – oxygen bonds at 572.86 cm^{-1} which conform that the synthesized material is ferrite.

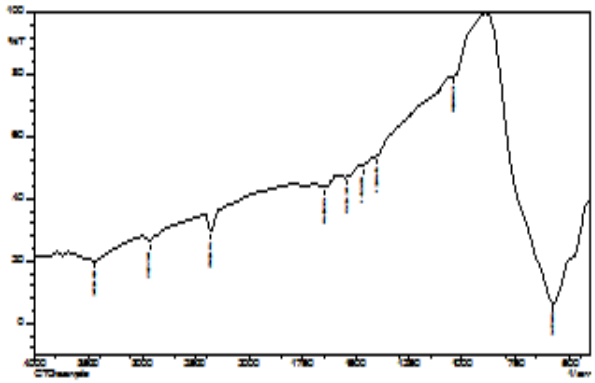

Figure 3. FTIR analysis $CdTiCoFe_2O_4$ nano ferrite

Probably, the absorption bands at 3439.08 cm^{-1} can be attributed to the N-H and O-H stretching vibrations of the residual by-product, respectively. The overlapping of the O-H vibration band of the H_2O molecule with these frequencies can lead to a broadening of these bands. The peaks that appeared at 1465.90 and 1535.34 cm^{-1} are assigned to N-H and N-O stretching vibrations of the Primary amines and Nitro compounds groups respectively. The peaks that appeared at 1606 and 1396.46 cm^{-1} are assigned to C-H and C-C stretching vibrations of the carbonyl groups, respectively.

UV-Visible Spectral Study

Optical analysis of $CdTiCoFe_2O_4$ sample was performed using UV-Visible spectroscopy. This characterization technique is generally used for determine the cut-off wavelength and band gap determination. UV-visible spectroscopical analysis was performed at room temperature in the wavelength region 200 nm to 1200 nm is shown in the figure 4. The cut-off wave length starts form 241.98nm. This indicates that synthesized ferrites can be used for any optoelectronic applications.

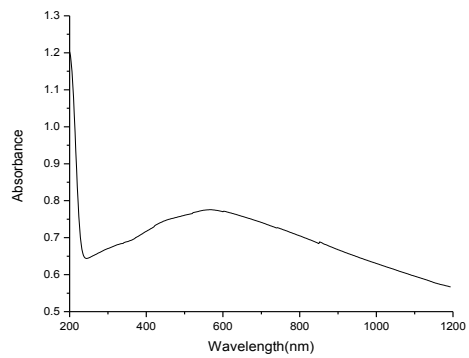

Figure 4. Absorbance spectra of $CdTiCoFe_2O_4$

The optical band gap of the nanopowders was determined by applying the Tauc relation as given below

$$\alpha h\nu = B(h\nu - Eg)^n$$

Where α is the absorption coefficient (α = 2.303A/t, here A is the absorbance and '*t* ' is the thickness of the cuvett), *B* is a constant, *h* is Planck's constant, *ν* is the photon frequency, and *Eg* is the optical band gap. For indirect optical band gap, n=1/2, and the cure $(\alpha E)^{1/2}$. E tends asymptotically towards a linear section other values of n (generally 2) suggest direct band gap [14]. An extrapolation of the linear region of a plot of $(\alpha h\nu)^2$ on the Y- axis versus photon energy ($h\nu$) on the *X*-axis gives the value of the optical band gap (*Eg*) as shown in Figure 5 [15,16]. From the figure 5 it is observed that the value of *Eg* of $CdTiCoFe_2O_4$ is ~5.10 eV. According to quantum confinement theory, the band gap of a semiconductor depends on the crystal size, and its value will increase as the crystal size decreases.

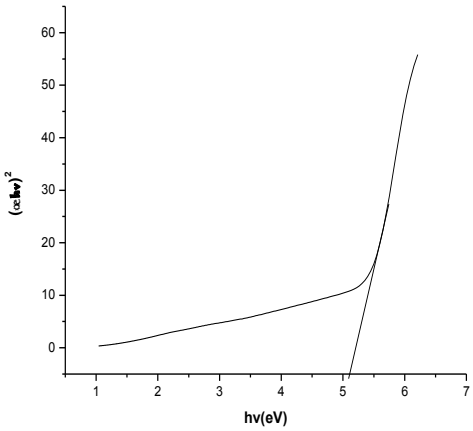

Figure 5. Plot of $(\alpha h\upsilon)^2$ vs. $h\upsilon$ for $CdTiCoFe_2O_4$

Vibrating Sample Magnetometer

The magnetic properties are studied using Vibrating Sample Magnetometer at room temperature. The magnetization curve of the $CdTiCoFe_2O_4$ drawn at room temperature is shown in figure 6.The sample exhibits an excellent magnetic property with weak ferromagnetic property [17], which may attributed to the charge transfer between capping agent and $CdTiCoFe_2O_4$ nanoparticles. The coercivity and retentivity of $CdTiCoFe_2O_4$ are 894.26 G and 27.529E-3 emu/g respectively. The saturation magnetization (*Ms*) and mass of samples are found to be 0.27826 emu/g and $45.400E^{-3}$ g respectively. The ferromagnetic behavior is learnt from the hysteresis with weak magnetic moment, which may be due to the long range ferromagnetic ordering in $CdTiCoFe_2O_4$ nanoparticles This significant property allows this type of ferrites can be used in fabrication of magnetic storage media and also Ferrites, typically spinel ferrite and magnetoplumbite ferrite, can be used as recording materials, microwave devices, humidity sensors, pigments etc [18].

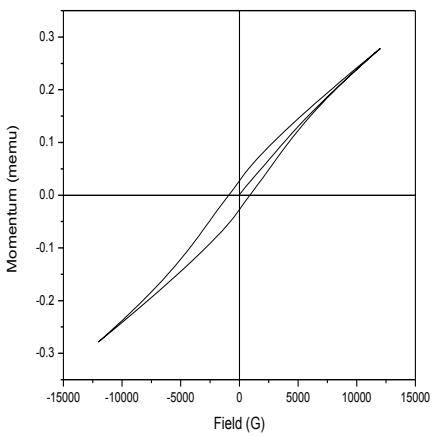

Figure 6. VSM Study for $CdTiCoFe_2O_4$nanoferrites

Surface Morphology

The surface morphology and the nano phase of the synthesized material are studied from SEM micrograph it shows uniform surface morphology and good quality of nanoferrites, which shown in the Figure 7. It shows the microstructure of the sintered specimen with the poly dispersed nature of particles and their agglomeration. The $CdTiCoFe_2O_4$ ferrites shows a bi-phasic microstructure constituted of dark ferrite matrix grains and small whitish grain at the grain junction/boundary [19, 20]

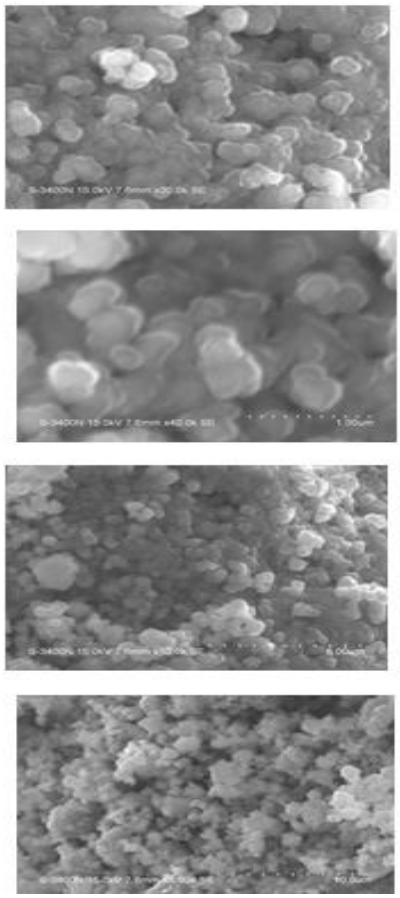

Figure 7. SEM images of $CdTiCoFe_2O_4$ for as 5 µm, 0.1 µm , 0.2 µm & 10 µm range

Conclusion

In conclusion, $CdTiCoFe_2O_4$ nanoparticles have been prepared by simple chemical route Sol-Gel method. The XRD results shows that the nanoparticles are crystalline in nature with tetragonal structure. The crystallite size of $CdTiCoFe_2O_4$ nanoparticles was found to be 8.7027 nm. The strain and dislocation density were found to be 0.19921 X $10^{-3}m^3$ & δ=1.3204X$10^{-16}m^{-2}$ respectively. The band gap of $CdTiCoFe_2O_4$ nanoparticles was found to be 5.10 eV. The surface morphology was studied from Scanning Electron Microscopy. The synthesized nanoferrites belong to the group of soft ferrites which is confirmed from VSM data. The coercivity value from the hysteresis shows ferromagnetic behavior of the synthesized nanoferrites. The increased retentivity allows that they can be used in magnetic storage media.

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