Synthesis and Electric behavior of Cd-doped nano-size spinel ferrites

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Abstract—In recent years, there has been considerable attention in the study of the properties of nano sized ferrite particles because of their significance in the fundamental understanding of the physical processes as well as their upcoming applications for many technological purposes. Spinel ferrite magnetic materials are an vital class of metal oxides containing ferric ions and having the general structural formula AB₂O₄. Spinel ferrites possess exclusive physicochemical properties including outstanding magnetic characteristics, high specific surface area, surface active sites, high chemical stability, tunable shape and size, and the ease with which they can be modified or functionalized. This report presents the synthesis of cadmium ferrite (CdFe₂O₄) by Sol-gel micro-oven method. Analysis confirmed the formation of monophasic CdFe₂O₄ powder with an average particle size in nanometer after synthesis at 800°C with urea, as a reducing agent. The cadmium ferrites characterized by using X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM), Electrical conductivity. The XRD of synthesized particles reveals the formation of single-phase CdFe₂O₄ nanoparticles. TEM confirms the formation of particle size in the range of 25–35 nm. SEM analysis also supported the desired morphological nature of the synthesized ferrites.

Keywords— Nanoferrite, Sol-gel method, XRD, SEM, TEM, Etc.

I. INTRODUCTION

As tremendous technological progress in electronics has inevitably been accompanied by the development of computing and transporting systems as well as non-volatile data storage technologies, miniaturization of the electronic devices has become critical, thus leading to the reduction in size of the magnetic materials to dimensions comparable to those of the atoms and molecules. Due to their reduced size, these nanoparticles may possess novel and improved properties in comparison to the bulk materials, which have been extensively used in electronic devices for high-frequency telecommunications [1]. the design and synthesis of nanomagnetic particles has the focus of the intense fundamental and applied research with special emphasis on their enhanced properties that are different from those of their bulk counterparts [2–7]. Also, cadmium ferrites (in bulk) having normal spinel structure where Cd²⁺ and Fe³⁺ ions occupying tetrahedral (A) sites and octahedral (B) sites respectively, have converged the insight of many researchers when formed in nano-regime due to their changing spinel structure from normal to partial inverse/mixed structure. Due to this change in their cationic distribution, they find extensive applications particularly in information storage devices, magnetic bulk cores, magnetic fluids, micro-wave absorbers and as catalysts [8]. Moreover, they have also conceived perception in being an important complex oxide in gas-sensing materials [9] as well as contrasting agents used in magnetic resonance imaging (MRI) [10] with enhanced magnetization when present in nano-regime. Different methods have been used to synthesize CdFe₂O₄ nanomaterials; high energy ball milling [11], co-precipitation [12-14] and sol-gel combustion [15, 16]. Sol-gel combustion is a cost-effective and simple process with a high production rate [17]. A chemical sol-gel and combustion process is combined involving exothermic and self-sustaining redox reaction between metal nitrates (oxidizer) and an organic complexant (reducing agent) [18].

In this work, we investigate the role of cadmium Cd¹² substitution at tetrahedral A- Side and there effect on structural and electrical properties of CdFe₂O₄ prepared by sol-gel micro-oven method obtain nano sized particles were undertaken.

II. EXPERIMENT

Nanoparticles of cadmium ferrites were prepared by sol-gel micro-oven method using analytical grade chemical. High purity cadmium nitrate hexahydrate [Cd(NO₃)₂.6H₂O], Ferric Nitrate[Fe(NO₃)₃.9H₂O] and urea were taken as a starting material to prepared
cadmium nanoparticles. Each material was weighed separately in stoichiometric ratio and dissolve in 30 ml de-
ionized water to obtain solution. Molar ratios between metal nitrates and the reducing agent were calculated by taking in account of oxidizing and reducing valences of various elements. In the next step, The cationic solution were mixed thoroughly using stirrer for complete dissolution and slowly evaporated at 80°C to form a viscous gel. After formation of viscous gel, it will be ready to fired nearly 1500°C in micro-oven. To obtaining ferrite powder ash using Samsung TDS micro-oven model operate on 800 Watt for 3 minute. The ferrite ash grounds in pestle mortar for 4 hour are necessary. The ferrite powders were pressed under a pressure of 20 MPa into pellets of uniform diameter of 1.5 cm and a varying thickness of 2 mm to 3 mm. The pellets were heat treated at 800°C in air for 4 hour and were again ground into fine powder in an agate mortar to obtained monophasic CdFe₂O₄ spinel ferries powder. The powders were characterized by XRD, FE-SEM.

III. RESULTS AND DISCUSSION


The precipitated fine particles were characterized by XRD as shown in “Fig. 1”. A Bruker AXS D8 Advance X-ray powder diffractometer was employed to obtain the X-ray diffraction patterns of the samples using CuKα (1.5406 Å) radiation. The structure and their crystallite size were evaluated. The crystallite size of the nanocrystalline samples was measured using Debye-Scherer formula [19],

\[ D_{XRD} = \frac{0.98\lambda}{\beta \cos \theta} \]  

(1)

![XRD pattern of CdFe₂O₄](image)

*Figure 1. XRD pattern of CdFe₂O₄*

Where \( \lambda \) is the wavelength of X-ray used in Å, \( \beta \) is the full width at half-maximum (FWHM) in radians in the 20 scale, \( \theta \) is the Bragg angle, \( D_{XRD} \) is the crystallite size in nm. The diffraction pattern of sample and experimental data shows that the sample having cubic spinel structure. The absence of additional lines in X-ray pattern confirms the formation of single phase ferrite. The sample calcined at 800 °C exhibited a sharp, intense diffraction peaks and single cubic phase with fcc structure of CdFe₂O₄ without any impurity phase [Ref. JCPDS card no.22-1063(79--1155) and Fd3m (227) space group], revealing the highly crystalline character of the sample. The crystallite size was found to be 23.19 nm with respect to calcination temperature, as 800 °C respectively and the calculated lattice parameters \( a \) for CdFe₂O₄ sample was found to be 0.873 nm, which is in good agreement with the literature value of 0.869 nm for CdFe₂O₄ sample [24,25]. This indicated that impurity free CdFe₂O₄ nanoparticles were obtained at 800 °C. The diffraction peaks observed at 20 = 28.725, 33.85, 41.182, 45.06, 51.089, 54.474, 59.806, 67.832 and 70.697° indicated the planes of (220), (311), (400),(331), (422), (511), (440), (620) and (533) respectively.

X-ray density (\( d_x \)) was calculated using the relation [26]:

\[ d_x = \frac{8M}{N a^3} \]  

(2)

Where, \( M \) is a molecular mass and \( N \) is Avogadro’s number (\( N = 6.02 \times 10^{23} \)). The X-ray density (\( d_x \)) of cadmium ferrites was found to be 5.735 g/cm³. From X-ray density and bulk density values (\( d_b \)) the pore volume distribution (\( P \% \)) was calculated using following relation:

\[ P = (1 - (dB/dX)) \times 100\% \]  

(3)

The value of porosity was found to be 38%. However, the bulk density of the powdered sample is much small than the x-ray density of the system of the composition indicating the porous nature of the synthesized material. All the detected peaks of samples were indexed with CdFe₂O₄ with an normal spinel structure. The diffraction patterns provide clear evidence of the formation of the pure inverse spinel structure of cadmium ferrite.

2. Electrical Properties
The observed low frequency dielectric distribution in ferrites may be due to space charge special effects. Elevated dielectric constant values at lower frequencies were observed for samples which tend to decrease at upper frequencies. This may occur from electronic polarization and interfacial polarization starting from the heterogeneous structure. P.B. Belavi et al. studied structural electrical and magnetic properties cadmium substituted nickel copper ferrites and found same variation in higher frequency [24]. As the frequency is increased, the time necessary for the interfacial charges to be polarized or for the dipoles to arrange is delayed, thus dropping the dielectric permittivity. The discrepancy of dielectric constant with frequency shows the distribution due to Maxwell-wagner [20-22] type of interfacial polarization in accordance with koops phenomenological theory [23]. The higher value of dielectric constant are occurs due to accessibility of space charge polarizations at the grain boundary. Charge carrier transport in spinel ferrites is provided by hopping mechanism between iron cations located in octahedral sites [25].

The variation of $\tan \delta$ with frequency (figure 3) shows a alike nature as the variation of $\varepsilon'$ with frequency and is considered to be caused by domain wall resonance. At upper frequencies, losses are found to be low down since domain wall motion is inhibited and magnetization is forced to vary by rotation.

The variation of dielectric loss tangent of cadmium ferrite sample with shown in fig.3. The value of $\tan \delta$ decrease with growing frequency reaching a constant value for all the composition of upper frequencies. This is obvious because of the facts that only varieties contributing to polarisability are bound to be lagging behind the applied field at higher frequency. According to, Rabinkin & Novikova [16] the polarization mechanism is analogous to the conduction process. The switch of electrons between ferrous ion and ferric ion Fe$^{3+}$ on octahedral site to local displacement of electron in track of the applied field and these electrons establish the polarization.

### IV. CONCLUSION

CdFe$_2$O$_4$ have been fabricated using urea as a reagent by sol-gel method and successfully formation of spinel structure, which is conform by X-ray diffraction analysis density has greater value as compare to bulk density. The polarization decrease with increasing frequency and then reached a constant value due to the fact that beyond a certain frequency of external field the electron hopping cannot follow the alternating field.

### REFERENCES

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