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LARGE SCALE SYNTHESIS OF $CdFe_2O_4$ NANOPARTICLES VIA SOL-GEL METHOD

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Abstract :- *In this work, $CdFe_2O_4$ are synthesized in large scales using simple sol-gel method. The use of Magnetic spinel ferrites in many fields such as ferrite cores, transducers, electromagnetic wave absorbers, magnetic recorders, drug delivery, magnetic fluids, magnetic resonance imaging, gas sensing and photo catalysts has made them more attractive among researchers. $CdFe_2O_4$ samples have been synthesized using Ferric nitrate, cadmium nitrate and citric acid as the starting materials. The samples were collected, dried and calcinated for 1 hour. The effect of calcination temperature has been investigated by varying it as 500°C and 700°C. The calcinated samples were subjected to various characterization techniques such as Scanning electron microscopy, X-ray diffraction, Fourier transform infrared spectroscopy, and Optical absorption spectra, analysis.*

Key words: *$CdFe_2O_4$, Spinel ferrites, Polycrystalline, band gap.*

1. Introduction

The spinels compound AB_2O_4, where A and B are metals. $CdFe_2O_4$ is a magnetic ferrets nanoparticles [1-3]. These materials are very attractive in scientific and technological importance, nanocrystalline spinel ferrites with the common formula MFe_2O_4, M=(Zn, Ni, Mn, Co, Mg, etc…) are the most significant magnetic materials [4]. Nanotechnology is mainly concerned with synthesis of nanoparticles of variable sizes and shapes. $CdFe_2O_4$ is one of the very important ferrite materials that has been considered in many application such as high density magnetic storage media, color imaging, ferro-fluids, high frequency devices, catalysts, gas sensor, magnetic fluids, photo magnetic materials, site-specific, drug delivery, microwave devices [5-7]. Various methods such as citric acid combustion method [8], sol-gel auto combustion method [9], co-precipitation method[10], chemical co-precipitation method, micro emulsion method, high energy ball milling method, have been developed to prepare nanocrystallite ferrite. This method has the advantages of simple preparation, cost effective and gentle chemistry route resulting in ultra fine and homogeneous powder. The phase purity of the

spinel ferrite products synthesized by sol-gel method strongly depends on the reducing agent. All reducing agents differ in their complexation ability, reducing power and the amount of generated gas, urea [$CO(NH_2)_2$], giycine (NH_2CH_2C OOH), and citric acid ($C_6H_8O_7$) are the most commonly used reducing agents for spinel ferrite synthesis. We will describe the influence of different organic reducing agents such as citric acid, glycine, or urea, on the phase purity of $CdFe_2O_4$. The $CdFe_2O_4$ nanoparticles have been prepared by simple sol-gel method. The prepared magnetic materials are characterized using X-ray diffraction (XRD), Fourier transform infrared spectra (FTIR), scanning microscopy (SEM), UV-visible spectroscopy and the properties of $CdFe_2O_4$ nanoparticles have been explored and discussed.

2. Preparation of material ($CdFe_2O_4$)

$CdFe_2O_4$ nanoparticles have been synthesized by sol-gel method. The precursor materials used were Fe $(NO_3)_2$ $9H_2O$, Cd $(NO_3)_2$ $4H_2O$ and citric acid. In the typical synthesis, 0.05m of ferric nitrate and 0.01m of cadmium nitrate were mixed in distilled water and stirred well. To the above mixture 0.2m citric acid was added. This solution was continuously stirred for an 10 hrs and the temperature was maintained at 80^{o}C using a water bath. Finally a gel appears which is heated for 4 hrs at 100^{o}C. The powder thus obtained was milled to a fine powder and was calcinated at 500^{o}C and 700^{o}C for 1hr. The final product is sent for characterization.

2.1 Material Characterization

Scanning electron microscopy (SEM) for the different samples is employed using (TESCAN VEGA3 SBU), the scan is performed at high vacuum mode using accelerating voltage 30kv, working distance 20mm, and magnification 3000 and 4000. The different compact are characterized using X-ray diffraction (XRD),(Rigaku miniflexll diffractometer) where the used X-ray tube is a copper tube operating 15Amp and the used wavelength is kα,with wavelength λ=0.1540nm, the scan is performed over the range of 2θ (10-80) degrees, the identification of the present crystalline phases is done using joint committeenon powder diffraction standards (jcpds) data base card numbers. Fourier transform infrared spectroscopy (FTIR) analysis (Perkin-Elmer), is carried out for the prepared using KBr disc technique, the scan is preformed in the region of 400-4000 Cm^{-1}. The UV-visible absorption spectra analysis (Varian Cary 50 bio) working at the absorption spectra were studied in the region 400-700nm at room temperature to analyze the optical absorption of the grown particles.

3. Results and discussion

3.1 Morphological Analysis

SEM measurements are carried out in order to understand the morphology and shape of the synthesized $CdFe_2O_4$ nanomaterial's. The SEM images show lots of agglomerated however in many cases of nanocrystalline ferrites, the particle are mostly irregular in shape with lots of agglomerations [1, 11]. The particles are very fine and due to agglomeration the particles shapes are not well define.

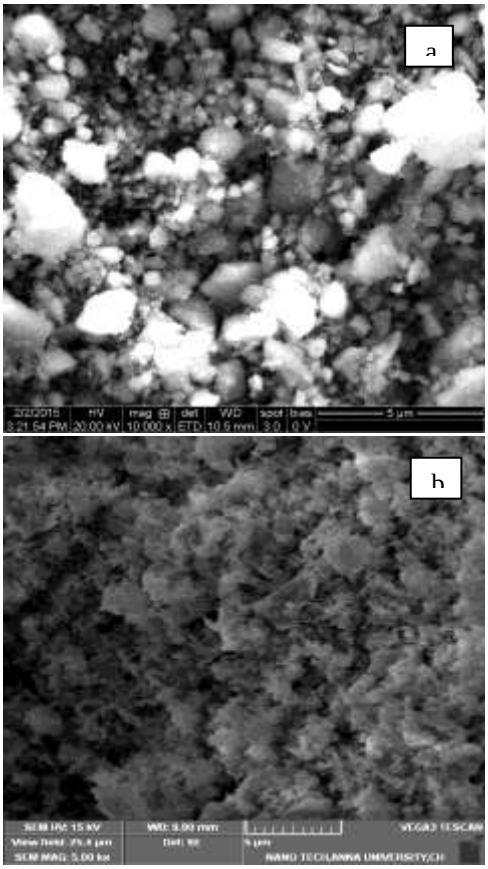

Fig.1 SEM images of the $CdFe_2O_4$ sample calcinated at (a) 500°C and (b) 700°C

3.2 Structural Analysis

The diffraction patterns of $CdFe_2O_4$ powder calcinated at 500 and 700°C for 1 hr has been presented in fig.2 a and b respectively. All of the diffraction peaks can be indexed to the pure cubic spinel $CdFe_2O_4$ and the XRD patterns clearly match with those in JCPDS 65-3115. The broadness of the peaks suggests that the particles formed should be of nanometer size. The $CdFe_2O_4$ calcinated samples show a spinal structure.The crystallite size been calculated using the Scherer equation $D = k\lambda/\beta\cos\theta$, where D is the average size of crystallite, K is 0.9, λ is the wavelength of the X-ray used, β is the full width at half maximum (FWHM) and θ is the angle of diffraction. The average crystallite size of sample calcinated at 500°C and 700°C are 15nm and 19nm respectively. As the calcinated temperature is increased, the crystallite size and the degree of crystalline also increases.

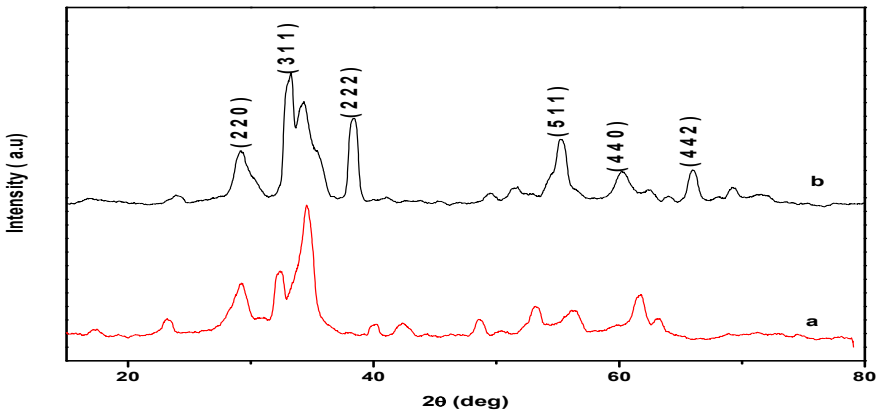

Fig.2 The XRD pattern of Fe_2CdO_4 sample calcinated at (a) 500°C and (b) 700°C.

2 ɵ	ɵ	Cos ɵ	sin ɵ	FWHM (^{o})	FWHM Radian	βCos ɵ	Size	d-spacing
23.322	11.661	0.9793	0.2021	0.337	0.0058	0.0057	24	0.382
29.227	14.612	0.9676	0.2522	0.749	0.0130	0.0125	11	0.305
32.395	16.197	0.9603	0.2789	0.565	0.0098	0.0094	14	0.276
34.563	17.281	0.9548	0.2970	0.814	0.0142	0.0135	10	0.259
40.122	20.061	0.9393	0.3430	0.533	0.0093	0.0092	14	0.224
42.402	21.201	0.9329	0.3616	0.695	0.0121	0.0112	12	0.212
48.452	24.226	0.9120	0.4103	0.455	0.0079	0.0072	19	0.187
53.332	26.666	0.8936	0.4487	0.379	0.0066	0.0059	23	0.171
56.148	28.074	0.8823	0.4706	0.872	0.0152	0.0134	10	0.163
61.764	30.882	0.8582	0.5132	0.729	0.0127	0.0109	12	0.150

3.3 Vibrational analysis

The FTIR spectrum is recorded at room temperature in the wave number region of 4000-400 cm^{-1}. The FTIR spectra of $CdFe_2O_4$ sample calcinated at 500^{o}C KBr. Minal et al. have also obtained similar peak around 3416cm^{-1} for their $CdFe_2O_4$ sample calcinated around 350-650°C.[12] The very small band at 335cm^{-1} is due to adsorbed or atmospheric CO_2.

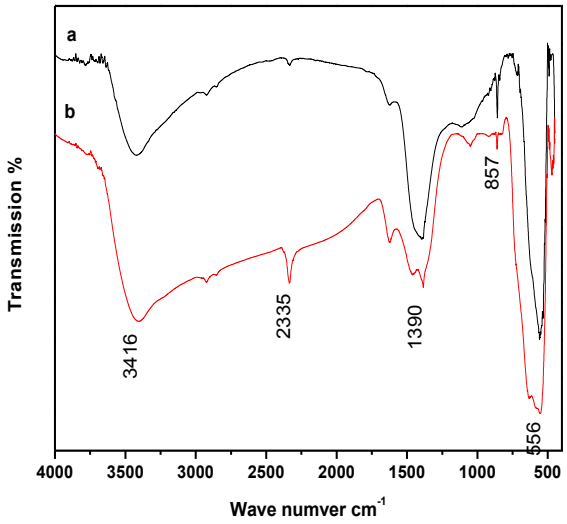

Fig.3 FTIR spectra of the $CdFe_2O_4$ sample calcinated (a) 500^{o}C (b) 700^{o}C

3.4 Optical analysis

The absorption spectra were studied in the region 270-700nm at room temperature to analysis the optical absorption of the grown particles. It can be clearly seen that the prepared nanoparticles show less than 30% absorption. Also it is evident that the sample calcinated at a higher temperature of 700°C shows more absorption when compared with the sample calcinated at 500°C. The probable reason for this may be the breaking up of particles when subjected to higher temperature. Similar results have been reported by

Dhayal Raj et al.[13] where in they have reported that the nanorods tend to break up into smaller spheres when exposed to higher calcinating temperature.

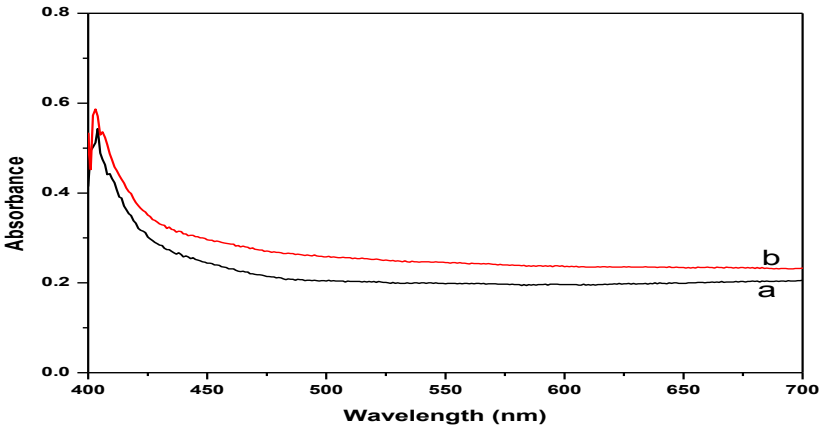

Fig.4The UV-Vis spectra of the $CdFe_2O_4$ samples calcinated at (a) 500°C and (b) 700°C

This breaking up of particles due to higher temperature may have resulted in increased surface to volume ratio. This must be the reason for the more absorption exhibited by the sample calcinated at 700°C.

4. Conclusions

The $CdFe_2O_4$ nanoparticles were prepared by sol-gel method and the effect of calcination temperature on the properties of the prepared nanoparticles has been analyzed. The XRD patterns reveal the polycrystalline nature of the sample. The optical absorption patterns suggest the breaking up of particles on exposure to high temperature and this result is being supported by the SEM results. The prepared nanoparticles can be tested for their magnetic properties and in future can be applied for gas sensing and photocatalytic applications.

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