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A facile method to prepare CdO-Mn_3O_4 nanocomposite

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Abstract: *CdO-Mn_3O_4 nanocomposite has been prepared by a simple solvothermal method using a domestic microwave oven. Cadmium acetate, manganese acetate and urea were used as the precursors and ethylene glycol as the solvent. The as-prepared sample was annealed for 1 hour in each case at different temperatures, viz. 100, 200 and 300°C. The as-prepared and annealed samples were characterized by X-ray diffraction and scanning electron microscopic analyses. Results indicate that annealing at 300°C is required to get the sample with high phase purity and homogeneity. The present study indicates that the method adopted can be considered as an economical and scalable one to prepare the proposed nanocomposite with reduced size, phase purity and homogeneity.*

Keywords*: Nanoparticles, Nanocomposites, Solvothermal method, X-ray diffraction, SEM analysis.*

I. Introduction

The preparation of inorganic nanomaterials that possess the desired properties is a great challenging task. The use of multiple components offers a high degree of flexibility for altering and controlling properties and functionalities of nanomaterials. Doped materials and composite nanomaterials, called as hybrid nanomaterials with improved optical, electronic and other properties are needed for many emerging technologies including solar energy conversion, optical devices, sensors, optical imaging and biomedical detection and therapy [1]. That is, the mechanical, thermal, magnetic, electronic, optical, sensing and catalytic properties can be improved by the association of two or more phases. Chen et al [2, 3] have studied $CdCO_3$-In_2O_3 samples with various phase compositions and investigated $CdCO_3$-In_2O_3 sensors along with replacement of $CdCO_3$ by CdO. They found that CdO-In_2O_3 is a better sensing material to formaldehyde. Priya and Mahadevan [4] have grown and characterized multiphased ternary mixed crystals of alkali halides with enhanced dielectric properties.

Transition metal oxide nanoparticles can exhibit enhanced optical, magnetic and electrical properties as compared to their bulk counterparts, rendering such nanoparticles interesting for a variety of applications [5]. Bulk Mn_3O_4 is a p-type semiconductor with a wide direct bandgap of 2.3eV and is the stablest among all the manganese oxides [6, 7]. Bulk CdO is an n-type semiconductor with a direct bandgap of 2.7eV [8]. Both Mn_3O_4 and CdO are used in sensors, electrode materials, batteries, catalysts and capacitors. Several reports are already available on the preparation of CdO and Mn_3O_4 nanaoparticles and nanostructured thin films. Meenakshi Sundar et al [9] have reported the preparation of ZnO-CdO nanocomposites by a simple microwave assisted solvothermal method. In the present study, an attempt has been made to prepare (in equimolar composition) CdO-Mn_3O_4 nanocomposites by the above simple solvothermal method. The results obtained are reported herein.

II. Experimental

CdO and Mn_3O_4 are not isomorphous systems. So it may not be possible for us to get lattice mixing sufficiently for the formation of single phased solid solution using these systems. However, multiphased nanocomposite can be expected to be possible when it is prepared directly from the precursors if they have molecular similarity. Hence, in the present study, the CdO-Mn_3O_4 nanocomposite (in equimolar composition) was prepared directly from the precursors cadmium acetate, manganese acetate and urea.

The chemicals used in the present study are of analytical reagent (AR) grade. Cadmium acetate dihydrate and manganese acetate tetrahydrate were taken in equimolar ratio and then mixed with urea in 1:3 molar ratio and dissolved in ethylene glycol and kept in a domestic (commercial) microwave oven operated with a frequency of 2.45GHz and power of 800W. Microwave irradiation was carried out till the solvent evaporated completely. The colloidal precipitate formed was cooled and washed several times with double distilled water and acetone to remove the water soluble impurities and organic impurities. The sample was then filtered, dried in open atmosphere and collected as the yield. In order to improve the ordering and quantity, the product was annealed at three different temperatures viz. 100, 200 and 300°C for 1 hour in each case. The as-prepared and three annealed samples were characterized by X-ray powder diffraction (XRD) and scanning electron microscopic (SEM) analyses.

X-ray powder diffraction data were collected for all the four samples using a PANalytical powder X-ray diffractometer in the 2θ range of 10-70° with monochromated CuK_α radiation (λ =1.54056Å). The morphology of the samples was examined by using a JEOL SEM (Model: ISM5600LV) scanning electron microscope equipped with energy dispersive X-ray absorption (EDX) spectrometer.

III. Results and discussion

3.1 Crystal structure:

The colour of the as-prepared sample is greenish brown. The intensity of the colour is found to increase with the increase in annealing temperature. Fig. 1 shows the XRD patterns observed. The XRD pattern observed for the as-prepared sample indicates the presence of two phases viz. $CdCO_3$ (JCPDS card no: 42-1342) and Mn_3O_4 (JCPDS card no: 24-734). There is no significant difference observed for the sample annealed at 100°C. The XRD patterns observed for the samples annealed at 200 and 300°C indicate the presence of two phases viz. CdO (JCPDS card no: 5-640) and Mn_3O_4. The XRD analysis shows that there is a phase transformation from $CdCO_3$ to CdO taking place when the as-prepared sample is annealed at about 200°C. The estimated lattice parameters are provided in Table 1. The estimated lattice parameters observed for the individual phases compare well with those available in the literature. The observed peaks in the XRD patterns indicate the crystalline nature and reduced crystallite (particle/grain) size of the samples. The Scherrer formula [10] was used to estimate the average crystallite sizes. The estimated average crystallite sizes are given in Table 1.

3.2 Morphology:

The observed SEM images are shown in Fig. 2. The SEM images show that the constituent particles of the nanomaterials is agglomerated and lead to mostly capsule shaped cluster like patterns. Other than capsule shape is not exhibited by all the samples except the sample annealed at 200°C. The SEM image of sample annealed at 200°C shows nanowires along with capsules. The observed average sizes of the capsules are given in Table 1.

The XRD and SEM analyses indicate that only the phases mentioned above are present in the samples within the limit of the equipments used. Eventhough annealing at 200°C is sufficient to obtain phase purity (as per XRD analysis), annealing at higher temperature (300°C) is required (as per the SEM analysis) to obtain the homogeneity. Also, the average crystallite size and the average capsule size are least for the sample annealed at 300°C.

3.3 Elemental composition:

The EDX measurement has provided the elemental composition (mass %) for the sample annealed at 300°C as 30.54 for oxygen, 16.91 for manganese and 52.55 for cadmium. This indicates that the actual metallic content in this sample is about 0.6 Cd and 1.2 Mn. Also, it indicates a significant amount of oxygen vacancies as normally expected in the case of metallic oxides. Thus, the sample annealed at 300°C is found to be CdO-Mn_3O_4 and a multiphased (two phased) nanocomposite with high purity, reduced size and homogeneity. Also, the method adopted in the present study is found to be an effective, scalable and economical one for preparing the CdO-Mn_3O_4 nanocomposite. In addition, the present method can be adopted to prepare similar two-component nanocomposites.

IV. Figures and Tables

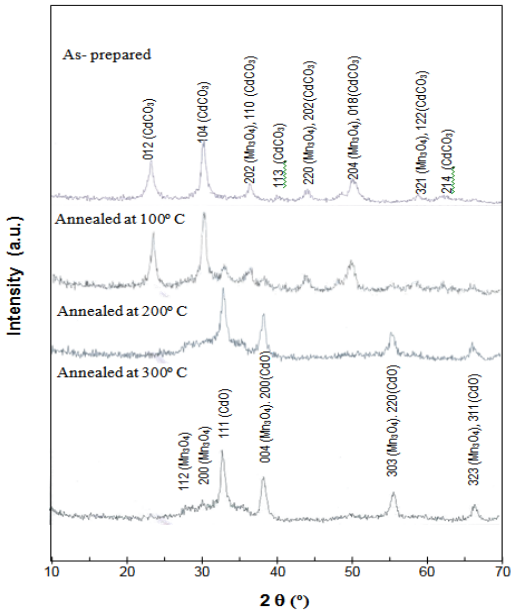

Figure 1: The observed XRD patterns

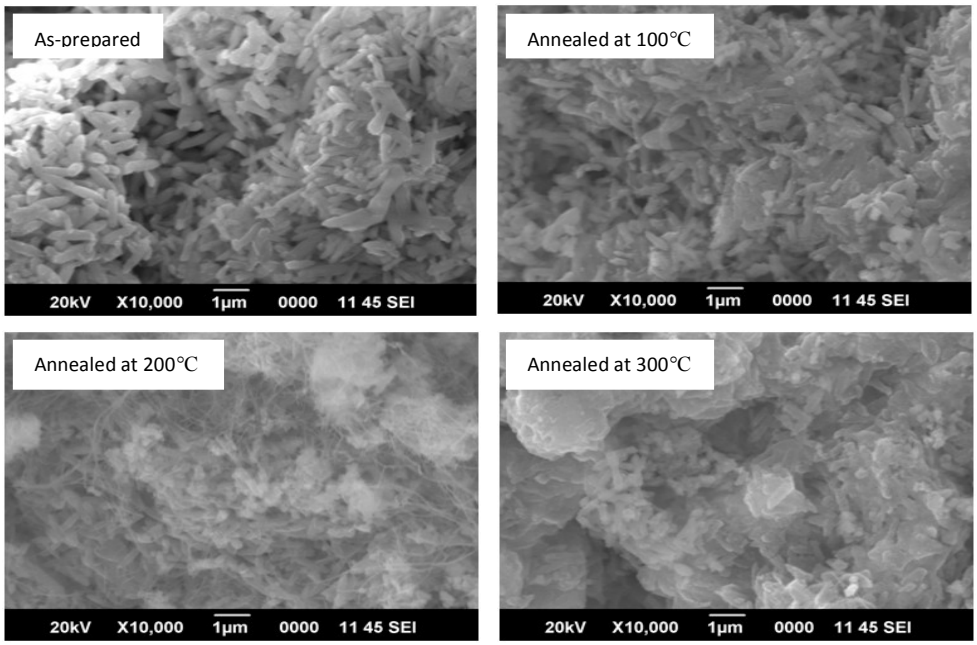

Figure 2: The observed SEM images

Table 1. Results obtained from XRD and SEM analyses. Values given in parentheses are from literature

Sample	Phase	Crystal system	Lattice parameters			Average crystallite size (nm) from XRD	Average capsule size(nm) from SEM	
			a (Å)	c (Å)	Volume (Å^3)		Length	Width
As-prepared	$CdCO_3$	Rhombohedral	4.88 (4.92)	16.33 (16.31)	336.8 (341.9)	9.48	761.6	250.6
	Mn_3O_4	Tetragonal	5.85 (5.76)	9.28 (9.47)	317.6 (314.2)			
Annealed at 100°C	$CdCO_3$	Rhombohedral	4.93	16.30	343.0	10.00	877.3	250.1
	Mn_3O_4	Tetragonal	5.44	9.81	290.3			
Annealed at 200°C	CdO	Cubic	4.69 (4.7)	4.69 (4.7)	103.2 (103.8)	9.12		
	Mn_3O_4	Tetragonal	5.61	9.38	295.2			
Annealed at 300°C	CdO	Cubic	4.69	4.69	103.2	8.15	688.0	228.0
	Mn_3O_4	Tetragonal	5.82	9.25	313.3			

IV. Conclusions

Multiphased (two-phased) CdO-Mn_3O_4 nanocomposite has been successfully prepared by a simple microwave assisted solvothermal method using a domestic microwave oven. Results of XRD and SEM analyses indicate that the as-prepared sample has to be annealed at 300°C to get the nanocomposite with high purity, reduced size and homogeneity. Present study indicates that the method adopted is an effective, scalable and economical one for preparing such two-component nanocomposites.

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