

Investigations on Solid State Synthesized CdO-Mn₃O₄ NanocompositesSagi Rani.C¹ and N.Joseph John²¹Department of Physics, Noorul Islam Centre for Higher Education, Noorul Islam University
Kumaracoil, K.K.Dist, Tamilnadu – 629180, India.²Department of Physics, Government Arts College, Udthagamandalam, Tamilnadu-643002, India***Corresponding Author: E-Mail: sagi_msu@yahoo.co.in****ABSTRACT**

Composite nano particles has gained significant attention due to its uses in a wide range of important applications CdO-Mn₃O₄ nano-composites in its molecular ratio 4:1 were prepared by solid state reaction. The prepared nano-composites were characterized by using PXRD, FTIR, UV, SEM and EDAX, TGA/DTA. The experimental results have confirmed the formation of CdO-Mn₃O₄ nano-composite.

KEYWORDS: nanocomposite, solid state reaction, PXRD, FTIR, TGA/DTA,UV, SEM, EDAX

1. INTRODUCTION

A combination of two or more materials combined on a macroscopic scale to form a useful material is known as a composite material (Shanmugam S, 2010)). Nano particles and nano composites offer unique properties due to their small size, large surface area and the interactions of phase at their interfaces and are attractive for their potential to improve performance of drugs, biomaterials, catalysts and other high value added materials (Kannan, 2011).

Metal oxide nano particles have found its use in many applications owing to its unique physical and chemical properties (Darezereshki E, Bakhtiari, 2013). Composite nano materials have enhanced optical, electrical, thermal and magnetic properties and are used for many emerging technologies like optical devices, sensors, optical imaging, solar cells and other optoelectronic devices (Reza Khayati G, et al., 2013) and (Deepa G and Mahadevan, 2013). Cadmium Oxide (CdO) is a well-known II-VI semiconductor having applications in catalysts, sensors, solar cells and non-linear materials. It is an n-type semiconductor having a direct band gap of 2.5 eV and an indirect band gap of 1.98eV and has enormous optoelectronic properties.

Transition metal oxide nano particles are very attractive due to their variety of applications. Transition metal oxide nano particles can exhibit enhanced optical, magnetic and electrical properties as compared to their bulk counter parts. Mn₃O₄ is a well known among the transition metal oxide for its low toxicity and its natural abundance (Li L, 2012 and (Vijaya Lakshmi, 2014). It is a p-type semiconductor having a direct band gap of 2.3 eV. Both CdO and Mn₃O₄ are used in sensors, electrode materials batteries and capacitors (Moses Ezhil Raj, 2010) and (Zhang W, 2003). In this present work an attempt has been made to prepare CdO-Mn₃O₄ nano composites (in the molecular ratio 4:1) by the method of solid state reaction: A solid state reaction is also called as dry media reaction or a solvent less reaction. It is a chemical reaction in which no solvents are used. Hence it is necessary to heat them to much higher temperatures for the reaction to occur at an appreciable rate. The solid-state method can be used to prepare a whole range of materials like mixed metal oxides, sulfides, nitrides etc (Lian G, 2010).

2. EXPERIMENTAL

Materials Used: The chemicals used in the present study are used as such received without any further purification and is of analytical reagent (AR) grade.

Synthesis of CdO-Mn₃O₄ Nanocomposite: Cadmium acetate dihydrate and manganese acetate tetrahydrate in its molecular ratio 4:1 were mixed thoroughly to a fine mixture using an agate mortar and pestle. This fine mixture is transferred to an alumina crucible and is heated for a period of 24 Hours in a muffle furnace maintained at a temperature of 950⁰C. The resulting sample is the CdO-Mn₃O₄ nanocomposite and the resultant sample thus obtained is collected for further characterizations.

3. RESULTS AND DISCUSSION

The CdO-Mn₃O₄ nano-composite thus obtained is characterized using various techniques like PXRD, FTIR, UV, SEM and EDAX and TGA/DTA.

PXRD Analysis: The powder XRD measurements were carried out by a XPERT-PRO diffractometer with monochromatized Cu K-Alpha radiation [K-Alpha= 1.54443Å]. Fig. 1. shows the XRD pattern for the prepared CdO-Mn₃O₄ nano composite. It indicates the presence of two phases namely CdO (JCPDS card no: 5-640) and Mn₃O₄ (JCPDS card no: 24-734).The XRD pattern has confirmed the formation of CdO-Mn₃O₄ nano composite that exhibits various peaks at 33.04⁰, 38.35⁰, 55.34⁰, 66.0⁰ and 69.29⁰.

The average particle size is estimated using the Debye-Scherrer formula (Cullity BD, 1978)

$$2d = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

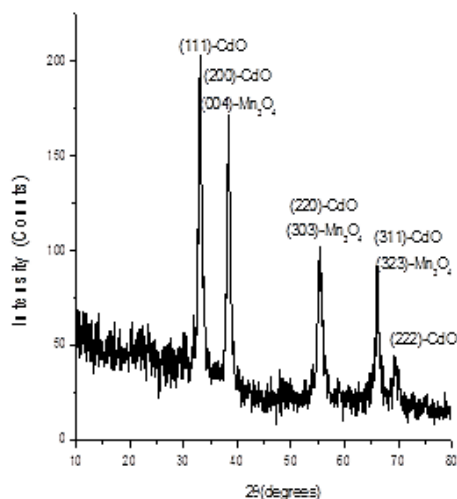


Fig.1.XRD Pattern for the Prepared CdO-Mn₃O₄ Nano-Composite

Where, β is the Full Width at Half Maximum intensity [FWHM] corresponding to the diffraction angle 2θ in radian, and λ is the wavelength of Cu- α radiation. The average particle size for the prepared nano composite is found to be 11.87 nm.

FTIR Analysis: The prepared CdO-Mn₃O₄ nanocomposite was analyzed by means of FTIR spectroscopy from 4000 to 400 cm^{-1} as shown in Fig.2. The broad band at 3440 cm^{-1} refers to bending vibrations of water molecule. The strong peak at 1640 cm^{-1} refers to the bending vibrations of the adsorbed water molecule. Vibration frequencies at 617 cm^{-1} , 518 cm^{-1} and at 419 cm^{-1} were assigned to be the distortion vibration of Mn-O stretching mode, which was in good agreement with MnO material of layered with tunnel structures (Kang L, 2007) and (Rui S, 2012). The weaker vibration bands located at 1415 cm^{-1} , 1316 cm^{-1} , and at 1020 cm^{-1} is found to be the vibration of cadmium molecule.

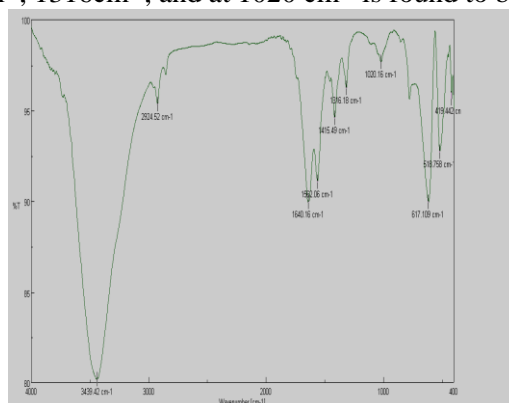


Fig.2.FTIR Spectra for the Prepared CdO-Mn₃O₄ Nano-Composite

SEM with EDAX Analysis: Fig .3. Shows the SEM image of the synthesized CdO-Mn₃O₄ nano composite. It clearly depicts that the particles were in nano dimension with an average particle size of about 12 nm. Some small particles and a few bigger particles were also seen. The bigger sized particles were found to be the overlapping of small particles.

The EDAX measurement has provided the elemental analysis of the sample and is shown in Fig. 4. Here each of the peak is unique to an atom and it corresponds to a single element. The concentration of an element from the peak is identified from the height of the peak in the spectrum. It confirms the presence of cadmium, manganese and oxygen. Thus the EADX spectrum confirms the absence of impurities in the prepared CdO-Mn₃O₄ nano composite (Saravanan R, 2015).

TGA/DTA Analysis: To examine the thermal stability of the prepared nano-composites, Thermal Gravimetric analysis (TGA) and Differential Thermal Analysis (DTA) were carried out between 0^oC and 1200^oC as shown in Fig. 6. In DTA, the temperature difference between a given material and a reference material is measured as a function of temperature by keeping the given and the reference material at a constant heating rate. From Fig. it is revealed that an exothermic DTA peak is recorded at 380^oC. The exothermic peak reaction indicates the formation of CdO and MnO phase through oxidation decomposition process. Moreover an intense endothermic peak is found nearly to 985^oC, and thus it indicates the possibility of phase change in the material (Fu P, 2013).

The TGA analysis reveals that there is no apparent weight loss in between the range 0-100^oC, and thus it clearly indicates the absence of adsorbed/coordinated water molecules. But inbetween 1000^oC and 1200^oC almost

90% of the material gets decomposed. From this it is confirmed that the prepared CdO-Mn₃O₄ nano-composite has high thermal stability (Aldwayyan AS, 2013).

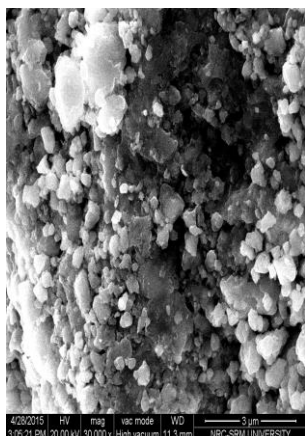


Fig.3.SEM with EDAX Analysis

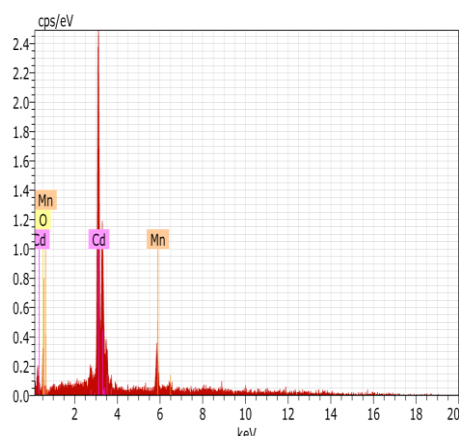


Fig.4.EDAX Spectrum of the Prepared CdO-Mn₃O₄ Nano-Composite

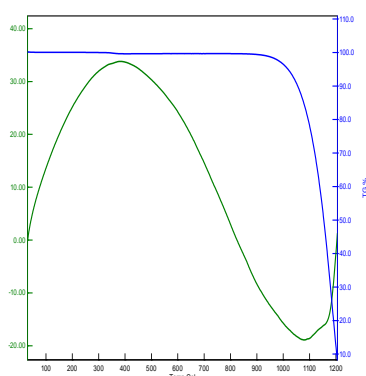


Fig.5.TGA/DTA Curve for the Prepared CdO-Mn₃O₄ Nano Composite

UV Analysis: The optical properties provide information regarding the electronic band structure. Fig: 7 shows the UV-Vis absorption spectra for the prepared CdO-Mn₃O₄ nanocomposite. The optical band gap is determined using the Planck's equation as follows (Viswananth, 2014) and (Radhika Kathyayini, 2014).

$$E_g = \frac{hc}{\lambda} \quad (2)$$

Where, h represents the Planck's constant, c is the velocity of light and λ is the wavelength. Fig. 6 shows a strong absorption peak at 327.17 nm and thus the optical band gap for the prepared nano-composites is found as 3.79 eV.

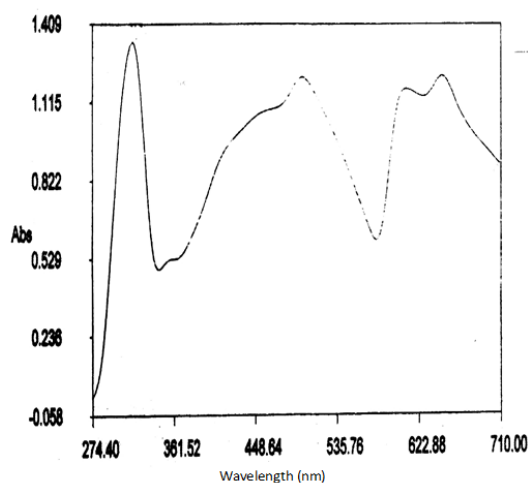


Fig.6.UV-Vis Absorption Spectra for the Prepared CdO-Mn₃O₄ Nano-Composite

4. CONCLUSIONS

In the present study CdO-Mn₃O₄ nano composites were successfully prepared by the method of solid state reaction by using the analytic reagents of Cadmium acetate dihydrate and manganese acetate tetrahydrate in its molecular ratio 4:1. The prepared composites are characterized by adopting various techniques like XRD, FTIR, UV, TGA/DTA and SEM with EDAX. From XRD it was found that the particle size of the prepared composites is found to be 11.87nm. The various spectral vibrations of the sample were analyzed from the FTIR characterization. The SEM/EDAX indicates that a high purity sample can be obtained using this method. The thermal stability of the sample is obtained from TGA/DTA analysis. The optical band gap for the prepared nano-composites is found as 3.79eV. This study also proves that the method adopted is an effective and economical one for preparing such nano-composites.

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