ISSN: 0974-2115
www.jchps.com Journal of Chemical and Pharmaceutical Sciences

XRD, FT-IR and SEM studies of Cr^{3+}doped $CdOZn_3(PO_4)_2$ nanopowder

Y. Naga Bhaskararao, K. Satyavathi, M. Subba Rao, Sandhya Cole*
Department of Physics, Acharya Nagarjuna University, Nagarjuna Nagar, Guntur - 522 510, India
***Corresponding author: E-Mail: sandhya.cole@gmail.com**

ABSTRACT

The Cr^{3+}doped $CdOZn_3(PO_4)_2$ nanopowders is prepared at room temperature by a chemical precipitation method. The prepared powder is characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared (FT-IR) techniques X-ray diffraction pattern exhibits peaks correspond to monoclinic phase of $Zn_3(PO_4)_2$ and cubic phase of CdO, the evaluated average crystallite size of prepared materials were in the range of 30-40 nm. The strain and dislocation density were also calculated from XRD studies. The SEM image shows the stone-like morphology of the nanopowder. The FT-IR spectrum reveals the characteristic vibrations of $CdOZn_3(PO_4)_2$.

KEY WORDS: $CdOZn_3(PO_4)_2$, Nano composites, XRD, SEM and FT-IR.

1. INTRODUCTION

In recent years one-dimensional semiconductor nano composites are of perennial concern due to the peculiar behavior exhibited by them in several optoelectronic nano scale devices and functional materials. $CdOZn_3(PO_4)_2$ nano composites gives a potential application in nano devises from the past decades. CdO is identified as a distinct, n-type semiconductor possessing various properties like low absorbance, low electrical resistivity etc., in the visible region with a direct band gap of about 2.5 eV and an indirect band gap of 1.98 eV respectively.

CdO is identified as a suitable material for a wide range of applications which includes phototransistors, solar cells, transparent electrodes, gas sensors and catalysts. For these applications, porosity, particle size and specific surface area play a major role. Zinc phosphate compound is a non-toxic material with better anti corrosive nature and can be used in several binders. Zinc phosphate is a can be promising material that synthesized either from zinc oxide and phosphoric acid or zinc salt and phosphates. The anticorrosive nature of zinc phosphate depends on the distribution of the particle size over a large degree. The doped semiconducting nano particles were considered as an emerging class of materials.

In this investigation, Chromium was selected as doping due to its specific nature in the lasing action and is primarily used as a component of paints. Its behavior extends to leather tanning in metal surface treatment and also as catalysts in several engineering processes. Chromium is found to exhibits a wide range of allowed oxidation states in which +3 state is more stable energetically whereas +3 and +6 states are generally observed in chromium compounds. The Cr^{3+} state is observed to be reducing with good stable nature. The stable Cr^{3+} state is an important aspect and is related to the high crystalline nature exhibiting stabilization energy from its d^3 electronic configuration. It can provide deep color and bright luminescence in spite of the presence of a low cost activator. Cr^{3+} ions form octahedral complexes and the colors of these complexes depend on the ligands attached to the Cr centre. The colors of several natural and synthetic gemstones such as ruby, emerald, corundum etc., results due to the presence of Cr^{3+} ions. Mainly, Cr^{3+} doped systems are prefered in modern technologies such as display devices, information storage and optical sensors. In nanoscience and nanotechnology, amalgamation forms an essential and prerequisite component.

Recent researchers have synthesized several nano materials with distinct physical methods such as laser ablation, arc discharge, vanishing chemical synthesis etc., by use of complex precursor materials, lengthy investigational procedures; low aspect ratio wires atmosphere controlled heating regimes that are highly complicated. But the nano materials synthesized by chemical methods are found to be efficient providing better control with ease of fabrication in to various sizes, shapes and fictionalization in comparison to the above physical methods.

Metal oxide nanoparticles can also be synthesized by adopting soft chemical methods namely, co-precipitation, sol-gel, Chemical Precipitation, simple mild solution and hydrothermal processes. Among the above methods, the Chemical Precipitation Method was chosen in the present study for the Synthesis of nano powder.

2. MATERIALS AND METHODS

Composite Preparation: Cr^{3+} doped $CdOZn_3(PO_4)_2$ nano powder is prepared by chemical precipitation method. Cadmium oxide (CdO: 99.99%) Zinc phosphate ($Zn_3(PO_4)_2$: 99.99%) and Chromium oxide (Cr^{3+}: 99.99%) are used as starting materials and mixed in the appropriate stoichiometric ratios. They are dissolved in 200 ml of ethanol in a beaker and the solution is stirred vigorously with the help of a magnetic stirrer at room temperature. A 20 gm of sodium hydroxide solution is dissolved in a 100 ml of distilled water in another beaker from which 16 ml of prepared sodium hydroxide solution is added to the above prepared solution under constant stirring drop wise touching the walls of the beaker. The aqueous solution now turns into a white colloid without any precipitation. This stirring process is allowed to continue for four hours and the solution is allowed to settle. The precipitates are collected

through centrifugation, washed three times with distilled water and are dried at 200°c for two hours inside an annealing chamber operated at ambient pressure. Thus the Cr^{3+} doped $CdOZn_3(PO_4)_2$) nano powder are prepared.

3. RESULTS AND DISCUSSION

X-ray diffraction: The XRD patterns of undoped and Cr^{3+}doped $CdOZn_3(PO_4)_2$ nano powders are shown in Fig.1. The annealed Cr^{3+}doped $CdOZn_3(PO_4)_2$ nano powder show quite similar XRD patterns irrespective of Cr^{3+} contents to that of the undoped sample. From the figure, it was confirmed that prepared samples shows crystalline nature. From the XRD data, lattice cell parameters and miller indices of crystal system can be evaluated. The evaluated lattice cell parameters, miller indices with corresponding observed 2θ values and calculated values are in good agreement with JCPDS Card No.: 29-1390 and 05-0640 of $Zn_3(PO_4)_2$, CdO respectively. The average crystallite size of prepared $CdOZn_3(PO_4)_2$ powder sample has been calculated for high intense peak (1 1 1) by using Debye-Scherer's formula.

$$D = (K \lambda / \beta \cos \theta) \quad (1)$$

Where K is a constant (about 0.9), λ is wavelength of X-ray radiation (1.5406 Å), β is full-width at half maximum (FWHM) intensity of the diffraction line and θ is its diffraction angle. Based on the value of FWHM, the average crystallite size is calculated to be 32 nm for undoped sample and 34-36 nm for Cr^{3+}doped sample respectively. Dislocation density is another important property of the material that determines the length of dislocations present per unit volume. Lattice strain and dislocation densities were also estimated by using the following relations.

$$\text{Strain } (\varepsilon) = \beta / 4\tan\theta \quad (2)$$

$$\text{Dislocation density } (\delta) = 15\varepsilon / aD \quad (3)$$

Where "a" is the lattice cell parameter in A°, D is the crystallite size in nm, θ is the diffraction angle in degrees. The calculated values of particle size and lattice strain are 34.96 nm and 34.88 (10^{-4} times of lattice strain value) respectively.

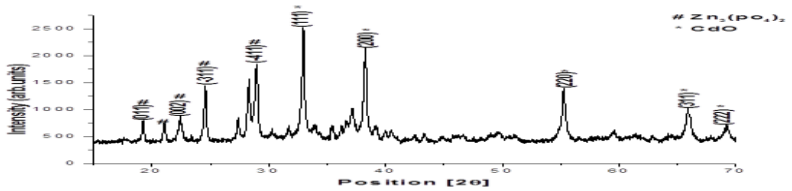

Fig.1.XRD pattern for Cr^{3+} doped $CdOZn_3(PO_4)_2$ nanocomposite

Morphological Studies: SEM and TEM studies are carried out to observe the morphological structures of Cr^{3+} doped $CdOZn_3(PO_4)_2$ nano composites, depicted in Fig.1 and Fig.2 respectively. SEM images reveal the inhomogeneously distributed sphere like patterns with agglomeration and are recorded at high magnifications. While in case of TEM images, rod like structures are observed with spheres coated on the rods appear like core and shell. EDX analysis was also carried out to analyze the chemical elemental composition of the prepared composite samples. The EDX pattern of Cr^{3+} doped $CdOZn_3(PO_4)_2$ nano composites is shown in Fig.3. The EDX spectrum confirms the existence of consisting elements Zn, P, Cd, O and Cr in the prepared nanopowder.

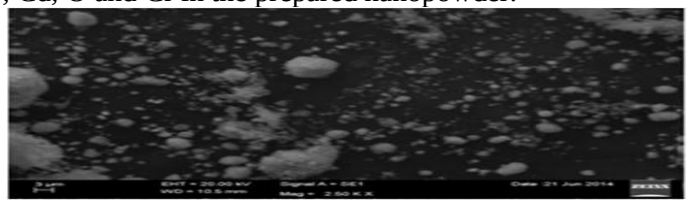

Fig.2.SEM images of Cr^{3+} doped $CdOZn_3(PO_4)_2$ nano composite

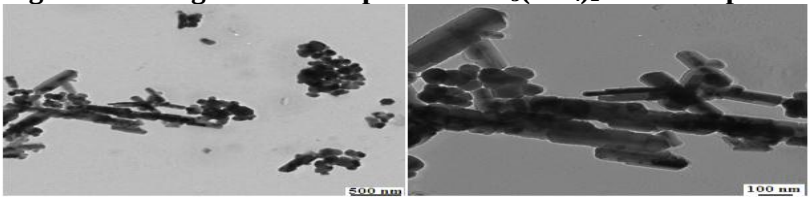

Fig.3.TEM images of Cr^{3+} doped $CdOZn_3(PO_4)_2$ nano composite

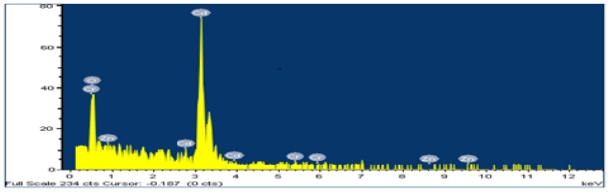

Fig.4.EDX spectrum of Cr^{3+} doped $CdOZn_3(PO_4)_2$nanocomposite

FT-IR Study: Fig. 1.4 depicts the FT-IR spectrum of Cr^{3+} doped $CdOZn_3(PO_4)_2$ nano composites gives characteristic mode of vibrations in the sample. The spectrum exhibits various metal vibrational modes corresponding to organic along with the hydroxyl group. The band observed at 634 cm^{-1} is the characteristic band of Zn-P, which is very weak represents smaller amount of $Zn_3(PO_4)_2$ presents in the sample also confirmed from EDS and XRD. The formation of CdO phase is characterized by an intense and strong IR band at 858 cm^{-1} with shoulders at 720 cm^{-1} also poor resolved band 448 and 505 cm^{-1}. The band observed at 1159 cm^{-1} is assigned to O-H stretching mode of H_2O molecule. Stretching and bending vibrational modes of hydroxyl groups are observed at 3436 and 1632cm^{-1}.

Table.1.Vibrational band assignments of Cr^{3+} doped $CdOZn_3(PO_4)_2$nanocomposite

Vibrational frequency (cm^{-1})	Band Assignment
448, 505 and 720	Cd-O
634	ZnP
858	Cd-OH metallic bond
1159	O-H stretching
2471	O-C-O stretching
1638	OH bending
3329	OH stretching

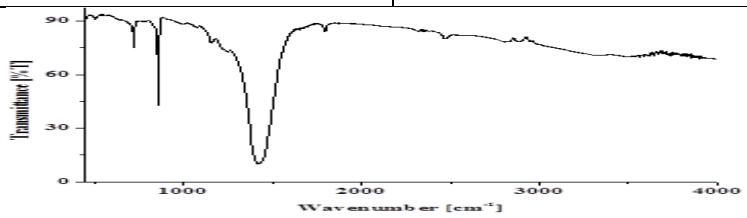

Fig.5.FT-IR spectrum of Cr^{3+} doped $CdOZn_3(PO_4)_2$nanocomposite

4. CONCLUSION

From the synthesis and characterization of Cr^{3+} doped $CdOZn_3(PO_4)_2$ nano composites, the following conclusions are drawn:

- The Particle size, strain and dislocation density were calculated from XRD studies.
- Surface morphology of prepared sample clearly depicts a significant change in the surface.
- TEM images are very clear with rod like patterns.
- FT-IR spectrum of this prepared nano powder has been analyzed in order to recognize the spectral contribution of each component on the structure, confirms the metal vibrations of CdO and $Zn_3(PO_4)_2$ with addition of organic compounds.

REFERENCES

Aswani T, Babu B, Pushpa Manjari V, Joyce Stella R, Thirumala Rao G, RamaKrishna Ch, Ravikumar R.V.S.S.N, Synthesis and spectral characterizations of trivalent ions (Cr^{3+}, Fe^{3+}) doped CdO nanopowder, Spectrochim. Acta, A 121, 2014, 544.

Ferro R, Rodriguez I.A, Influence of F-doping on the transmittance and electron affinity of CdO thin films suitable for solar cells technology, Sol. Energy Mater. Sol. Cells, 64, 2000, 363.

Kuck S, Laser-related spectroscopy of ion-doped crystals for tunable solid-state lasers, Appl. Phys. B, 72, 2001, 515.

Kumar K, Chitkara M, Sandhu I.S, Mehta D, Kumar S, Photo catalytic, optical and magnetic properties of Fe doped ZnO nanoparticles prepared bychemical route, J. Alloys Compd., 588, 2014, 681.

Kuo T, Huang M.H, Gold-catalyzed low-temperature growth of cadmium oxide nanowires by vapor transport, J. Phys. Chem., B 110, 2006, 13717.

Lin C.H, Chen S.Q, Effect of Cu Substituted in Ni-Zn Ferrite by Chemical Co-Precipitation method, J. Master. Sci., 15, 1983, 31.

Moholkar A.V, Agawane G.I, Kyu-Ung Sim, Ye-bin Kwon, Rajpure K.Y, Kim J.H, Influence of deposition temperature on morphological, optical, electrical and opto-electrical properties of highly textured nano-crystalline spray deposited CdO:Ga thin films, Appl. Surf. Sci., 257, 2010, 93.

Ohtake T, Sonoyama N, Sakata T, Electrochemical luminescence of $ZnGa_2O_4$ semiconductor electrodes activated with Cr and Co, Chem. Phys. Lett., 318, 2000, 517.

Pal K, Maiti U.N, Majumder T.P, Debnath S.C, Bennis N, Oton J.M, Synthetic strategy of porous ZnO and CdS nanostructures doped ferroelectric liquid crystal and its optical behavior, J. Mol. Struct., 1035, 2013, 76.

Raj D.S, Jayaprakash R, Prakash T, Kumar S, Neri G, Krishnakumar T, Synthesis of ZnO Nanostructures by Microwave Irradiation Using Albumen as a Template, App. Surf. Sci., 266, 2013, 268.

Ramasamy V, Synthesis and study of optical properties of transition metals doped ZnS nanoparticles, Mater. Sci. Semicond. Proce., 15, 2012, 136.

Rao C.N.R, Govindaraj A, and Vivekchand S.R.C, Ann. Rep. Prog. Chem, Ann. Rep. Prog. Chem. Royal Society of Chem. A, 102, 2006, 20.

Rao C.N.R, Muller A, Cheetham A.K, Nanomaterials Chemistry: Recent Developments, Wiley-VCH, Weinheim, 2007.

Rao C.N.R, Muller A, Cheetham A.K, The Chemistry of Nanomaterials: Synthesis, Properties and Applications, The Chemistry of Nanomaterials, Wiley-VCH, Weinheim, 2004.

Rao K.V, Sunandana C.S, Structure and microstructure of combustion synthesized Mg nanoparticles and nanocrystalline MgO thin films synthesized by solution growth route, Solid State Phys., 50, 2005, 235.

Ristic M, Poporic S, Music S, Formation and properties of Cd(OH) and CdO particles, Mater. Lett., 58, 2004, 2494.

Rout C.S, Kulkarni G.U, and Rao C.N.R, Room-temperature hydrogen and hydrocarbon sensors based on single nanowires of metal oxides, J. Phys. D: Appl. Phys., 40, 2007, 2777.

Schmid G, Clusters and Colloids from theory to applications, VCH, Weinheim, 1994.

Srivastava A.K, Pandey S, Sood K.N, Halder S.K, Kishore R, Novel growth morphologies of nano- and micro-structured cadmium oxide, Mater. Lett., 62, 2008, 727.

Subramanyam T.K, Uthanna S, Srinivasulu Naidu B, Preparation and characterization of CdO films deposited by dc magnetron reactive sputtering, Mater. Lett., 35, 1998, 214.

Tiwary C.S, Sarkar R, Kumbhar P, Mitra A.K, Yellow-orange light emission from Mn^{2+}-doped ZnS nanoparticles, Phys. Lett., A 372, 2008, 5825.

Wenger O.S, Gudel H.U, Optical spectroscopy of $CrCl_6^{3-}$ doped $Cs_2NaScCl_6$: Broad band near-infrared luminescence and Jahn-Teller effect, J. Chem. Phys., 114, 2001, 5832.

Yang P, Chin, Blue-to-Orange Tunable Luminescence from Europium Doped Yttrium–Silicon–Oxide–Nitride Phosphors, J. Lumin., 10, 1989, 230.

Zhang Y.Z, Wang G.L, Solvothermal synthesis of CdO hollow nanostructures from CdO_2 nanoparticles, Mater. Lett., 62, 2008, 673.