

## Synthesis and spectral characterizations of chromium doped cadmium borate nanopowder

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### ABSTRACT

Chromium doped Cadmium borate nanopowder was synthesized at room temperature by chemical precipitation method. Metal borates have excellent mechanical properties with good chemical inertness and high stability under high temperature, lightweight, and low thermal expansion coefficients. The prepared nanopowder is characterized by X-ray diffraction (X-RD), Scanning Electron Microscopy (SEM) with EDX, Electron paramagnetic resonance (EPR), Optical studies, Photoluminescence studies and Fourier transform infrared (FT-IR) techniques. From XRD powder pattern structure and size of the crystallites are evaluated. SEM images show the stone like morphology of nanopowder. Optical absorption spectrum indicates the distorted octahedral site symmetry of Cr<sup>3+</sup> ions. The crystal field Dq and interelectronic repulsion parameters B and C are evaluated. PL spectra indicate clearly that the doping of Cr<sup>3+</sup> in the host matrix lead to significant changes in PL intensity. EPR spectrum gives a resonance broad signal at g=1.97 for Cr<sup>3+</sup> ions. FT-IR spectrum reveals the characteristic vibrations of Cadmium borate. This unique feature of these materials has widespread a technological importance in the development of tunable solid state lasers and new luminescent materials.

**Keywords:** Cadmium borate nanopowder, Chemical synthesis, X-ray diffraction, SEM/EDX Photoluminescence with CIE, EPR and IR.

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### I. INTRODUCTION

Nano particles have attracted great interest recently due to their unique physical and chemical properties, which are different from those of either the bulk materials or single atoms [1]. In the past decade, nano scale research has opened revolutionary opportunities for a wide number of technological applications. Due to their special optical, magnetic, electrical and catalytic properties, and improved physical properties like mechanical hardness, thermal stability or chemical passivity [2]. Nanostructures based on zinc oxide and cadmium oxide are particularly interesting because of their n-type conductivity with a wide band gap (3.3 eV and 2.2 eV respectively) which make these materials more suitable for modern technologies. CdO have promising applications in catalysts [3,4], gas sensors [5,6], solar cells [7,8], paint pigments and so on. Transition metal-doped metal oxide nano crystals have attracted considerable interest in the scientific community due to their unique optical and electro-optical properties[9].

A lot of work has been done on the synthesis of these nanomaterials but a wet chemical method is most suitable and accepted as a promising

technique because of the ability of the production of various size and large quantities of nanoparticles. Most of the study related to CdS and/or ZnS nanomaterials is focused on the optical properties [10]. Borate compounds are well known remarkably for their use in industries and mineralogy. Metal borates have excellent mechanical properties, good chemical inertness and high stability under high temperature, lightweight, and low thermal expansion coefficients [11]. Borate nanoparticles have received much attention [12-14]. Borate materials are now interesting and technologically relevant subject of research because of a series of unique properties and specific features which make them attractive for numerous practical applications as optical compounds.

Among various optically active systems, materials doped with chromium ions in inorganic hosts are widespread because of deep colors and bright photoluminescence [15]. Trivalent chromium is the most stable state of chromium and it effectively interacts with lattice and has high luminescent quantum efficiency as dopant [16]. Ions with the d<sup>3</sup> electronic configuration Cr<sup>3+</sup> in particular are widely used as paramagnetic probe to investigate the

symmetry of intra crystalline electric fields [17,18].  $\text{Cr}^{3+}$  ions tend to form octahedral complexes and the colors of these complexes can be determined by the ligands attached to the Cr center. This unique feature has attained a technological importance in the development of tunable solid state lasers and new luminescent materials [19-21]. Dittmann et al. [22], reported the emission, reflection- and excitation spectra of a manganese activated  $\text{Cd}_2\text{B}_2\text{O}_5$ , powder phosphor at different temperatures. This paper deals the synthesis of  $\text{Cr}^{3+}$  doped Cadmium borate nanopowder by chemical precipitation method and spectral studies.

#### Experimental details:

**Chemicals required:** Cadmium nitrate, Borax, Chromium trioxide, deionized water and ethanol.

Cadmium nitrate ( $\text{Cd}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ ), Borax ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ), Chromium oxide ( $\text{Cr}_2\text{O}_3$ ), ethanol were purchased from Merck Chemicals, India. And de-ionized water used as solvent all of the chemical

reagents used in this experiment were analytical grade and used without further purification.

Cadmium nitrate (1.54gms of  $\text{Cd}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$  dissolved in 50 ml de-ionized water (0.1 M)) and borax (1.9068gms of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$  dissolved in 50 ml de-ionized water (0.1 M)) both the solutions are mixed and stirred, during stirring processes Chromium trioxide (0.00499gms of  $\text{Cr}_2\text{O}_3$  dissolved in 50 ml of ethanol (0.001M)) solution was added drop wise to the mixer solution. The uniform magnetic stirring was provided for better atomic diffusion during the length of the reaction. The resulting precipitates were filtered off and washed several times with de-ionized water. The precipitate was then dried in a hot air oven at  $350^\circ\text{C}$ . The final product was then obtained by crushing the dried precipitate using pestle and mortar. The powder is yellow in color. The synthesized  $\text{Cr}^{3+}$  doped  $\text{Cd}_3(\text{BO}_3)_2$  nanopowder was shown in Fig.1.



Fig. 1 Synthesized  $\text{Cr}^{3+}$  doped  $\text{Cd}_3(\text{BO}_3)_2$  nanopowder

**Characterizations:** Powder X-ray diffraction pattern of the prepared sample is recorded on PANalytical Xpertpro diffractometer with  $\text{CuK}\alpha$  radiation. Scanning Electron Microscope and Energy Dispersive Spectrum (EDS) images were taken from ZEISS EVO18. Optical absorption spectrum is recorded at room temperature on JASCO V-670 Spectrometer in the wave length region of 300-800nm. EPR spectrum is recorded at room temperature on JES-FA series X-band EPR Spectrometer having 100 KHZ field modulations. To recorded the photoluminescence (PL) Spectrum Horiba Jobin-Yvon Fluouolog3 Spectro fluorimeter with Xe continuous (450W) and pulsed (35W) lamps as excitation sources was used. FT-IR spectrum of

the prepared sample was obtained from Bruker's FT-IR Spectrometer in the region of  $4000\text{-}500\text{cm}^{-1}$ .

## II. RESULTS AND DISCUSSION

### Powder X-ray Diffraction Study

Fig.2 shows the powder X-ray diffraction patterns of  $\text{Cr}^{3+}$  doped  $\text{Cd}_3(\text{BO}_3)_2$  nanopowder. The incorporation of dopant ions does not much have change the phase composition of prepared samples. The diffraction data is indexed to orthorhombic crystal system and the corresponding lattice cell parameters are evaluated  $a = 0.592$ ,  $b = 0.484$ ,  $c = 0.898$  nm and  $V = 25.767$   $\text{nm}^3$  [23].

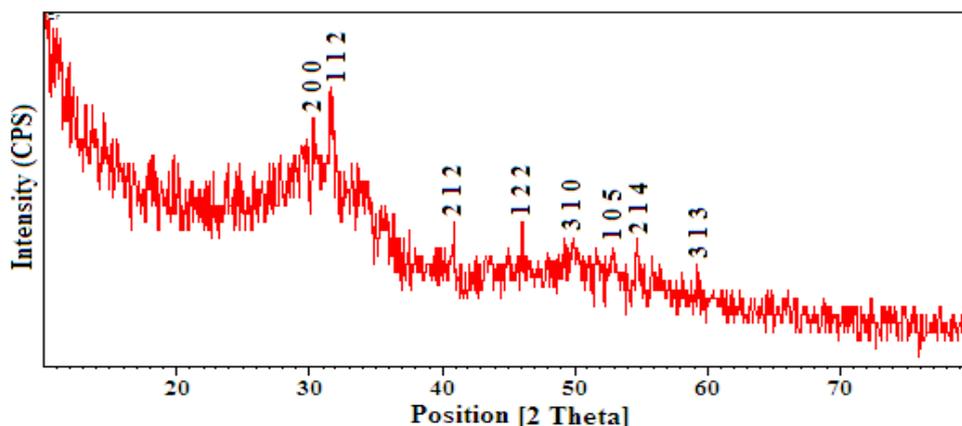


Fig. 2 Powder XRD pattern of Cr<sup>3+</sup> doped Cd<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub> nanopowder

Scherrer's equation has been applied to evaluate the average crystallite size of the sample.

$$D = 0.9 \lambda / \beta \cos \theta$$

where D is crystallite size (nm),  $\lambda$  is X-ray wavelength,  $\beta$  is full width at half maximum intensity and  $\theta$  is diffraction angle. The evaluated average crystallite size is 31 nm for Cr<sup>3+</sup> doped sample.

#### SEM and EDX Analysis:

The morphology and microstructure of prepared nanopowder was investigated using SEM. The SEM image shows the nano powder particles with some conglomeration among the crystalline grains and it possess an irregular morphology. The flower like grains seen in the SEM images are the domains formed by aggregation of the nano-size crystallites shown in Fig.3

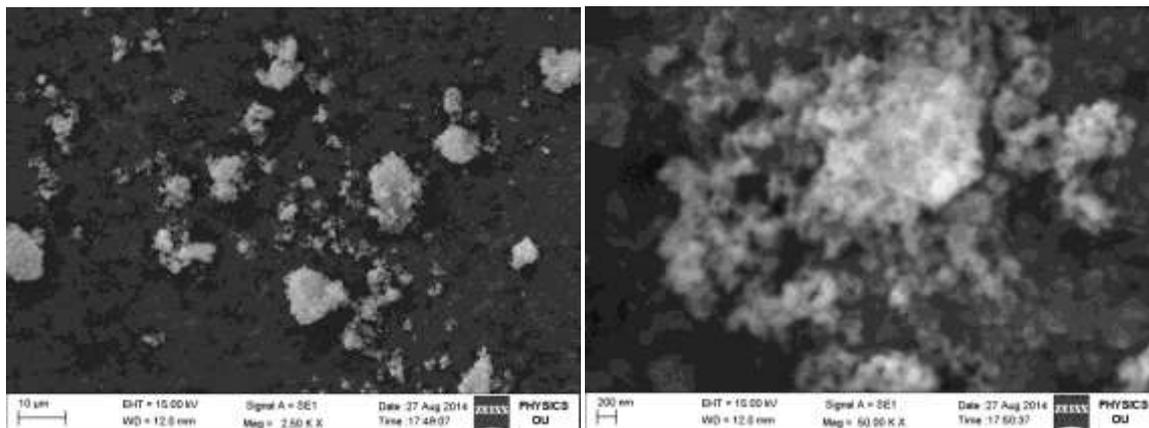


Fig.3.SEM of Cr doped Cd<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub>

To determine the composition of the prepared sample was performed for EDX measurements for characterizing the compositions of the nano powder. The selected area of the prepared sample was also performed for EDS measurement for characterizing the compositions of the borate nanopowder. The spectrum reveals the elemental

composition as per the stoichiometric ratio of the cadmium and confirms the formation of the sample by the synthesis method. It is found that EDS data demonstrates the prepared sample is composed of cadmium, borate, and chromium species. This is shown in Fig.4.

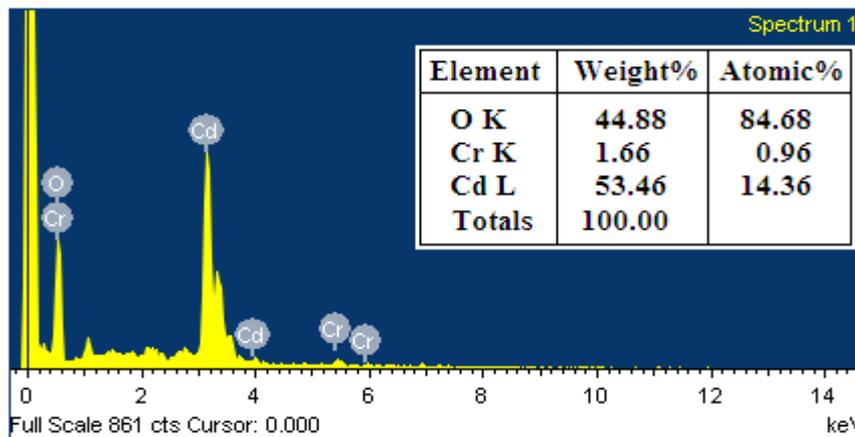


Fig.4 EDX pattern of Cr<sup>3+</sup> doped Cd<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub> nanopowder

**EPR studies:**

The electron paramagnetic resonance (EPR) method provides a detailed description of the nature of the electric field symmetry produced by the ligands around the paramagnetic ions [24-27] and spectrum shown in Fig.5. EPR spectra of Cr(III) ions

in cadmiumborate nanopowder as host material, Cr<sup>3+</sup> is a d<sup>3</sup> system and with electronic spin S = 3/2 and nuclear spin I = 3/2 the fine structure EPR lines of Cr<sup>3+</sup> with an effective spin S = 3/2. The interpretation of EPR spectrum is usually performed with the help of a spin Hamiltonian.

$$H = \mu_B g H \cdot S + D[S_z^2 - S(S+1)/3] + E(S_x^2 - S_y^2)$$

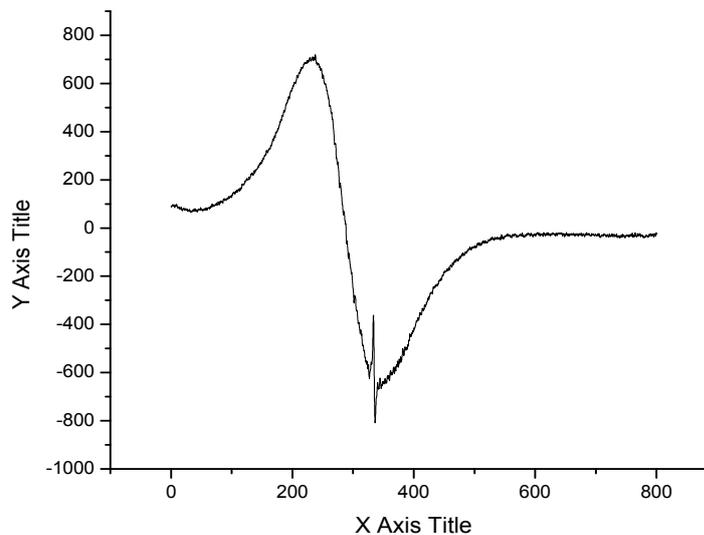


Fig.5. EPR Spectra of Cr doped Cd<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub>

where D and E are zero field splitting parameters and the other symbols have their usual meaning [24]. Where the terms involving D and E are the axial and orthorhombic crystal-field components respectively, S is the spin operator and g is the gyro magnetic tensor. In this study Cr(III) ions in cadmium borate nano powder the spin Hamiltonian parameters were found to be g = 1.970, which is typical for the Cr<sup>3+</sup> ion and its value agreed fairly well with the previously reported values [28]. These values are in the order of the parameters given

in the literature for the Cr<sup>3+</sup> doped in various compounds [29, ]. The g value obtained from EPR results Cr<sup>3+</sup> doped cadmium borate nano powder is indicative of a distorted octahedral site in the host. The bonding can be predicted using the expression [30].

$$h = [(B_{free} - B) / B_{free}] / k_{cr}^{3+}$$

where h and k are nephelauxetic parameters of the ligands and the central metal ion, respectively. For Cr<sup>3+</sup> ion, the value of k = 0.21 [31]. The calculated value of h is 1.862. The value of h indicates

increased delocalization of the d-electrons suggesting covalent bonding nature between  $\text{Cr}^{3+}$  and the ligands. By correlating optical and EPR data, the chemical bonding parameter is evaluated using the formula [32]

$$g_0 = g_e - (8 \alpha \lambda) / \Delta$$

where  $g_e$  represents the free-electron  $g$  value (2.0023),  $\lambda$  is the spin orbit coupling constant ( $91 \text{ cm}^{-1}$ ),  $\Delta$  is the gap between the excited and ground levels. From experimental values  $g_0 = 1.970$  and  $\Delta = 17600 \text{ cm}^{-1}$ , the value of  $\alpha$  is evaluated to be 0.4435. The evaluated value of  $\alpha = 0.4435$  suggests that the  $\text{Cr}^{3+}$  ion exhibits the covalent nature in the  $\text{Cd}_3(\text{BO}_3)_2$  nanopowder.

### Optical studies:

Optical absorption properties of semiconductors are related to energy band structure, which are estimated by optical absorption spectrum. Absorbance is expected to depend on several factors such as band gap, surface roughness and impurity centres.  $\text{Cr}^{3+}$  ions belong to  $d^3$  configuration and ground state is  ${}^4A_{2g}(\text{F})$  at all strengths of the crystal field. In an octahedral crystal field,  $\text{Cr}^{3+}$  ions are characterized by three spin allowed transitions  ${}^4A_{2g}(\text{F}) \rightarrow {}^4T_{2g}(\text{F})$ ,  ${}^4A_{2g}(\text{F}) \rightarrow {}^4T_{1g}(\text{F})$  and  ${}^4A_{2g}(\text{F}) \rightarrow E_g(\text{G})$ . Optical absorption spectrum of  $\text{Cr}^{3+}$  doped  $\text{Cd}(\text{BO}_3)_2$  nanopowder at the room temperature (RT) and is shown in Fig.6.

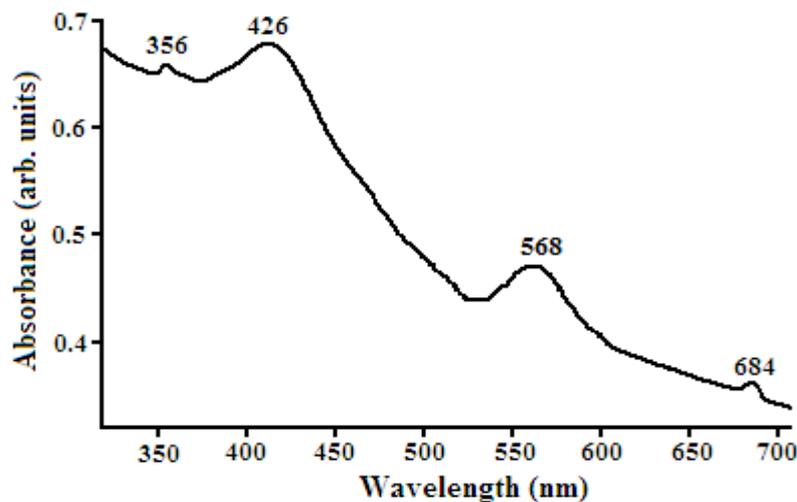


Fig.6. Optical Spectra of Cr doped  $\text{Cd}_3(\text{BO}_3)_2$

In present investigation, optical absorption spectrum of  $\text{Cr}^{3+}$  doped cadmium borate nanopowder contains four absorption bands. The RT spectrum exhibits two strong bands at 568, 426nm and two weak bands at 356, 684 nm. The broad bands 568 and 426 nm are observed and attributed to the spin allowed transitions  ${}^4A_{2g}(\text{F}) \rightarrow {}^4T_{2g}(\text{F})$ ,  ${}^4A_{2g}(\text{F}) \rightarrow {}^4T_{1g}(\text{F})$  respectively. The band position ( $\nu_1 = 17600\text{cm}^{-1}$ ) corresponding to the transition  ${}^4A_{2g}(\text{F}) \rightarrow {}^4T_{1g}(\text{F})$  gives the 10 Dq value. The wave number of the band corresponding to  ${}^4A_{2g}(\text{F}) \rightarrow {}^4T_{2g}(\text{F})$ ,  $23477 \text{ cm}^{-1}(\nu_2)$  [33]. By using the following formula the value of B is evaluated and found to be  $559\text{cm}^{-1}$  [34].

$$B = (2 \nu_1^2 + \nu_2^2 - 3 \nu_1 \nu_2) / 15 \nu_2 - 27 \nu_1$$

The value of C is evaluated using the relation given by [35].

$$C/B = 1/3.05 [E^2(E) / B - 7.9 + 1.8 (B / Dq)].$$

The calculated value of C is  $3450\text{cm}^{-1}$ . The other weak bands at 684nm and 356 nm are attributed, with the help of Tanabe-Sugano diagram [36,37] to the spin forbidden transitions from  ${}^4A_{2g}(\text{F})$  to  ${}^2E_g(\text{G})$  and  ${}^2A_{1g}(\text{G})$  respectively. Based on the above assignments the energy matrices for different values of Dq, and C are solved and the following values give good agreement between the calculated and observed band positions:  $Dq = 1760$ ,  $B = 559$  and  $C = 3355\text{cm}^{-1}$ . The Band head data along with their assignments are presented in table 1.

### PL Analysis:

PL emission spectra of cadmium borate were recorded for an excitation wavelength of 400 nm and emission spectrum of Cr(III) ions doped cadmium borate with an excitation of 420 nm. The emission spectrum shows three sharp emission peaks in the visible region at 462, 519 and 689nm. Chromium doped cadmiumborate nanopowder crystals prepared in the present study exhibit two prominent peaks in the PL spectra and the peaks are observed at 462 and 519 due to (i) band-edge

luminescence band and (ii) surface state luminescence bands. The PL spectra indicate clearly that the doping of  $\text{Cr}^{3+}$  in the host material leads significant changes in PL intensity. Fig.7 shows the

emission spectrum of  $\text{Cr}^{3+}$  ions doped cadmium borate, To study the influence of  $\text{Cr}^{3+}$  doping blue emission peak at 462 nm.

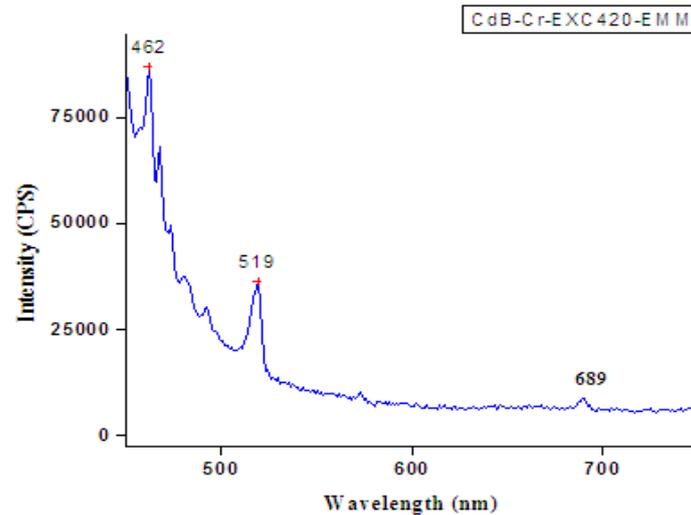


Fig.7. PL Spectra of Cr doped  $\text{Cd}_3(\text{BO}_3)_2$

The blue emission peak at 462 nm is a host lattice emission due to intrinsic impurities in host lattice, which was observed in undoped cadmium borate 436nm. The green emission band near 519 nm is assigned to the transition  ${}^4\text{T}_{2g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$ . The red emission band at 689 nm (R line) is a deep level emission band characteristic of Cr(III) ions which is caused by the spin-forbidden  ${}^2\text{E}_g \rightarrow {}^4\text{A}_{2g}$  transition [[38,39] and also the emission band results from coupling of phonon with emission photon of  $\text{Cr}^{3+}$  ions [40,41]. Similar emission was also observed in various chromium doped hosts [42]. The emission of Cr(III) ions with cadmium borate might be used in the field of display and lighting.

**Chromatic properties:**

The CIE chromaticity coordinates of Cr(III) ions doped  $\text{Cd}_3(\text{BO}_3)_2$  nanopowder are calculated from their corresponding emission spectrum under excitation of 420 nm. The colour of the prepared borate nanopowder is graphically represented in a two dimensional CIE x-y plot and is depicted in the Fig.8. The CIE coordinates are observed at  $(x = 0.2086, y = 0.2355)$  indicated with a solid sign (\*). This indicates that the emission color of prepared borate is near to that of pale blue region.

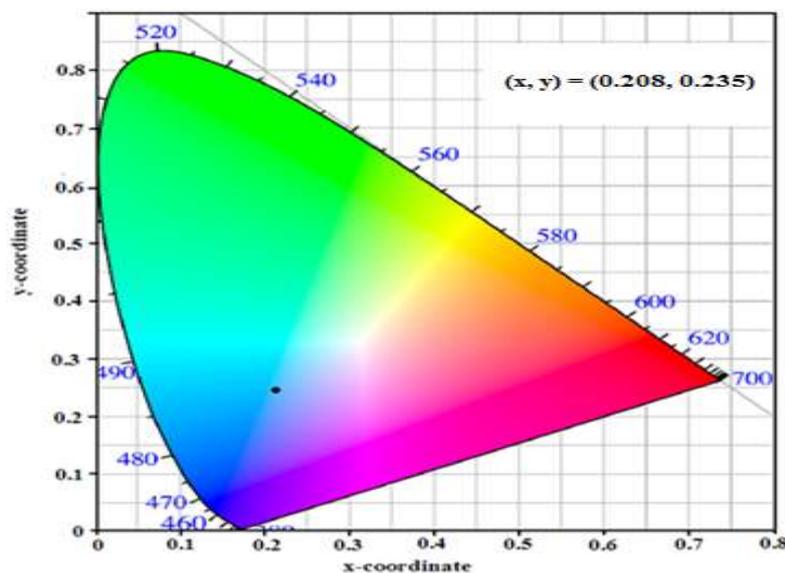


Fig.8. CIE of Cr doped  $\text{Cd}_3(\text{BO}_3)_2$

### FT-IR studies:

The FT-IR spectrum of Cr<sup>3+</sup> doped Cd<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub> the spectrum exhibited the symmetric asymmetric stretching and bending vibrations of water, and hydroxyl groups of the compound. The FTIR spectrum of pure cadmium borate and Cr-doped cadmium borate nano powder is as shown in Fig.9. In the higher energy region, the peak at 3200-3450 cm<sup>-1</sup> is assigned to O-H stretching due to rise to absorption in this region and the possibility of some adsorbed water [43]. The spectra of the Cr<sup>3+</sup> doped Cd<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub> can be divided in to the following three regions in that 600-800cm<sup>-1</sup> is related to the bending vibrations of various borate arrangements B-O-B, the next is 800-1000 cm<sup>-1</sup> is taken as B-O stretching vibrations of tetrahedral BO<sub>4</sub> and finally 1200-1400 cm<sup>-1</sup> the peak in this region correlated to B-O stretching vibrations of BO<sub>3</sub> units [44-46]. The band around 1357 cm<sup>-1</sup> is assigned to B-O stretching vibrations of trigonal (BO<sub>3</sub>)<sup>3-</sup> units in metaborates [47]. The band 640 - 684 cm<sup>-1</sup> can be attributed to bending vibrations of BO<sub>3</sub> triangles and stretching vibrations of BO<sub>3</sub> units with non bridging oxygens respectively. The peak at around 560 cm<sup>-1</sup> -590 cm<sup>-1</sup> can be attributed to the borate deformation modes such as the inplane bending of boron-oxygen triangles [48].

### III. CONCLUSIONS

Cr<sup>3+</sup> doped Cd<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub> nanopowder was synthesized and characterized the following conclusion are drawn: From the XRD data, it is confirmed that average crystalline size was in the order of nanoscale and belongs to orthorhombic crystal cell. The lattice cell parameters are evaluated as a = 0.592, b = 0.484, c = 0.898 nm and V = 25.767 nm<sup>3</sup>. SEM images showed the formation of irregular shaped particles. EDX data indicates, presence of doped metal ion along with chemical mapping. From optical and EPR data, the evaluated parameters exhibited that site symmetry of doped metal ion confirmed as octahedral with host lattice. Crystal field and Racah parameters are evaluated. The bonding nature between metal ions with its ligands is covalent. PL spectrum exhibited three sharp emission peaks in the visible region at 462, 519, 689 nm and their corresponding CIE coordinates are in blue region at (x = 0.208, y = 0.235). FT-IR data showed the characteristic vibrations of borate groups.

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#### Highlights

- Production of nanopowder by simple chemical precipitation method
- Incorporation of transition metal ions reduced the crystallite size of host lattice
- Octahedral site symmetry of doped ion in host with covalent bond
- Exhibit good Photoluminescence properties with CIE chromaticity coordinates

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