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Synthesis and Photoluminescence Properties of $CaB_4O_7:Pb^{2+}$

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Abstract –

The powder sample of $CaB_4O_7:Pb^{2+}$ has been prepared by a novel method which is slight variation of solution Combustion Synthesis Method. The synthesis is based on the exothermic reaction between the fuel (Urea) and Oxidizer (Ammonium nitrate). The synthesized materials were determined by the powder X-ray diffraction and found in good agreement with ICDD file. The photoluminescence properties of prepared phosphor were investigated using a spectroflurometer at room temperature. The synthesized phosphors give the emission in the UV region. It was observed that the optimum concentration of Pb^{2+} in $CaB_4O_7:Pb^{2+}$ is found to be 0.5% (relative to Ca) respectively. The Stoke shifts of phosphors $CaB_4O_7:Pb^{2+}$ was calculated to be 7097 cm^{-1}.

***Keywords*- Borate, Combustion synthesis, UV Emitting, Photoluminescence.**

I. INTRODUCTION

Ultraviolet radiation (UVR) makes up a fraction of the electromagnetic spectrum, which can be further subdivided into UVC, UV Band UVA [1].

UVC is used in germicidal applications, and most drinking water purifiers use UVC sources. UVB and UVA are used in many applications such as photocopying, phototherapy, sunlamp, laundry marking, blueprinting, instrumental dial illumination, window displays, etc.[2]. Many attempts have been made upon synthesizing Pb^{2+} activated phosphors which emits in varying range from 290-470 nm when excited by UV light, given in table 1. Inrecentyears, great efforts have been taken by many researchers to discover and develop new rare-earth and transition-metal ion-doped material systems as luminescent materials with high absorption in the UV spectral region [3]. Inorganic materials containing metal ions with s^2 (Pb^{2+}, Tl^+, Sn^{2+}, Sb^{3+}, Bi^{3+}) configuration can be used in X-ray imaging devices, low pressure lamps, and high-energy physics. For example, Pb^{2+} doped BaSi2O5, which emits a broad band around 350 nm under UV excitation, is one of the earliest known phosphors for photocopying lamps [4].

Host lattices which are easily synthesizable and structurally viable for doping at multiple cationic sites present in the lattice are of recent interest. The borate atom has two types of hybridized orbitals, the planar sp^2 and the three dimensional sp^3, to coordinate three or four oxygen atoms to form various B_xO_y complex anionic groups. Therefore many types of borate crystals have been found to be constructed based on these complex anionic groups [5]. Therefore, inorganic borates have long been a focus of research. Inorganic borates are excellent host materials because of their variety of structure type, large electronic band gap, transparency to a wide range of wavelengths, high optical damage threshold and high optical quality [6-9].

In the present work, CaB_4O_7material with different molar ratios of Pb^{2+} were synthesized by novel combustion synthesis method, which is described in our previous work[10]. The synthesized material was characterized by using the powder X-Ray Diffraction and t heir photoluminescence properties at room temperature were studied using a spectro fluorometer.

II. METHOD and MATERIALS

Inorganic borate phosphor was prepared by a method which is a variation of the combustion synthesis [11-13]. The method based on exothermic reaction in which ammonium nitrate used as oxidizer and urea is used as fuel. The stoichiometric amounts of high purity starting materials $Ca(NO_3)_2$ (A.R.), $Pb(NO_3)_2$ (A.R.), H_3BO_3 (A.R.), $CO(NH_2)_2$ (A.R.), NH_4NO_3 (A.R.) have been used for sample preparation. The starting materials with little amount of DD water were mixed thoroughly in agate mortar to obtain homogeneous solution. The excess water have been removed by slow heating (70^{0}C) and the solution then transferred directly to the pre-heated furnace (550°C) for combustion. Following the combustion, the resulting fine powders were annealed for 2 hr at temperature 700 °C and then suddenly cooled to room temperature. For confirmation of compound formation, XRD of synthesized material was taken on Rigaku Miniflex X-ray Diffractometer and compared with the ICDD file. PL measurements at room temperature were performed on Hitachi F-7000 spectrophotometer in the range 200–500 nm.

III. RESULTS and DISCUSSION

X-Ray Diffraction pattern of synthesized materials

The XRD of synthesized material shown in Fig.1, was taken on Rigaku Miniflex X-ray Diffractometer, which is in good agreement with the ICDD file (00-031-0253). The crystal structure of CaB4O7 is monoclinic with space group P21/n.

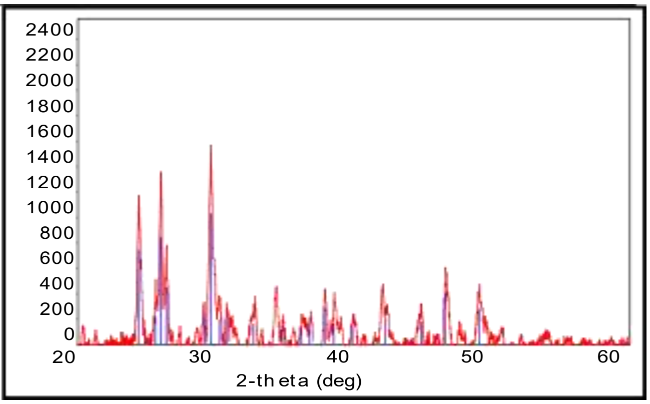

Fig. 1. XRD pattern of CaB_4O_7

IV. Photoluminescence Analysis

The luminescence of Pb^{2+} in host materials is diverse. It can be described by the $^1S_0 \rightarrow {}^3P_{0,1}$ transition which originates from $(6s)^2 \rightarrow (6s)^1(6p)^2$ inter configurational transition. Typically at room temperature, emission is observed from the $^3P_1 \rightarrow {}^1S_0$transition, although at low temperatures the highly forbidden $^3P_1 \rightarrow {}^1S_0$emission is also observed [14,15]. In Fig. 3, the excitation band of CaB_4O_7:Pb^{2+} was observed at 270 nm, which is assigned to the$^1S_0 \rightarrow {}^3P_1$transition. The emission band was observed at 334 nm from the $^3P_1$ excited state level to the $^1S_0$ ground state upon excitation with 270 nm Additionally, we observed no splitting or multiple bands in the emission spectra for prepared phosphors, therefore, we believe that the Pb^{2+} ions are incorporated in to only one site in the crystal lattice. Thus, Pb^{2+} ions in CaB4O7are occupy the Ca^{2+} sites in the phosphors according to the ionic size considerations. Also, the emission and excitation spectra of the synthesized materials $CaB_4O_7$with differentPb^{2+} doping concentrations were analyzed at room temperature. The optimum concentration of Pb^{2+} in CaB_4O_7 was found to be 0.5% (relative to Ca), which is due to concentration quenching of Pb^{2+} in hosts. The concentration quenching in inorganic phosphors has been attributed to the migration of excitation energy to the quenching centers (traps) or to the cross-relaxation mechanisms. Also, it is observed that for different

Pb^{2+} doping concentrations, the shapes and positions of the excitation and emission bands have exhibited no changes for the phosphors, which also conforms the repeatability of prepared samples. The Stokes shift of $CaB_4O_7:Pb^{2+}$ phosphor was calculated to be7097 cm^{-1}.

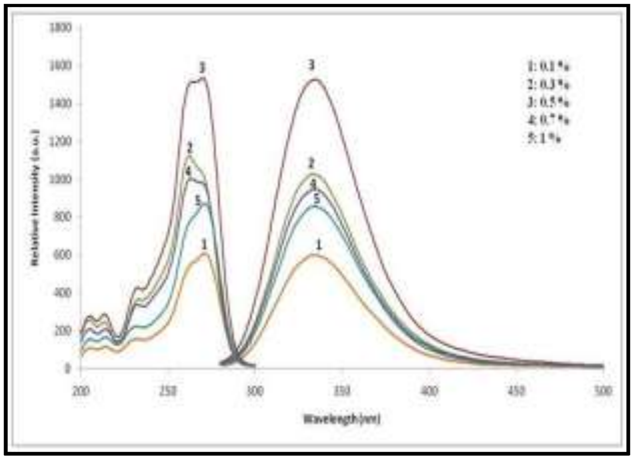

Fig. 2: Photoluminescence emission and excitation spectra.

TABLE I

Spectroscopic properties of some Pb^{2+} doped inorganic borate host phosphors

Materials	λ_{ex} (nm)	λ_{em} (nm)	Stockshift (cm^{-1})	Ref.
CaB_4O_7	270	334	7097	Present work
$LiCaBO_3$	265	296	3952	13
$Li_6CaB_3O_{8.5}$	268	307	4740	14
$Sr_2Mg(BO_3)_2$	260	330	8159	15
$Ba_2Mg(BO_3)_2$	293	381	7883	15
$SrLaBO_4$	254	470		16
$CaLaBO_4$	254	465		16

V. CONCLUSION

Pb^{2+} doped CaB_4O_7 material was successfully prepared by a novel combustion synthesis method followed by heating of the precursor combustion ash for 2 hours at 700^{0}C in air. Powder XRD conform the structure of synthesized material. The materials achieve ab and emission at 334 nm (λ_{ex}=270 nm),corresponding to the transition $^3P_1 \rightarrow ^1S_0$under the excitation of 270 nm. The Stokes shift of $CaB_4O_7:Pb^{2+}$ phosphor was calculated to be 7097 cm^{-1}.

REFERENCES

[1] C. Hoare, A. Li Wan Po and H. Williams, *Health technology assessment*, Vol 4: No. 37, pp. 88-89, 2000.

[2] Ilhan Pekgozlu , Ertugrul Erdogmus, Bilal Demirel, M. Sabri Gok, Hakan Karabulut and Ali Sadi Basak, *Journal of Luminescence*, 131,2290–2293,(2011).

[3] J.A. Groenink and G. Blasse, *J. Solid State Chem.*, 32 1, 1980.

[4] C.K. Lin, M. Yu, M.L. Pang and J. Lin, *Opt. Mater.*,28, 913,2006.

[5] Y. Zhang, X.L. Chen, J.K. Liang, Y.G. Cao and T. Xu, *J. Alloys Compd.*, 315, 198,2001.

[6] L.Wu, X.L.Chen, Y.Zhang, Y.F.Kong, J.J.Xu and Y.P.Xu, *J.Solid State Chem.* 179,1219, 2006.

[7] L.Wu, X.L.Chen, H.Li, M.He, Y.P.Xu and X.Z.Li, *Inorg. Chem.,* 44, 6409, 2005.

[8] P.Gravereau, J.P.Chaminade, S.Pechev, V.Nikolov, D.Ivanova and P.Peshev, *Solid State Sciences*, 4, 993, 2002.

[9] W.Park, C.J.Summers, Y.R.Do and H.G.Yang, *J. Mater. Sci.* 37, 4041, 2002.

[10] A. B. Gawande, R.P. Sonekar, S.K. Omanwar,*Optical Materials,* 36,1143, 2014.

[11] A.B. Gawande, R.P. Sonekar and S.K. Omanwar, *J. Lumin*,149, 200, 2014.

[12] A.B. Gawande, J.T. Ingle, R.P. Sonekar and S.K. Omanwar,*J. Lumin.*, 149, 236, 2014.

[13] A.B. Gawande, R.P. Sonekar and S.K. Omanwar, *Mater. Res. Bull.*, 60, 285, 2014.