Research Article

Synthesis and Photoluminescence Study of Bi^{3+} and Pb^{2+} Activated $\text{Ca}_3(\text{BO}_3)_2$

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The photoluminescence properties of Pb^{2+} and Bi^{3+} doped $\text{Ca}_3(\text{BO}_3)_2$ prepared by solution combustion synthesis technique are discussed. The structure of the prepared phosphor is characterized and conformed by XRD and FTIR. SEM images of the prepared materials show irregular grains with agglomerate phenomena. Prepared phosphors achieved the band emissions, respectively, at 365 nm and 335 nm corresponding to the transition $^3\text{P}_1 \rightarrow ^1\text{S}_0$. Optimum concentration, critical transfer distance, and Stokes shift of the synthesized materials were measured. These phosphors may provide an efficient kind of luminescent materials for various applications in medical and industry.

1. Introduction

Several researchers studied the luminescence properties of Bi^{3+} doped phosphors [1–6]. Bismuth can exist in materials in different valence states, such as 0, +1, +2, +3, and +5, or even mixed valence states of +1 and +5. In all of these valence states only Bi^{3+} is normally most stable in most host materials. Usually, the emission peaks of Bi^{3+} occur in the ultraviolet, blue, green, or even red wavelength regions with variation of host materials. For example, $\text{GaBO}_3:\text{Bi}^{3+}$ [7] and $\text{YBO}_3:\text{Bi}^{3+}$ [8] show UV emission, $\text{LnNbO}_4:\text{Bi}^{3+}$ [9] gives blue emission, $\text{CaWO}_4:\text{Bi}^{3+}$ [10] and $\text{Ca}_3\text{Al}_2\text{O}_6:\text{Bi}^{3+}$ give green emission [11], and $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ [12] displays red emission at low temperature. Pb^{2+} doped phosphors have been the interest of many researchers. Many attempts have been made to synthesize Pb^{2+} activated phosphors which emit in varying range from 290 to 470 nm when excited by UV light [13–22]. In recent years, 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 [23]. 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 BaSi_2O_5 , which emits a broad band around 350 nm under UV excitation, is one of the earliest known phosphors for photocopying lamps [24].

In the present work, two inorganic phosphors $\text{Ca}_3(\text{BO}_3)_2:\text{Bi}^{3+}$ and $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$, were prepared using solution combustion synthesis technique. The synthesized phosphors were characterized by using the powder X-Ray Diffractometer (XRD), Fourier Transform InfraRed (FTIR), and Scanning Electron Microscope (SEM). The photoluminescence properties of these phosphors at room temperature were studied using a Spectrofluorometer.

2. Experimental

The phosphors were prepared by a novel technique which is slight variation of solution combustion synthesis method. Detailed description of the method is given in our previous work [25–30]. Bi^{3+} and Pb^{2+} doped $\text{Ca}_3(\text{BO}_3)_2$ phosphors were obtained by the combustion of aqueous solution containing stoichiometric amounts (using oxidizer/fuel ratio) of calcium nitrate (AR), lead nitrate (AR), bismuth nitrate (AR),

TABLE 1: Molar ratio of ingredients used for material preparation.

Material	Molar ratio
$(\text{Ca}_{1-x}\text{Bi}_x)_3(\text{BO}_3)_2$	$\text{Ca}(\text{NO}_3)_2 : \text{Bi}(\text{NO}_3)_3 : \text{H}_3\text{BO}_3 : \text{CO}(\text{NH}_2)_2 : \text{NH}_4\text{NO}_3$ $3 \times (1-x) : 3 \times x : 2 : 6 : 3$ $(x = 0.001, 0.002, 0.003, 0.005, 0.01)$
$(\text{Ca}_{1-x}\text{Pb}_x)_3(\text{BO}_3)_2$	$\text{Ca}(\text{NO}_3)_2 : \text{Pb}(\text{NO}_3)_2 : \text{H}_3\text{BO}_3 : \text{CO}(\text{NH}_2)_2 : \text{NH}_4\text{NO}_3$ $3 \times (1-x) : 3 \times x : 2 : 6 : 3$ $(x = 0.001, 0.003, 0.005, 0.01, 0.02, 0.03, 0.04)$

ammonium nitrate (AR), urea (AR), and boric acid (AR) as boron source (Table 1). All the precursors were dissolved in a china dish using little amount of double distilled water. The dish containing the solution was introduced into a muffle furnace maintained at $550 \pm 10^\circ\text{C}$. The solution undergoes dehydration followed by decomposition with the evolution of large amount of gases (oxides of nitrogen and ammonia) and ignited to burn with a flame yielding voluminous powder of $\text{Ca}_3(\text{BO}_3)_2 : \text{Bi}^{3+}$ and $\text{Ca}_3(\text{BO}_3)_2 : \text{Pb}^{2+}$. These raw powders were sintered for 3 h at $800 \pm 10^\circ\text{C}$ and quenched to room temperature on aluminum plate and crushed into a fine powder. The prepared powder samples were then subjected to the powder XRD analysis. PL measurements at room temperature were performed on Hitachi F-7000 Spectrofluorometer in the range 200–500 nm.

3. Results and Discussion

3.1. X-Ray Diffraction Pattern of Synthesized Materials. The powder XRD of synthesized material, analyzed on Rikagu Miniflex II X-ray Diffractometer, is shown in Figure 1 and found in good agreement with the ICDD file (01-070-0868). The crystal structure of the prepared material $\text{Ca}_3(\text{BO}_3)_2$ can be refined to be rhombohedral, space group R-3c with lattice parameters $a = b = 8.638 \text{ \AA}$ and $c = 11.849 \text{ \AA}$.

3.2. FTIR Spectra of Synthesized Materials. The FTIR spectra measured at room temperature are shown in Figure 2. Generally, the FTIR analysis of the studied borate material shows four distinct frequency regions, from 1200 to 1600 cm^{-1} and from 800 to 1200 cm^{-1} , which are assigned to the stretching vibrations of both triangular BO_3 and tetrahedral BO_4 units, respectively. As usual, frequencies of B–O vibrations depend on the boron coordination. Stretching frequencies of a coordinated BO_n groups decrease as the coordination number n increases. Thus, the experimental results indicate that B–O bond asymmetric stretching of BO_3 groups is located in the region from 1150 to 1500 cm^{-1} . Moreover, the condensation of BO_3 groups (either chains or rings) leads to two types of B–O bands. One is short B–O bands (oxygen bonded to a single B atom or nonbridging oxygen). The other is longer B–O–B bonds. The responding B–O stretching frequencies are accordingly distributed into two bands or groups of bands, centered near 1450 and 1200 cm^{-1} [31]. As seen in

FIGURE 1: X-ray powder diffraction patterns of $\text{Ca}_3(\text{BO}_3)_2$ synthesized by solution combustion synthesis. FIGURE 2: FTIR spectra of $\text{Ca}_3(\text{BO}_3)_2$ synthesized by solution combustion synthesis.

Figure 2, the strong bands observed above 1200 cm^{-1} should be assigned to the B–O stretching mode of the triangular $[\text{BO}_3]^{3-}$ groups, while the bands with maxima at about 700 – 800 cm^{-1} should be attributed to the B–O out of plane bending, which confirms the existence of the $[\text{BO}_3]^{3-}$ groups [32, 33].

3.3. SEM Images of Synthesized Phosphors. Figure 3 shows the Scanning Electron Microscope (SEM) images of powders prepared at 800°C . It was observed that the microstructure of the phosphors consisted of irregular grains with agglomerate phenomena. The average size of the powders is about 2 – $12 \mu\text{m}$. The results show that phosphors have a good crystallinity and a relatively low sinter temperature.

3.4. Photoluminescence of $\text{Ca}_3(\text{BO}_3)_2 : \text{Bi}^{3+}$. Figure 4 displays the excitation and emission spectra of $\text{Ca}_3(\text{BO}_3)_2 : \text{Bi}^{3+}$. The ground state of Bi^{3+} ion with $6s^2$ configuration is the 1S_0 spin-orbit singlet state and the excited states of the $6s6p$ configuration are 3P_0 , 3P_1 , 3P_2 , and 1P_1 states in sequence of energy increase. The transition between 1S_0 and 1P_1 is parity and spin-allowed, while the transition between 1S_0 and 3P_1

FIGURE 3: SEM images of (a) $\text{Ca}_3(\text{BO}_3)_2:\text{Bi}^{3+}$ and (b) $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$.

FIGURE 4: PL and PLE spectra of $(\text{Ca}_{1-x}\text{Bi}_x)_3(\text{BO}_3)_2$ ($x = 0.001, 0.002, 0.003, 0.005, 0.01$), excitation spectra monitored at 365 nm emission, and emission spectra monitored at 290 nm excitation.

is largely allowed due to spin-orbit coupling. Other transitions are strictly forbidden. Typically at room temperature, emission is observed from the $^3\text{P}_1 \rightarrow ^1\text{S}_0$ transition. As an activator, excitation usually occurs from the $^1\text{S}_0$ ground state to the $^3\text{P}_1$ or $^1\text{P}_1$ excited state because the $^1\text{S}_0 \rightarrow ^3\text{P}_0$ and $^1\text{S}_0 \rightarrow ^3\text{P}_2$ transitions are strongly forbidden. The emission of Bi^{3+} ions originates from the $^3\text{P}_0$ state at low temperatures [34], while at higher temperatures the emission occurs mainly from the $^3\text{P}_1$ level, in which transition is allowed by spin-orbit mixing of the $^3\text{P}_1$ and $^1\text{P}_1$ states. In Figure 4, broad excitation bands are in the region from 260 nm to 310 nm with maximum at 290 nm which can be ascribed to $^1\text{S}_0 \rightarrow ^3\text{P}_1$ transition of Bi^{3+} . The emission band is observed at 365 nm from the $^3\text{P}_1$ excited state level to the $^1\text{S}_0$ ground state upon excitation with 290 nm. Additionally, we observed no splitting or multiple bands in the emission spectra, which indicate that Bi^{3+} here is supposed to occupy the position of Ca^{2+} ions in the lattice. In view of the extensive literature on the luminescence of Bi^{3+} in inorganic host, it can be seen that

FIGURE 5: Variation in emission intensity of $(\text{Ca}_{1-x}\text{Bi}_x)_3(\text{BO}_3)_2$ ($x = 0.001, 0.002, 0.003, 0.005, 0.01$).

Bi^{3+} in inorganic hosts absorbs light of wavelengths mainly in the 200–310 nm range and emits in the 330–450 nm range.

Further, the photoluminescence spectra of $\text{Ca}_3(\text{BO}_3)_2:\text{Bi}^{3+}$ with different Bi^{3+} doping concentrations were investigated. The variation of emission intensity of Bi^{3+} with different Bi^{3+} doping concentration is shown in Figure 5. It is observed that, with increasing Bi^{3+} doping concentration, the emission intensity of $\text{Ca}_3(\text{BO}_3)_2:\text{Bi}^{3+}$ increases and reaches a maximum at 0.003 mol. After this concentration, emission intensity decreases due to concentration quenching phenomenon. In this case, the energy transfer occurs from one activator to another until an energy sink in the lattice is reached. Finally, the Stokes shift of the synthesized phosphor was calculated to be 7086 cm^{-1} using the excitation band at 290 nm and the emission band at 365 nm.

3.5. Photoluminescence of $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$. The photoluminescence spectrum of Pb^{2+} doped in $\text{Ca}_3(\text{BO}_3)_2$ host material is shown in Figure 6. It can be described by the $^1\text{S}_0 \rightarrow ^3\text{P}_{0,1}$ transition, which originates from the $6s^2 \rightarrow 6s16p^2$

FIGURE 6: PL and PLE spectra of $(\text{Ca}_{1-x}\text{Pb}_x)_3(\text{BO}_3)_2$ ($x = 0.001, 0.003, 0.005, 0.01, 0.02, 0.03, 0.04$), excitation spectra monitored at 335 nm emission, and emission spectra monitored at 270 nm excitation.

interconfigurational transition. Typically at room temperature, emission is observed from the $^3\text{P}_1 \rightarrow ^1\text{S}_0$ transition [35], although at low temperatures the highly forbidden $^3\text{P}_0 \rightarrow ^1\text{S}_0$ emission is also observed [36]. The excitation band for the synthesized phosphor $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$ was observed at 270 nm, which is assigned to the $^1\text{S}_0 \rightarrow ^3\text{P}_1$ transition and the emission band was observed at 335 nm which can be ascribed to transition from $^3\text{P}_1$ excited state to the $^1\text{S}_0$ ground state. No splitting or multiple bands in the emission spectra for prepared phosphor are observed. Hence, we believed that the Pb^{2+} ions are incorporated at only one site (Ca^{2+} ion site) in the crystal lattice. In many inorganic hosts, the emission band of Pb^{2+} ion is in the UV region. It is also known that, in some hosts, Pb^{2+} ion emits in visible region. This diversity is depending strongly on the site occupied by Pb^{2+} ions, electronegativity of the ligand, crystal structure of the host lattice, and temperature [14, 37].

The luminescence intensities of Pb^{2+} doped phosphors always depend on the doped Pb^{2+} ions concentration [38–40]. Therefore, the photoluminescence spectra of $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$ with different Pb^{2+} doping concentrations were investigated. The variation of emission intensity of Pb^{2+} with different Pb^{2+} doping concentration is shown in Figure 7. It is observed that the position and shape of the photoluminescence bands have exhibited no obvious changes with Pb^{2+} concentration. The emission intensity of $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$ increases and reaches a maximum at 0.005 mol. The Stokes shift was calculated to be 7186 cm^{-1} using the excitation peak at 270 nm and the emission peak at 335 nm.

According to Blasse [41], for the critical concentration the average shortest distance between nearest activator ions

FIGURE 7: Variation in emission intensity of $(\text{Ca}_{1-x}\text{Pb}_x)_3(\text{BO}_3)_2$ ($x = 0.001, 0.003, 0.005, 0.01, 0.02, 0.03, 0.04$).

is equal to the critical transfer distance R_0 . R_0 is, in fact, the critical separation between donor (activator ion) and acceptor (quenching ion), at which the nonradiative rate equals that of the internal single ion relaxation. The R_0 value can be practically calculated using the following equation:

$$R_0 = 2 \times \left(\frac{3V}{4\pi\chi_c N} \right)^{1/3}, \quad (1)$$

where χ_c is the critical concentration, N the number of Ca^{2+} ions in the $\text{Ca}_3(\text{BO}_3)_2$ unit cell, and V the volume of the unit cell. By taking the values of $\chi_c = 0.003$ (for Bi^{3+}) and 0.005 (for Pb^{2+}), $N = 8$ and $V = 255.20\text{ \AA}^3$, respectively; the critical transfer distance R_0 for $\text{Ca}_3(\text{BO}_3)_2:\text{Bi}^{3+}$ and $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$ was measured to be about $27\text{ \AA}$ and $23\text{ \AA}$, respectively.

4. Conclusion

Two inorganic phosphors were prepared by a solution combustion synthesis method followed by heating of the precursor combustion ash at 800°C for 3 h in air. SEM images of the synthesized phosphors show irregular grains with agglomerate phenomena. Synthesized phosphors $\text{Ca}_3(\text{BO}_3)_2:\text{Bi}^{3+}$ and $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$ achieved the band emissions, respectively, at 365 nm and 335 nm corresponding to the transition $^3\text{P}_1 \rightarrow ^1\text{S}_0$. The optimum concentration of Bi^{3+} and Pb^{2+} in $\text{Ca}_3(\text{BO}_3)_2$ is measured to be 0.003 and 0.005 mol, respectively. For these values of optimum concentrations the critical transfer distance, R_0 , was measured to be about $27\text{ \AA}$ and $23\text{ \AA}$, respectively. Finally, the Stokes shifts of $\text{Ca}_3(\text{BO}_3)_2:\text{Bi}^{3+}$ and $\text{Ca}_3(\text{BO}_3)_2:\text{Pb}^{2+}$ were measured to be 7086 cm^{-1} and 7186 cm^{-1} , respectively. The emission bands of both the phosphors are in the UV region and the phosphors can be potential candidates for application in UV lamps.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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