

Photoluminescence properties of $\text{CaMgB}_2\text{O}_5: \text{RE}^{3+}$ (RE = Dy, Eu, Sm) environmental friendly phosphor for solid state lighting

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Received: 22 April 2023 / Accepted: 18 September 2023 / Published online: 18 October 2023

Abstract: This article reports the photoluminescence studies of $\text{CaMgB}_2\text{O}_5: \text{RE}^{3+}$ (RE = Dy, Eu, Sm) phosphors. The reliable, low cost combustion method is used to prepare a series of phosphors for Solid state lighting. In this work, environmental friendly Eu^{3+} doped CaMgB_2O_5 phosphor was first time synthesized by combustion method as the aim of our research is to produce and study the environment friendly phosphors for solid state lighting. The crystal structure of the prepared phosphor was studied by X-ray diffraction method and it almost closely matches with the standard JCPDS file No. 730618 showing the phase purity. The photoluminescence (PL) properties of prepared phosphors were broadly studied by excitation and emission spectra in this work. For the excitation wavelength of 352 nm, the emission spectra of $\text{CaMgB}_2\text{O}_5: \text{Dy}^{3+}$ phosphor showed two peaks observed at 483 nm and 576 nm corresponds to the well known ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{15/2}$ and ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{13/2}$ energy transitions of Dy^{3+} ion respectively. The photoluminescence spectra of $\text{CaMgB}_2\text{O}_5: \text{Eu}^{3+}$ phosphor revealed strong red emission at 614 nm (${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$) at n-UV excitation of 394 nm wavelength. The photoluminescence spectra of the $\text{CaMgB}_2\text{O}_5: \text{Sm}^{3+}$ phosphor exhibit strong orange-red emission at 600 nm wavelength (${}^4\text{G}_{5/2} \rightarrow {}^6\text{H}_{9/2}$) under the UV excitation of 405 nm wavelength. As a result of observed photoluminescence characteristics, the produced phosphors may be a revolutionary material for the solid state lighting applications point of view.

Keywords: Phosphors; Solid state lighting; Photoluminescence; Combustion method; Rare earth

1. Introduction

Lanthanide ions play an essential role in the white light emitting diode (wLED) technology because of their outstanding luminescence properties, which provide an intense sharp emission peaks due to the presence of several allowed energy levels [1–5]. Now a day's new phosphors excited by the rare earth ions have earmarked considerable attention in the fields of solid state lighting. Since, the important parameters for efficient phosphor required are the good luminescence efficiency, long durability, better stability and environmental friendliness, which can be achieved by doping of suitable lanthanide ions and therefore it attracts widespread attention in the field of solid state lighting [6–10]. Rare earth elements are commonly employed in the preparation of luminescent centers in

almost all the phosphors as well as some host materials to create multicolor lights for the solid state lighting.

Lanthanide ions doped inorganic phosphor materials have recently seen fast development and are being used in a variety of sectors such as light emitting diodes (LED's), cathode ray tube (CRT), fluorescent lights, optical devices, etc. [11–15]. That's why luminescence materials are extremely important in the current research progression. Therefore, the various lanthanide ions activated inorganic phosphors are good candidates for the development of ecofriendly and environment friendly solid state lighting materials [16–18]. Therefore, the proposed study will concentrate on the development of few lanthanides activated inorganic phosphors which may be excellent candidates for solid state lighting. Recently, researchers are working on a variety of phosphors including organic and inorganic compounds, hybrid compounds and others [19–26]. Because of their resilience, excellent luminous performance, high chemical and thermal stability and environmental friendliness, the lanthanides doped

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inorganic phosphors are among the most important compounds for the solid state lighting. Borate-based phosphor materials have outstanding luminous qualities as host matrix; because of their low synthesis temperature, ease of production, large energy band gap and excellent chemical and physical stability [27–32]. Therefore, in this recent work, a series of borate phosphors which are doped with different lanthanides have been synthesized for solid state lighting application. In this research work, we have studied the phase purity, morphology, photoluminescence properties and color coordinates of CaMgB_2O_5 : RE^{3+} (RE = Dy, Eu, Sm) phosphors synthesized by combustion method. Also, we have studied the effect of different concentration of Dy^{3+} , Eu^{3+} and Sm^{3+} doped CaMgB_2O_5 phosphors on relative luminescent intensities. In this recent work, we have synthesized CaMgB_2O_5 : Eu^{3+} which was first time reported by the combustion method. The main advantages of combustion method are rapid and straightforward processing (configuration results in the oxide mixture within a few seconds), inexpensive preliminary materials (usually nitrate salts), no need of extraordinary equipments and low energy utilization.

2. Experimental details

By using the conventional combustion method, the CaMgB_2O_5 : RE^{3+} (RE = Dy, Eu, Sm) phosphors were prepared, using urea as the combustion fuel. The $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, H_3BO_3 , $\text{CH}_4\text{N}_2\text{O}$, dysprosium oxide [Dy_2O_3], europium oxide [Eu_2O_3] and samarium oxide [Sm_2O_3] of analytical grade were used as starting materials. Initially the series of CaMgB_2O_5 : Dy^{3+} phosphors were prepared by varying the concentration of Dy^{3+} (i.e. $\text{Dy}^{3+} = 0.1$ mol%, $\text{Dy}^{3+} = 0.3$ mol%, $\text{Dy}^{3+} = 0.5$ mol%, $\text{Dy}^{3+} = 0.7$ mol% and $\text{Dy}^{3+} = 1$ mol%). All the starting materials were weighed by using the stoichiometric ratios for the different concentrations of dopants as mentioned above and then all the ingredients were mixed with the little deionized water in an agate mortar and pestle and then the mixture was vigorously crushed for 30 min to produce a thick paste. The crucible containing thick paste was then transferred into a high temperature muffle furnace pre heated at 550°C of temperature. Initially the mixture boils and undergoes dehydration followed by decomposition with the evolution of large amount of gases. The process is very highly exothermic due to which spontaneous ignition occurs producing white foamy voluminous ash. The ash was then collected, crushed it in agate mortar and pestle to made fine powder. Then this powder was utilized for further characterization like X-ray diffraction, scanning electron microscopy, photoluminescence study, etc. A similar combustion technique mentioned above was

used for the synthesis of series of CaMgB_2O_5 : Eu^{3+} [viz. (a) $\text{Eu}^{3+} = 0.3$ mol%, (b) $\text{Eu}^{3+} = 0.5$ mol%, (c) $\text{Eu}^{3+} = 0.7$ mol%, (d) $\text{Eu}^{3+} = 1$ mol% and (e) $\text{Eu}^{3+} = 1.2$ mol% of dopants concentration] phosphors and also the same method mentioned above was used to prepared a series of CaMgB_2O_5 : Sm^{3+} [viz. (a) $\text{Sm}^{3+} = 0.3$ mol%, (b) $\text{Sm}^{3+} = 0.5$ mol%, (c) $\text{Sm}^{3+} = 0.7$ mol%, (d) $\text{Sm}^{3+} = 1$ mol% and (e) $\text{Sm}^{3+} = 1.2$ mol%] phosphors. Further, by using combustion method and stoichiometric ratios of prerequisite, we have synthesized CaMgB_2O_5 host material. The produced phosphor was then characterized by using X-ray diffraction (XRD) technique for phase and purity, scanning electron microscopy (SEM) for surface morphology and photoluminescence (PL) measurements of all the lanthanides doped CaMgB_2O_5 phosphors for measuring excitation and emission wavelengths to incorporate it in the solid state lighting technologies such as white light emitting diode (wLED).

3. Results and discussion

3.1. X-ray diffraction (XRD) studies of the host CaMgB_2O_5

The prepared phosphor crystallinity and phase purity was examined using a powder x-ray diffraction (XRD) method. A CaMgB_2O_5 powder sample's x-ray diffraction (XRD) pattern synthesized by combustion technique at 550°C was shown in Fig. 1. When compared to the standard JCPDS file No. 730618, all the diffraction peaks in the x-ray diffraction (XRD) pattern of prepared CaMgB_2O_5 powder matches well with the standard JCPDS data with monoclinic phase structure with space group P21/b. This indicates that the prepared CaMgB_2O_5 phosphor formed with the exact phase and purity.

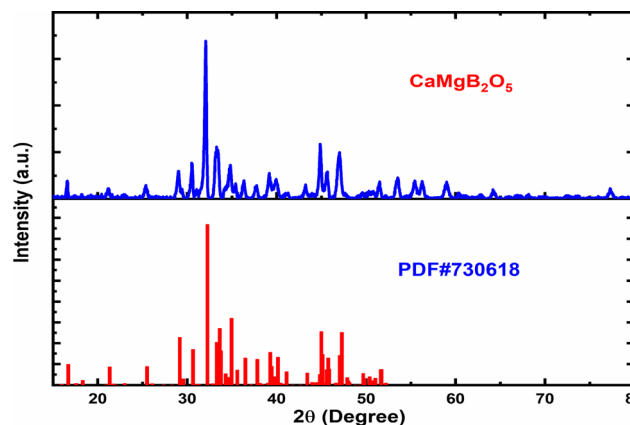


Fig. 1 X-ray diffraction pattern (XRD) of the host CaMgB_2O_5

3.2. Morphology of the host CaMgB₂O₅

Figure 2 shows the scanning electron microscope (SEM) images signifying the surface morphology of the host CaMgB₂O₅ powder sample. The prepared CaMgB₂O₅ powder has the grains in the order of micrometer in size and an irregular shape with a homogeneous composition [33]. Non-uniform and irregular shapes of the particles could be due to the non-uniform distribution of the temperature and mass flow in the combustion flame. The rare earth doped CaMgB₂O₅ phosphors can be utilized for solid state lighting applications since the average particle size of the host CaMgB₂O₅ is less than 10 μm.

3.3. Photoluminescence (PL) properties of CaMgB₂O₅: Dy³⁺ phosphors

The spectral properties of series of CaMgB₂O₅: Dy³⁺ phosphors with different concentrations were investigated by the excitation and emission spectra of photoluminescence (PL) at room temperature. The excitation spectrum of the Dy³⁺ activated CaMgB₂O₅ phosphor is shown in Fig. 3 while maintaining a constant emission at 483 nm of wavelength. The excitation spectrum was recorded in wavelength range of 300–400 nm. The characteristic peaks in the excitation spectrum are located at 326 nm, 352 nm, 367 nm and 389 nm wavelengths. The maximum excitation intensity was found at the wavelength of 352 nm [34]. Figure 4 shows the emission spectra of CaMgB₂O₅ phosphors doped with the different concentrations of the Dy³⁺ ions at 352 nm excitation wavelengths. The emission spectra consist of two characteristic bands located at

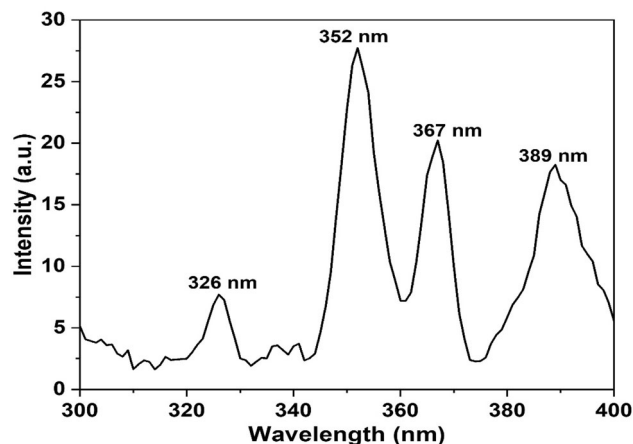


Fig. 3 Excitation spectrum of CaMgB₂O₅: Dy³⁺ phosphor

483 nm and 576 nm wavelengths. It also observed that the emission intensity is more for the band depicted at the 483 nm as compared to the 576 nm wavelength. The emission bands which are located at 483 nm and the other at 576 nm wavelength attributed to the $^4F_{9/2} \rightarrow ^6H_{15/2}$, $^4F_{9/2} \rightarrow ^6H_{13/2}$ transitions of Dy³⁺ ions respectively [35–37]. The blue emission (483 nm) is observed due to the electric dipole transition and yellow emission (576 nm) is ascribed to the magnetic dipole transition [38, 39]. The emission spectrum in the Fig. 4 revealed that the peak at 483 nm (blue color emission) wavelength indicates that Dy³⁺ ions are located at high-symmetry sites in the host matrix, whereas the peak at 576 nm (yellow color emission) wavelength indicates that Dy³⁺ ions are located at low-symmetry sites in the host matrix [38, 39]. In our

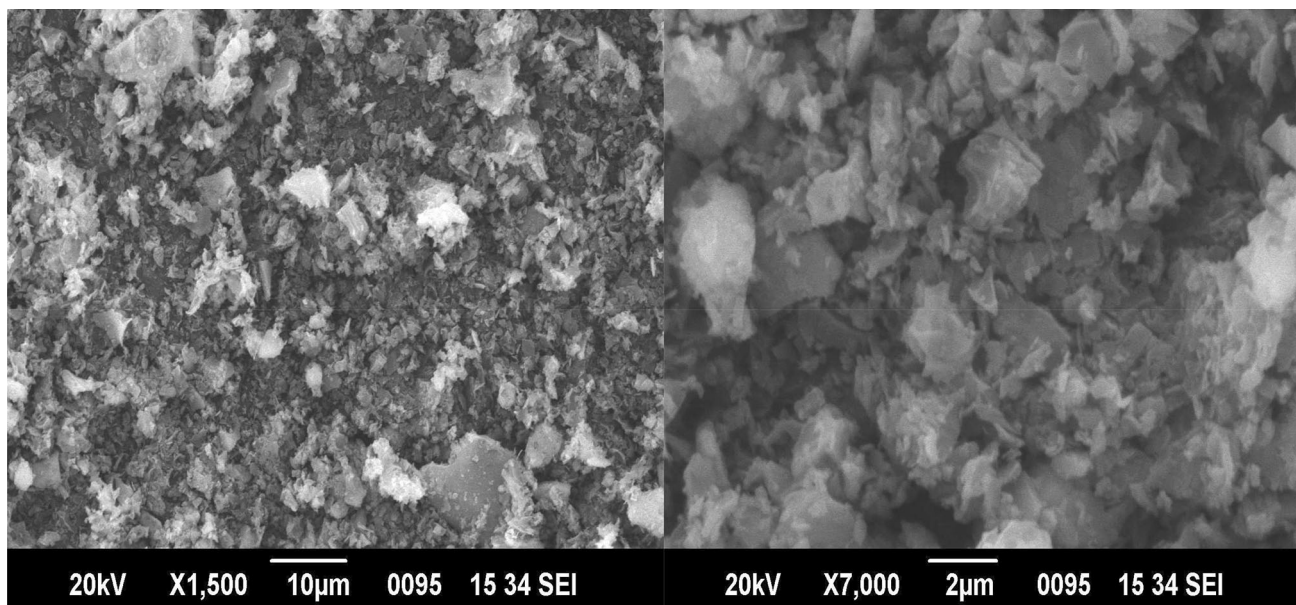


Fig. 2 Scanning electron microscope (SEM) images of the host CaMgB₂O₅

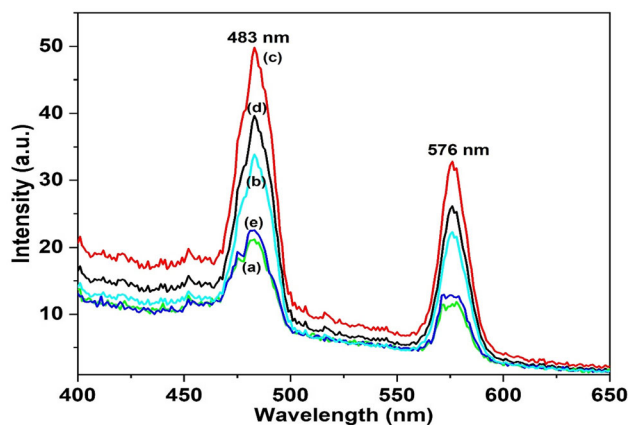


Fig. 4 Emission spectra of $\text{CaMgB}_2\text{O}_5:\text{Dy}^{3+}$ phosphor, where (a) $\text{Dy}^{3+} = 0.1$ mol%, (b) $\text{Dy}^{3+} = 0.3$ mol%, (c) $\text{Dy}^{3+} = 0.5$ mol%, (d) $\text{Dy}^{3+} = 0.7$ mol% and (e) $\text{Dy}^{3+} = 1$ mol%

investigation, the intensity of blue emission is the strongest, which shows that Dy^{3+} ions occupy high symmetry sites in the host matrix [40–42].

To determine the concentration quenching in prepared phosphors, we have prepared various Dy^{3+} doped CaMgB_2O_5 phosphors with dopants concentrations ranging from 0.1 to 1 mol%. The emission intensity was increased as we increased the Dy^{3+} ion concentration from 0.1 to 0.5 mol% as shown in the Fig. 5. The researchers reported that the increase in PL intensity is due to the fractional energy transfer from the excited ions to the other ions (cross-relaxation between rare earth ions). Consequently, with increasing concentrations of the rare earth (RE^{3+} , where $\text{RE} = \text{Eu}, \text{Dy}, \text{Sm}, \text{etc.}$) ions, the number of luminescence centers in the material increases and the corresponding emission intensity is eventually increased [43–46]. As a result of the concentration quenching effect, the emission intensity then gradually decreased after 0.5 mol%. The Fig. 5 revealed that the highest intensity of

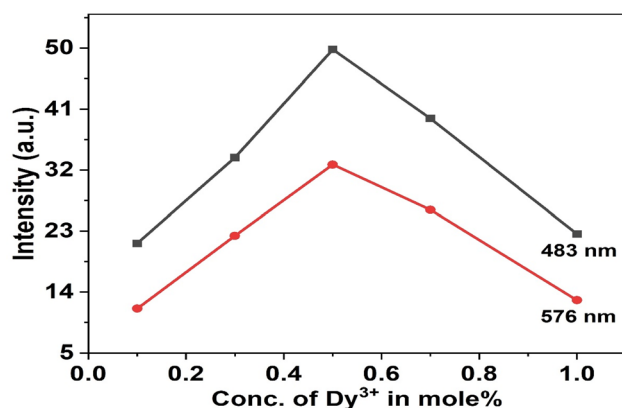


Fig. 5 Effect of concentration of Dy^{3+} doped CaMgB_2O_5 phosphor on relative luminescence intensity at 483 nm and 576 nm emission bands

emission for the studied phosphor was found to be at 0.5 mol of Dy^{3+} ions. The interaction between the rare earth ions grows as the concentration of rare earth increases, which must be the cause of self-quenching. Consequently, the emission intensity drops and concentration quenching is occurring in $\text{CaMgB}_2\text{O}_5:\text{Dy}^{3+}$ (0.5 mol%) phosphors [47–49]. The prepared phosphor may be a suitable candidate for the applications requiring environmental friendly solid state lighting, as per the observed PL results.

3.4. Photoluminescence (PL) properties of $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphors

Figure 6 depicts the PL excitation spectrum of $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphor. The excitation spectrum of $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphor was studied in the wavelength range of 320–440 nm. The excitation peaks were observed at 363 nm, 384 nm, 395 nm and 416 nm wavelengths, corresponding to the transitions ${}^7\text{F}_0 \rightarrow {}^5\text{D}_4$, ${}^7\text{F}_0 \rightarrow {}^5\text{G}_2$, ${}^7\text{F}_0 \rightarrow {}^5\text{L}_6$ and ${}^7\text{F}_0 \rightarrow {}^5\text{D}_3$ of Eu^{3+} ions respectively [50]. As a consequence, the $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphor was shown to be an excellent candidate for use in n-UV stimulating solid state lighting. The emission spectra of $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphors were shown in Fig. 7 for the wavelength ranges from 550 to 700 nm after n-UV excitation of the wavelength of 395 nm. It was observed that the emission peaks located at 593 and 614 nm wavelengths, corresponding to the ${}^5\text{D}_0 \rightarrow {}^7\text{F}_1$ and ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$ transitions of the Eu^{3+} ions respectively [51]. For the study of concentration quenching effect, the Eu^{3+} ions concentration was changed from 0.3 to 1.2 mol% in the CaMgB_2O_5 host. The Fig. 8 depicts the effect of concentration of Eu^{3+} doped CaMgB_2O_5 phosphor on relative luminescence intensity at 614 nm emission wavelength. The emission spectra also revealed that due to the increase

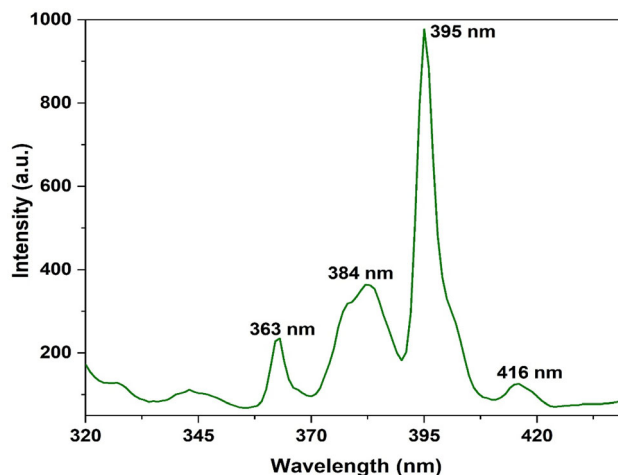


Fig. 6 Excitation spectrum of $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphor

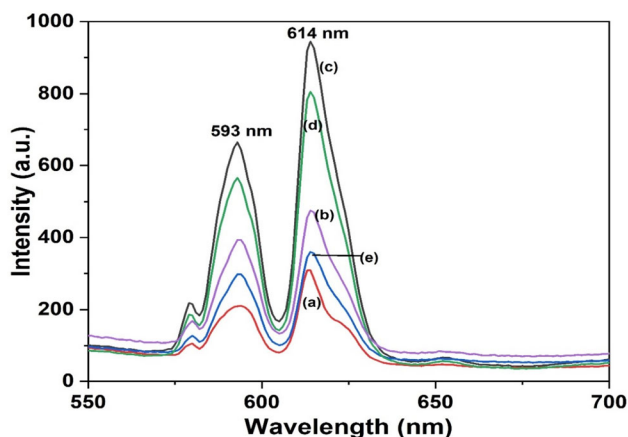


Fig. 7 Emission spectra of $\text{CaMgB}_2\text{O}_5: \text{Eu}^{3+}$ phosphor, where (a) $\text{Eu}^{3+} = 0.3$ mol%, (b) $\text{Eu}^{3+} = 0.5$ mol%, (c) $\text{Eu}^{3+} = 0.7$ mol%, (d) $\text{Eu}^{3+} = 1$ mol%, and (e) $\text{Eu}^{3+} = 1.2$ mol%

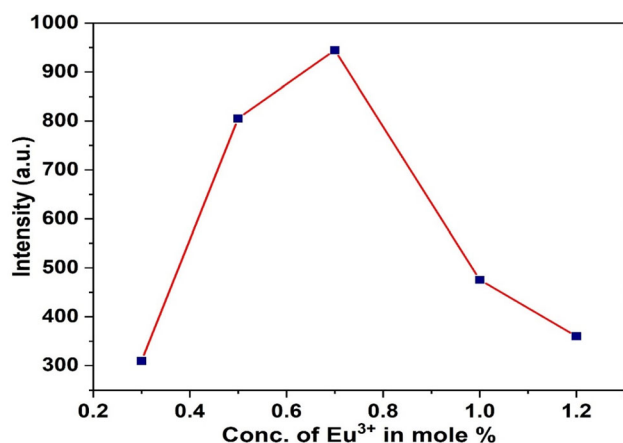


Fig. 8 Effect of concentration of Eu^{3+} doped CaMgB_2O_5 phosphor on relative luminescence intensity at 614 nm emission band

in Eu^{3+} ion concentration the PL intensity increases up to 0.7 mol% and then it is decreases due to concentration quenching effect [52].

3.5. Photoluminescence (PL) properties of $\text{CaMgB}_2\text{O}_5: \text{Sm}^{3+}$ phosphors

Figure 9 depicts the excitation spectrum recorded for $\text{CaMgB}_2\text{O}_5: \text{Sm}^{3+}$ phosphor. While monitoring the excitation spectrum, the emission at 600 nm wavelength showed excitation peaks located at 345 nm, 362 nm, 376 nm and 405 nm wavelengths, which correspond to the Sm^{3+} ions transitions from the ground state $^6\text{H}_{5/2}$ to $^4\text{H}_{9/2}$, $^4\text{D}_{3/2}$, $^4\text{D}_{1/2}$, and $^6\text{F}_{7/2}$ states, respectively [53]. The emission spectra of $\text{CaMgB}_2\text{O}_5: \text{Sm}^{3+}$ phosphor shown in Fig. 10 which was recorded at 405 nm excitation wavelengths of Sm^{3+} ions. The distinctive emission spectra located at 564 nm, 600 nm and 647 nm wavelengths are

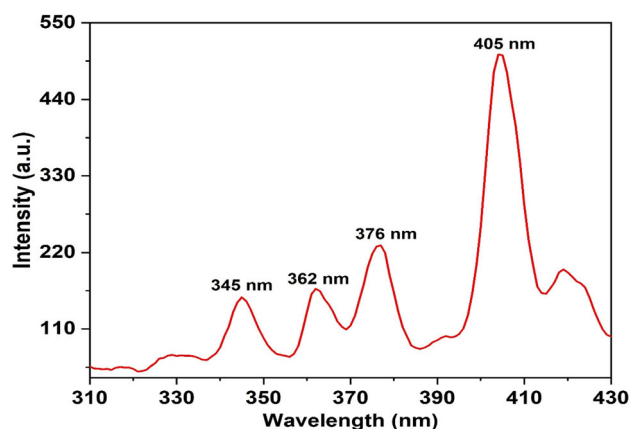


Fig. 9 Excitation spectrum of $\text{CaMgB}_2\text{O}_5: \text{Sm}^{3+}$ phosphor

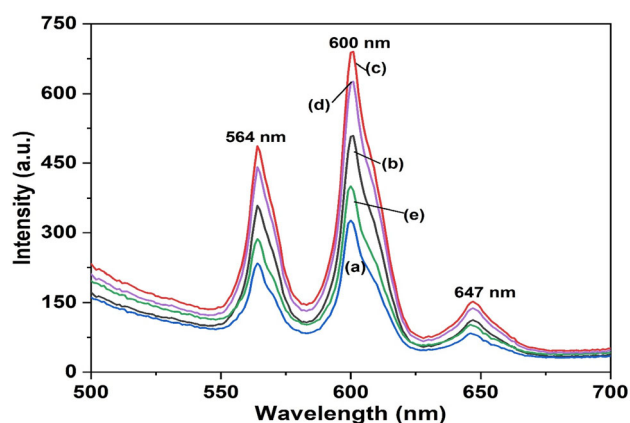


Fig. 10 Emission spectra of $\text{CaMgB}_2\text{O}_5: \text{Sm}^{3+}$ phosphor, where (a) $\text{Sm}^{3+} = 0.3$ mol%, (b) $\text{Sm}^{3+} = 0.5$ mol%, (c) $\text{Sm}^{3+} = 0.7$ mol%, (d) $\text{Sm}^{3+} = 1$ mol% and (e) $\text{Sm}^{3+} = 1.2$ mol%

ascribed to the transitions from the excited state $^4\text{G}_{5/2}$ to the $^6\text{H}_J$ ($J = 5/2, 7/2$ and $9/2$) ground state [54, 55]. The luminescence characteristics of $\text{CaMgB}_2\text{O}_5: \text{Sm}^{3+}$ phosphors were studied using various concentrations of Sm^{3+}

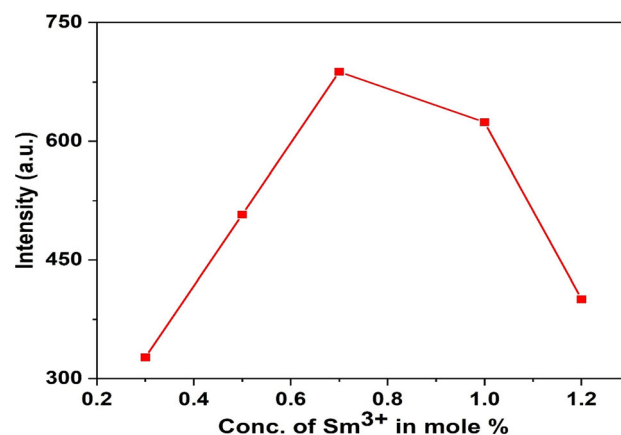


Fig. 11 Effect of concentration of Sm^{3+} doped CaMgB_2O_5 phosphor on relative luminescent intensity at 600 nm emission band

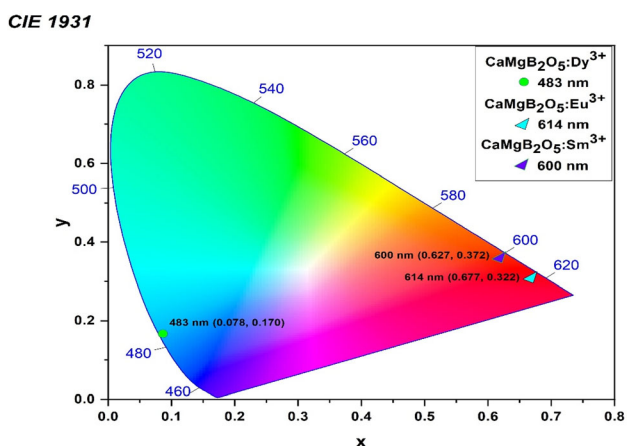


Fig. 12 CIE chromatic diagram of $\text{CaMgB}_2\text{O}_5:\text{RE}^{3+}$ ($\text{RE} = \text{Dy}, \text{Eu}, \text{Sm}$) phosphor

ions ranging from 0.3 to 1.2 mol%. As demonstrated in Fig. 11, the PL intensity increased initially with Sm^{3+} ion substitution up to 0.7 mol% and thereafter decreased due to concentration quenching [56].

3.6. Color chromaticity coordinates of $\text{CaMgB}_2\text{O}_5:\text{RE}^{3+}$ ($\text{RE} = \text{Dy}, \text{Eu}, \text{Sm}$) phosphors

Figure 12 depicted the Commission Internationale de l'Éclairage (CIE) 1931 chromaticity coordinates diagram plotted for $\text{CaMgB}_2\text{O}_5:\text{RE}^{3+}$ ($\text{RE} = \text{Dy}, \text{Eu}, \text{Sm}$) phosphors. The CIE coordinates of the $\text{CaMgB}_2\text{O}_5:\text{Dy}^{3+}$ phosphor are $x = 0.078$ and $y = 0.170$ which represents the blue color. The CIE coordinates of the $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphor are $x = 0.677$ and $y = 0.322$ indicating the red color [56, 57]. The CIE coordinates of the $\text{CaMgB}_2\text{O}_5:\text{Sm}^{3+}$ phosphor are $x = 0.627$ and $y = 0.372$ indicative of the orange-red color. The finding suggested that the measured color coordinates are close to the edge of the CIE diagram indicates that the prepared phosphors have great color purity [58–61]. As a result, $\text{CaMgB}_2\text{O}_5:\text{RE}^{3+}$ ($\text{RE} = \text{Eu}, \text{Dy}, \text{Sm}$) phosphors may be a promising candidates for the solid state lighting applications.

4. Conclusions

The series of $\text{CaMgB}_2\text{O}_5:\text{RE}^{3+}$ ($\text{RE} = \text{Dy}, \text{Eu}, \text{Sm}$) phosphors were successfully prepared by the simple, less time consuming and promising combustion technique. The phase purity, surface morphology, photoluminescence and optical properties were investigated in details. The XRD analysis verified that the prepared phosphors were formed with the exact phase and purity. The PL study revealed that for $\text{CaMgB}_2\text{O}_5:\text{Dy}^{3+}$ phosphor at 352 nm excitation

wavelengths, the emission occurs at 483 nm and 576 nm wavelengths corresponding to blue and yellow colors. The $\text{CaMgB}_2\text{O}_5:\text{Eu}^{3+}$ phosphors samples exhibit strong red emission at 614 nm wavelength under the n-UV excitation at 394 nm. The $\text{CaMgB}_2\text{O}_5:\text{Sm}^{3+}$ phosphor sample shows the strong orange-red emission at 600 nm upon UV excitation of 405 nm wavelength. The concentration quenching was observed for all the prepared rare earth doped phosphors. According to the PL measurements of prepared phosphors, the excitation peaks were located away from the Hg excitation, which showed that its environment friendly approach. In view of this, we have recommended that the $\text{CaMgB}_2\text{O}_5:\text{RE}^{3+}$ ($\text{RE} = \text{Dy}, \text{Eu}, \text{Sm}$) phosphors might be used for the environmental friendly state lighting (SSL) applications.

Data availability Data will be made available on request.

Declarations

Conflicts of interest The authors declare that they do not have any known competing financial interests or personal relationships that could appear to have influenced the work reported in this paper.

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