



Enhancement of photoluminescence in Eu^{3+} co-activated $\text{Ca}_2\text{MgSi}_2\text{O}_7:\text{Dy}^{3+}$ phosphors for solid state lighting application

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ABSTRACT

A series of $\text{Ca}_2\text{MgSi}_2\text{O}_7:\text{Dy}^{3+}$, $\text{Ca}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$ and $\text{Ca}_2\text{MgSi}_2\text{O}_7:\text{Dy}^{3+}, \text{Eu}^{3+}$ phosphors were synthesized by conventional solid state reaction method. The X-ray diffraction (XRD) patterns were recorded to confirm the phase purity of $\text{Ca}_2\text{MgSi}_2\text{O}_7$ phosphors. The XRD results showed that the prepared phosphors have single-phase tetragonal structure and incorporation of dopants has no effect on the crystal structure of phosphors. The surface morphological studies were performed using field emission scanning electronic spectroscopy (FESEM). Photoluminescence (PL) properties were systematically investigated in detail. With increasing, Dy^{3+} concentration the PL intensity first increases, reaches the maximum and then it was decreased. A similar trend was shown in $\text{Ca}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{3+}$ phosphors. The PL emission intensity was increased at the transitions ${}^5\text{D}_0 \rightarrow {}^7\text{F}_1$ and ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$ with increasing Eu^{3+} concentration while decreased the intensity at the transitions ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{15/2}$ and ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{13/2}$. The calculated band gap values of the sample were found to be 4.29, 4.38, 4.11 and 4.23 eV.

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1. Introduction

In the current scenario, the development of luminescent materials has been the subject of extensive research. Especially interesting in inorganic compound have been extensively used a variety of potential applications such as cathode ray tubes (CRTs), lamp industries, field emission display (FEDs), radiation dosimetry and white light emitting diodes (WLEDs) [1,2]. Solid state light using white light emitting diode becomes a great worldwide strategy for energy saving, reliability, safety (lack of toxic mercury), environmental friendliness, high brightness, fast switching, color quality and less complex characteristics, which promises a more luminescence efficiency than of incandescent lighting and a real chance of replacing conventional lighting sources such as fluorescent and incandescent lamp [3–6].

When light of sufficient energy is incident on a material, photons are absorbed and electronic excitations are created. Accordingly, these excitations relax and the electrons back to the ground state. If the radiative relaxations are occurring, then the emitted light is called photoluminescence (PL) [7].

A more feasible approach to find such a material that can

produce white light emitting diode via the combination of different color emission from an identical luminescent centre [8]. Presently, a lot of research works have been done to develop a single-phased white light emitting phosphors based on the mechanisms of energy transfer from sensitizer to activator, such as $\text{Tb}^{3+}/\text{Sm}^{3+}$, $\text{Ce}^{3+}/\text{Tb}^{3+}$, $\text{Tm}^{3+}/\text{Dy}^{3+}$, $\text{Dy}^{3+}/\text{Tb}^{3+}$ for white light emission application [9–12]. Analysis of Dy^{3+} ions have been drawn significant interest owing to its white emission. Dy^{3+} having configuration $4f^9$ has complex f-block energy levels. Therefore, several transitions are possible between these levels and show sharp line spectra. Generally Dy^{3+} exhibits a strong blue emission band in the 470–500 nm range due to ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{15/2}$ transition and a yellow emission band in the 570–600 nm range due to ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{13/2}$ transition. However, the ratio of two dominant emission band arising from transitions ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{15/2}$ (blue) and ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{13/2}$ (yellow) is different in different hosts. The emission intensity of the Dy^{3+} doped singly phosphor is poor under the UV excitation due to the forbidden f-f transition of Dy^{3+} ions and low absorption cross-section ($0.01\text{--}1\text{ cm}^{-1}$). However, the quality of white light emission of singly Dy^{3+} doped phosphor is poor due to lack of red light component. This is so essential that a red emitting material should be codoped in Dy^{3+} doped phosphor. Eu^{3+} is an operable activator ion for many different host materials for achieving rich red emission and it was also shown that the energy transfer from Dy^{3+} ion to Eu^{3+} ion was efficient, so the color controllable emissions were

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expected to be obtained in $\text{Dy}^{3+}/\text{Eu}^{3+}$ co-doped materials [13]. In order to recompense the red component of different concentration of Eu^{3+} ions were pierced to the single doped Dy^{3+} because of Eu^{3+} ions provided orange and red emission, which are essential for achieving artificial white light emission [14].

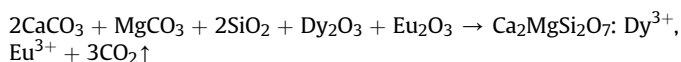
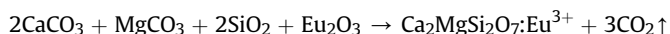
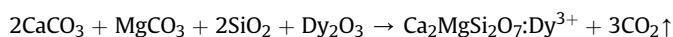
Various types of phosphors such as oxides, sulphides, aluminates, nitrides, phosphates etc. are used in LEDs. However, it is difficult to couple oxide based phosphors with blue LED due to their low absorption in the visible light spectrum [15]. Generally, silicate group phosphor has received great attention because of their brilliant luminescent properties such as high structure, low cost, easy preparation, excellent water resistance, and strong absorption in near – UV region, physical and chemical stability [16,17]. $\text{Ca}_2\text{MgSi}_2\text{O}_7$ (CMS) is an alkaline earth silicate belongs to melilites groups called sorosilicates which can be represented by general formula $\text{A}_2\text{MT}_2\text{O}_7$ (A = Ca, Sr, Na; M = Mg, Zn; T = Si, Al, B). They generally crystalize in tetragonal structure. Owing to their tetragonal structure without an inversion symmetry the lanthanide and transition metal are easily incorporated as an activator in the melilite host. Due to this comfort of incorporation, melilites group are recently widely used as a host material for rare earth dopant and transition metal ion to prepare phosphors which can be used in many potential application such as light emitting diodes, flat panel display, solar cell and many other variety of light emitting devices [18]. RE doped silicate phosphors have a considerable candidate for various applications such as display devices, scintillation, detector systems and LEDs [19,20]. Herein, a variety of CMS: Dy, Eu phosphors have been developed as a distinguishing material used in different optical application fields. Q. Fei et al. reported luminescence properties of $\text{Ca}_2\text{MgSi}_2\text{O}_7$ long lasting phosphors activated by Eu^{2+} , Dy^{3+} [21]. T. Aitasalo et al. reported persistent luminescence and synchrotron radiation study of the $\text{Ca}_2\text{MgSi}_2\text{O}_7$: Eu^{2+} , R^{3+} materials [22]. H. Zhang et al. reported Detection of stress distribution using $\text{Ca}_2\text{MgSi}_2\text{O}_7$: Eu, Dymicroparticles [23].

To the best of our knowledge, until, no reports found on the detail luminescent properties of Dy^{3+} , Eu^{3+} codoped $\text{Ca}_2\text{MgSi}_2\text{O}_7$ (CMS) phosphors synthesized by the solid state reaction method. In this work, the structural and luminescent properties of singly Dy^{3+} and Eu^{3+} doped and also codoped Dy^{3+} , Eu^{3+} of CMS phosphors by varying concentrations were systematically investigated and also energy transfers between Dy^{3+} and Eu^{3+} ions is also discussed. The white light emission is observed by adjusting Eu^{3+} concentration in CMS: Dy^{3+} phosphor. The present prepared CMS: Dy^{3+} , Eu^{3+} phosphors is used as a potential application for WLEDs.

2. Experimental section

2.1. Phosphors preparation

Samples CMS: $x\text{Dy}^{3+}$ ($x = 0.5\text{--}2$ mol%), CMS: $y\text{Eu}^{3+}$ ($y = 1\text{--}6$ mol%) and CMS: $x\text{Dy}^{3+}$, $y\text{Eu}^{3+}$ ($x = 1\%$, $y = 1\text{--}3\%$) were synthesized by conventional solid state reaction technique. The starting materials were calcium carbonate (CaCO_3 99.90%), magnesium carbonate (MgCO_3 99.90%), silicon dioxide (SiO_2 99.99%), dysprosium oxide (Dy_2O_3 99.99%) and europium oxide (Eu_2O_3 99.99%). All of the chemicals were analytical grade (A.R.) employed in this experiment. Initially the raw materials were weighed accordingly nominal composition of $\text{Ca}_2\text{MgSi}_2\text{O}_7$: Dy^{3+} , $\text{Ca}_2\text{MgSi}_2\text{O}_7$: Eu^{3+} and $\text{Ca}_2\text{MgSi}_2\text{O}_7$: Dy^{3+} , Eu^{3+} phosphors after that the powders were mixed and milled thoroughly for 2 h using agate mortar and pestle. The chemical reaction which used for stoichiometric calculation is:



The obtained samples were placed in 30 ml alumina crucible and fired at 1200°C for 3 h in air. At last the final products obtained after the cooling down of programmable furnace.

2.2. Characterization techniques

The phase purity and structural properties of undoped, single doped and codoped of CMS phosphors were checked by using XRD diffractometer recorded in the range of Bragg's angle 2θ ($10^\circ \leq 2\theta \leq 80^\circ$) using a Bruker D8 Focus X-ray diffraction (XRD) with target Cu-K α radiation. The surface morphology of the prepared phosphors was examined by using field emission scanning electron microscopy (FESEM) studied were carried out with the help of 55- Supra (Germany) FESEM measuring instrument. The photoluminescence studies were carried out using Hitachi Fluorescence spectrometer F-2500 with a 150 W Xe lamp in the wavelength range of 220–800 nm while the Ultra Violet Visible (UV Vis) of prepared samples were done using CARRY – 5000 spectrophotometer. The decay kinetics was studied using Agilent Technologies Cary Eclipse Fluorescence Spectrophotometer.

3. XRD studies

The phase purity and crystal structure of prepared samples are observed by XRD. Fig. 1 shows the XRD pattern of undoped, Dy^{3+} , Eu^{3+} doped and Dy^{3+} , Eu^{3+} co-doped of prepared $\text{Ca}_2\text{MgSi}_2\text{O}_7$ (CMS) phosphors. The XRD peak of undoped CMS phosphors was well indexed with the JCPDS file no. 79-2425. The XRD patterns shows an impurity peak about $2\theta = 33.52$ due to untreated MgCO_3 used as starting material. No other impurity peaks are detected, implying that the doping contents and the doping ions Dy^{3+} , Eu^{3+} are incorporated in the host lattice. The phase structure of undoped, single doped and codoped CMS phosphors is akermanite structure which belongs to tetragonal shape, having space group $P4_21m$ (113) and the lattice constants $a = b = 7.828 \text{ \AA}$, $c = 5.005 \text{ \AA}$, $\alpha = \beta = \gamma = 90^\circ$ (JCPDS 79-2425). The approximate atomic radius of Dy^{3+} (0.0912 nm) and Eu^{3+} (0.095 nm) are nearer to that of Ca^{2+} (0.099), while much larger than those of Mg^{2+} (0.072 nm) and Si^{4+} (0.040 nm). Therefore, the possibility of Dy^{3+} and Eu^{3+} ions

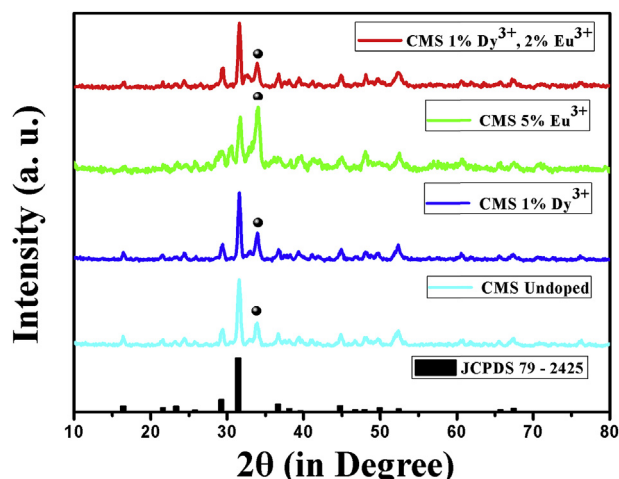


Fig. 1. The XRD pattern of the representative samples.

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