Fabrication of Phosphor Converted White LED for Solid State Lighting Application

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Abstract
Solid state lighting (SSL) refers to a type of lighting that utilizes light-emitting diodes (LEDs) as sources of illumination rather than electrical filaments or gas. It is emerging as a promising option for domestic lighting due to several advantages like energy saving, high luminous efficiency, environment-friendliness, small volume, and long persistence. The combination of Blue LED and yellow phosphor is considered as the least expensive method for producing white light.

The yellow emitting phosphor Ca2BO3Cl:Eu2+ is prepared by conventional solid state reaction method in reductive atmosphere produced by half burned charcoal at 1173k. The powder XRD pattern is in good agreement with JCPDS card no.29-0302.

The Photoluminescence excitation spectrum monitored at 560 nm shows broad excitation band from 300 nm to 500 nm peaking at 380 nm. The excitation band shows excellent spread in both nUV and blue region of the spectrum. The phosphor shows emission wavelength of 560 nm and CIE 1931 colour coordinates as (0.41, 0.52).

The as-synthesized phosphor is then crushed and sieved properly in order to get fine powder. The phosphor in desired quantity was dispersed in a transparent silicone resin and white LED was fabricated by coating the blue LED chip with this epoxy resin. The CIE 1931 color coordinates of the fabricated white LED was observed as (0.36, 0.35) for optimum epoxy to phosphor ratio. Further CRI was improved by adding Red phosphor.

Key words: Luminescence, phosphor, solid state lighting

1. Introduction
Solid state lighting (SSL) refers to a type of lighting that utilizes light-emitting diodes (LEDs) as sources of illumination rather than electrical filaments or gas. SSL creates visible light with virtually no heat or parasitic energy dissipation. In addition, its solid-state nature provides for greater resistance to shock, vibration, and wear, thereby increasing its lifespan significantly [1–3].

SSL technology advances with the invention of blue emitting InGaN-based LED by S. Nakamura in 1991[4]. With years the scientists were able to produce more and more efficient blue LEDs[5-6], which became the basis for white LEDs [7-8]. At the same time the development of new phosphors for white LED applications is highly desirable. Researchers in this field are exploring with new hosts and dopants along with simplification in synthesis methods that can lead to cost reduction [9-13].

Blue LED and yellow phosphor is considered the least expensive method for producing white light. Blue light from an LED is used to excite a phosphor which then re-emits yellow light. This balanced mixing of yellow and blue lights results in the appearance of white light.

In this paper we are reporting phosphor converted white LED fabrication by coating yellow emitting phosphor Ca2BO3Cl:0.04Eu2+ on blue LED chip. The crystal structure of Ca2BO3Cl was first reported by J. Majling [14] et al. and after that several researchers tried to develop this phosphor in various aspects[15-18] so as to emerge it as a potential candidate to replace YAG:Ce phosphor.
2. Experimental

The yellow emitting phosphor Ca$_2$BO$_3$Cl:Eu$^{2+}$ is prepared by conventional solid state reaction method in slightly reductive atmosphere. The starting materials were stoichiometric CaCO$_3$ (A.R.), CaCl$_2$ (A.R.), H$_3$BO$_3$ (A.R.) and Eu$_2$O$_3$ (99.99%). The raw materials were mixed thoroughly in an agate mortar, then sintered at 1173K for 3 hours in reducing atmosphere. The obtained product is further ground thoroughly to produce fine powder.

The phosphor powder obtained was then subjected to XRD analysis using Rigaku mini flex automatic diffractometer with Cu Kα radiation (λ=1.540598 Å) operated with scan speed of 4.000 °/min. The XRD data was collected in a 2θ range from 10 ° to 70 ° at room temperature.

The measurements of photoluminescence (PL) over the range of 400–700 nm and photoluminescence excitation spectra (PLE) over 200–550 nm excitation range were carried out on Hitachi-F7000 fluorescence spectrophotometer at room temperature. The colour chromaticity coordinates were obtained according to Commission International de l’Eclairage (CIE) using Radiant Imaging colour calculator.

3. Result and discussion

3.1 XRD analysis

The powder XRD pattern is in good agreement with JCPDS card no.29-0302 indicating monoclinic crystal structure with P21/c space group. Fig 1 represents the XRD patterns of the as-synthesized Ca$_2$BO$_3$Cl:xEu$^{2+}$ sample. The reported lattice constants are a=3.948 (Å), b=8.692, (Å), c=12.4 (Å). The cell volume is V=418.8 (Å$^3$), with number of anions and cations in the unit cell (Z) are 4 and β=100.27(1)$^\circ$.

![JCPDS29-0302](image1)

**Fig.1.** The powder XRD pattern of as-synthesised Ca$_2$BO$_3$Cl:0.04Eu$^{2+}$ sample

3.2 Photoluminescence

In Ca$_2$BO$_3$Cl:host Eu$^{2+}$ shows yellow emission Fig 2 shows the PLE (a) and PL (b) curve for Ca$_2$BO$_3$Cl:xEu$^{2+}$ for x=0.04. The Photoluminescence excitation spectrum monitored at 560 nm shows broad excitation band from 300 nm to 500 nm peaking at 380 nm. There are several small peaks superimposed. The excitation band shows excellent spread in both nUV and blue region of the spectrum. This makes it potential candidate for LED application.

The Emission spectrum monitored at 380 nm as well as 450 nm shows emission wavelength of 560 nm corresponding to 4f$^6$5d$^1$$\rightarrow$4f$^7$ transition of Eu$^{3+}$ ion.
Fig. 2. PLE (a) and PL (b) curve for Ca$_2$BO$_3$Cl:xEu$^{2+}$ for x=0.04

The maximum intensity of emission was observed at x=0.04 in Ca$_2$BO$_3$Cl:xEu$^{2+}$ after that intensity drops due to internal concentration quenching because of quadrupole–quadrupole interaction. The phosphor shows CIE 1931 colour coordinates as (0.41, 0.52) indicating yellow colour emission.

3.3 White LED fabrication

The prepared phosphor is crushed and sieved properly in order to get fine powder. The phosphor in desired quantity was dispersed in a transparent silicone resin and white LED was then fabricated by coating the blue LED chip with this epoxy resin.

Fig. 3. Emission spectra of LED for various phosphor concentrations.

Fig. 3 shows emission spectra of LEDs for various phosphor concentrations. Phosphor concentration (wt % of epoxy resin) for LED1 (3%), for LED 2 (6%), for LED3 (9%) and for LED4 (12%). The required amount of blue light is converted to yellow for phosphor concentration of 12%. However the LEDs fabricated showed color coordinates to the cool white side due to the lack of red emission components. The improvement in CRI value was observed by adding as-synthesized red phosphor K$_2$SiF$_6$:Mn$^{4+}$ along with the yellow emitting Ca$_2$BO$_3$Cl:xEu$^{2+}$phosphor. K$_2$SiF$_6$:Mn$^{4+}$ phosphor was synthesized by the method suggested by Chenxing Liao et-al.[19].

By varying the concentration of the phosphor in the resin color temperature, CIE chromaticity coordinates, CRI, lumen output, all these parameters could be changed. Typical testing report for the LED fabricated is enclosed. It shows CIE colour coordinates as (0.27, 0.28).
### LED Integrated Testing System test report

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Curve of spectrum power distribution

![Spectrum Parameters](image)

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<td>(\lambda_{Ep}^c)</td>
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<tr>
<td>(\lambda_{Em}^c)</td>
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<td>(\lambda_{Ek}^c)</td>
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<tr>
<td>(\tau_{Em}^c)</td>
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<tr>
<td>(V_{f}^c)</td>
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<td>(E_{f}^c)</td>
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Fig.4. Testing Report For Blue LED chip coated with Ca$_2$BO$_3$Cl:xEu$^{3+}$ (12%) + K$_2$SiF$_6$:Mn$^{4+}$ (1.5%)  

#### References: