$BA_3CAK(PO_4)_3:EU^{2+}$ PHOSPHOR TO ENHANCE GREEN LIGHT FOR WHITE LED APPLICATIONS

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Abstract. The $Ba_3CaK(PO_4)_3:Eu^{2+}$ (Eu-BCKPO) is synthesized with the high-temperature solid-state reacting route. The as-synthesized phosphor compound can result in cool white light with high quantum yield due to the site substitution of the Eu^{2+}. To examine the effect of Eu-BCKPO phosphor on the green emission range and the total luminous flux of the conventional LED, different amounts of Eu-BCKPO are combined with blue chip (465 nm) and YAG:Ce^{3+} yellow phosphor. The increasing amount of Eu-BCKPO phosphor supports the stability of color temperature and improvement of luminosity of the examined LED. The green wavelength region in the total LED spectrum also increases with higher Eu-BCKPO doping amounts. However, the color rendering efficiency of LED white light goes downward as the concentration of Eu-BCKPO phosphor increases owing to the insufficient of the red component. Thus, the Eu-BCKPO can be utilized in green-LED fabrication or combine with blue and red luminescent materials for forming the multicolor/white LED devices.

Keywords. $Ba_3CaK(PO_4)_3:Eu^{2+}$, Color temperature, Light-emitting diodes, Luminescent materials, YAG:Ce^{3+}

1 INTRODUCTION

The light-emitting diodes (LEDs) have been recognized significantly in the modern days owing to theirs outstanding features, for example, being energy-saving and eco-friendly nature while providing faster response and longer-term service than halogen and incandescent illuminating sources [1, 2]. Furthermore, the growth of multicolor LEDs has been the imperative factor of stimulating the development of multi-function illumination in various fields, including display, biomedical, agricultural, and cosmetic aspects [3, 4]. In comparison to other LED fabrication methods, using the phosphor materials to convert light from the chip is the simpler and cost-efficiency method [5]. Though the conventional incorporation of YAG:Ce^{3+} yellow phosphor and blue chips can offer high luminescence, the aging process of the phosphor is relatively fast due to the low thermostability, leading to the shorter serving time of the package [6]. In addition, such a YAG:Ce-converting LED package suffers from poor color rendition efficacy as the essential red light amount is shortage. Besides, the development of blue/red/green emitters are crucial for multicolor LED devices to realize quality white light. The red phosphors have been well researched with many potential materials for enhancing the chromatic rendition of the LED. However, the LEDs also suffer from the "green gap" issues, in which the luminescence efficiency is reduced from blue towards the green/orange spectral band, making the green-light LED exhibits the lower efficiency than the red or blue-light LEDs [7]. Thus, it is essential to produce a phosphor material to promote the green luminescence with high thermostability to overcome this problem, supporting the development of multicolor/white LEDs [8, 9].

The rare-earth ions are widely used as dopants for phosphor composition to achieve the requested emission colors. The Eu^{2+} ion is one of the most investigated ionic dopants as its 4f-5d transition enables a broad-band emission from near-ultraviolet to red wavelength ranges, depending on the crystalline strength of the cation sites it replaces in the host lattice. Therefore, the host with various cation sites for the Eu^{2+} entering is a suitable composition. The phosphate host with the structure of β-$Ca_3(PO_4)_2$ is a potential candidate owing to its flexible structure including five Ca^{2+} sites, allowing more compositions to be formed when different doping different ions [10]. It is possible to tune the emission color of the Eu^{2+} when it is integrated into the of β-$Ca_3(PO_4)_2$ phosphate compositions and then occupying different cation sites in the host composition. The $Ba_3CaK(PO_4)_3$ (BCKPO) is a new composition of β-$Ca_3(PO_4)_2$-type phosphate structure. It is formed from the replacement of two K^+ ions and six Ba^{2+} ions to one Ca^{2+} and six Ca^{2+} sites in the structure, respectively. The Eu^{2+}-doped $Ba_3CaK(PO_4)_3$ (Eu-BCKPO) phosphor composition is barely

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investigated and reported for conventional LEDs. This paper's purpose is to examine the Eu-BCKPO phosphor emission spectra and the phosphor influences on the commercial LED package. The thermostability of the Eu-BCKPO luminescence is also calculated. The Eu-BCKPO phosphor shows the potential in enhancing the green emission in the light spectrum as well as helps the LED achieve noticeable luminous enhancement [11, 12].

2 METHOD

The ingredients for synthesizing the Eu-BCKPO are listed in Table 1. The fabricating method is the solid-phase reaction at high temperatures, which is also presented in Table 1. The luminescence characterization of the phosphor includes the measurements on luminescence emission and excitation spectra, thermostability, and lifetime. Table 2 is used to describe the characterizing methods for the as-prepared phosphor. Figure 1 illustrates the LED package utilized for this paper. The real package in experiments is shown in Figure 1a while the 3D simulation of the LED, created by LightTools software, is depicted in Figure 1b. The blue-chip cluster for the LED and the cross-section of the simulated LED model are illustrated in Figure 1c and 1d, respectively. The essential of the LED package includes blue chips with a peak wavelength of 463 nm, YAG:Ce^{3+} yellow phosphor, Eu-BCKPO phosphor, and silicone gel [13, 14].

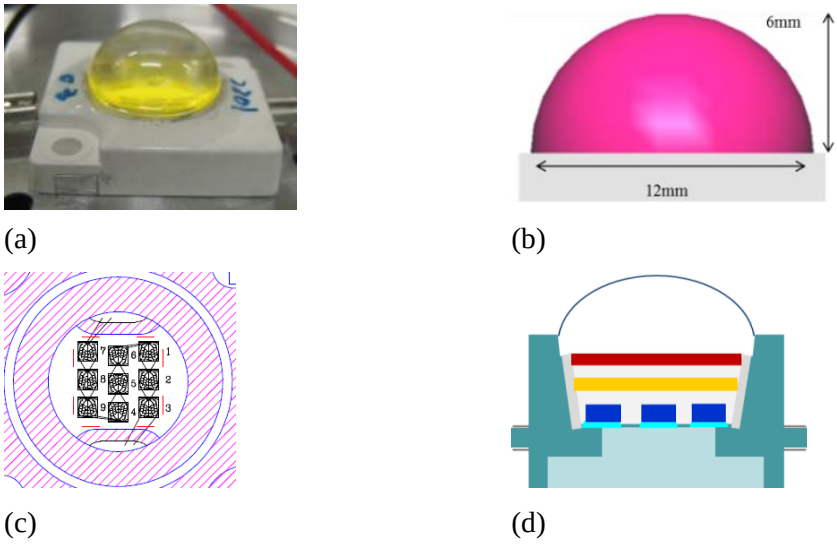


Figure 1: LED structure using in the work: (a) The actual as-prepared LED model, (b) simulation of the LED model obtained with LightTools, (c) the LED-chip packet, and (d) the LED model's cross-section

Table 1: Materials and synthesizing process of Eu-BCKPO and Mn^{2+}-Eu-BCKPO phosphors

Materials	Process
$CaCO_3$ K_2CO_3 $BaCO_3$ $NH_2H_2PO_4$ Eu_2O_3	1. The materials are weighed in a stoichiometric ratio and blended well in an agate mortar with the ethanol as an additive. 2. The mixture is dried for an hour at 70 degrees Celsius. 3. Sintering the mixture at 600 degrees Celsius for 3 hours. 4. To get the desired phosphor product, continue to heat the product at 1250 degrees Celsius for 8 more hours in the CO reduced atmospheric condition.

Table 2: Phosphor luminescence characterization

Patterns	Determining tools
Luminescence emission and excitation	Obtained at liquid helium temperature by Edinburgh FLS920 spectrophotometer combined with an excitation source of a 450-W Xe lamp
Luminescence lifetime	Edinburgh FLS920 spectrophotometer with a pumping source of EPL-375 pulse laser
Temperature-dependent luminescence	Edinburgh FLS920 spectrophotometer incorporated with a temperature-control system comprised of Oxford OptistatDN2 nitrogen cryogenics and CTI-Cryogenics

3 RESULTS AND DISCUSSION

In the BCKPO, there are five cation sites, so the Eu^{2+} ions are likely to enter these sites, resulting in the probability of five different emission peaks for the Eu-BCKPO phosphor. With 1% doping concentration of Eu^{2+}, the phosphor luminescence presented significant excitation band of 250-400 nm under 460 nm emission. The excitation band of the Eu-BCKPO is well matched with the near-UV chips. The emission of the phosphor under 365 nm near-ultraviolet excitation was collected within 400-670 nm, indicating that the phosphor exhibited more than one emission peaks [15, 16].

The luminescence examined at liquid helium temperature was taken into consideration to determine the position the phosphor emission peaks [17, 18]. The broadband emission from near-ultraviolet (400 nm) to red (700 nm) regions are noticed with five peaks at 443 nm, 460 nm, 481 nm, 500 nm, and 535 nm. The connection between the cation sites in the host and the Eu^{2+} emission peaks can be determined using the Van-Uitert expression shown in Equation (1):

$$E = Q\left[1 - (V/4)^{1/V} \times 10^{-nEar/80}\right] \quad (1)$$

where E and Q denote the energy position of the low d-band edge of the Eu^{2+} ions and the free ions, respectively. V and n depict the cation valance and the coordination number of the cation sites that the Eu^{2+} entered, respectively. Ea is constant in the same host compound while r indicates the cation radius. For phosphor designing, the Van-Uitert expression can be utilized for approximating which crystal location lead Eu^{2+} to discharge within the green and identifying the doping dosage where concentration abatement manifests, so that it is possible to adjust green phosphors to see proper color as well as highest brightness feasible in WLED apparatuses. Van Uitert suggested an experimental relationship associating the discharge power of Eu^{2+} with the localized surrounding of the trigger ion. Through said expression, it is possible to approximate the way Eu^{2+} 5d-4f discharge is altered as Eu^{2+} settles within the expected green zone roughly between 500 and 550 nm. For instance, in the case of the green, $Ca_6La_4(SiO_4)_2(PO_4)_4O_2:Eu^{2+}$, experimental relationship of Van Uitert was employed alongside formational readings for associating the greater-power band under roughly 492 nm with Eu^{2+} upon a nine-coordinate Ca location as well as the lesser-power band under roughly 539 nm with Eu^{2+} upon a more joint-valent seven-coordinate location. Said assessment enables flexible designing or selecting a host in which Eu^{2+} is more likely to settle in the location with computed discharge lies within the green, essential for filling the green gap within WLED spectra [19].

Desirable phosphor-converted WLED apparatuses necessitates a potent, wide, and thermically consistent green element for boosting chroma rendition as well as adjusting CCT. Practically, Van Uitert analysis plays a role in the designing process of said apparatuses. Utilizing the Van Uitert discharge-power relationship, it is possible to select host latticework as well as cationic locations in which Eu^{2+} presumably discharge within the green. Said method has been conducted in earlier research in the case of apatite as well as borate hosts in which location-specific assessment exhibits Eu^{2+} or Ce^{3+} bands focalized adjacent to the range of 500 to 540 nm that is appropriate for WLED apparatuses [20]. As dopant samples are made, the concentration featuring maximal intenseness prior to abatement would be selected when it comes to apparatus construction, yielding an optimal Eu^{2+} green phosphor presence [21]. Having optimized discharge chroma as well as concentration, the green phosphor would be integrated into one blue or n-UV chip as

well as supplementary red or blue phosphor samples. When a green phosphor such as $CaLu_2Sc_2Al_2SiO_{12}:Ce^{3+}$ is employed alongside a blue as well as red phosphors upon one chip under 395 nm, it is possible to acquire a warm-white device with high CRI, showing that the adjustment of green phosphor based on Van Uitert would result in desirable WLED proficiency [22].

As can be seen, the emission peaks of the phosphor are not in the red regions but in the blue-to-green regions, indicating that the phosphor can be used to bridge the green gap in multicolor LED emission. For the Eu-BCKPO phosphor composite to be suitable for high-power LED packages, it must have good thermostability because the heat emitted by the LED chip during its operation can reach to 100 degrees Celsius. The luminescence intensity of the Eu-BCKPO phosphor dropped about 48% of the initial intensity as the temperature increased to 100 degrees Celsius. This indicates that the Eu-BCKPO phosphor has adequate thermostability for LED applications. The decrease of luminescence as the temperature increase can be ascribed to the thermal quenching mechanism induced by non-radiative relaxation. The thermal-quenching activating power (P_a) could be estimated using Equation (2) [23]:

$$\frac{I_a}{I_0} = 1 + Aexp\left(\frac{-P_a}{ka}\right) \quad (2)$$

where a indicates the temperature value, so I_a and I_0 represent the emission intensity at the varied temperature a and at $a = 0$. A is a constant, and k is the Boltzmann constant.

When applied in the LED model, the Eu-BCKPO phosphor concentration was adjusted, leading to significant influence on the scattering, transmitting, and converting efficiency of the blue-light in the package. One of the impacts of Eu-BCKPO phosphor on the LED components is the change in YAG:Ce^{3+} dosage. With higher doping Eu-BCKPO phosphor amounts, the concentration of YAG:Ce^{3+} declines gradually to serve the objective of getting the color temperature (CCT) consistent, as shown in Figure 2. Moreover, the difference in concentration of phosphor amount affects the lighting characteristics including light forward and backward scattering and conversion [24]. For the scattering efficiency of the light in the presence of Eu-BCKPO phosphor, the scattering coefficients are estimated and illustrated in Figure 3. The increase of Eu-BCKPO phosphor concentration elevates the light scattering in the package. Such an event indicates that the phosphor can enhance the scattering of the blue light, from which the light conversion and transmission efficiencies can be greatly supported [25, 26].

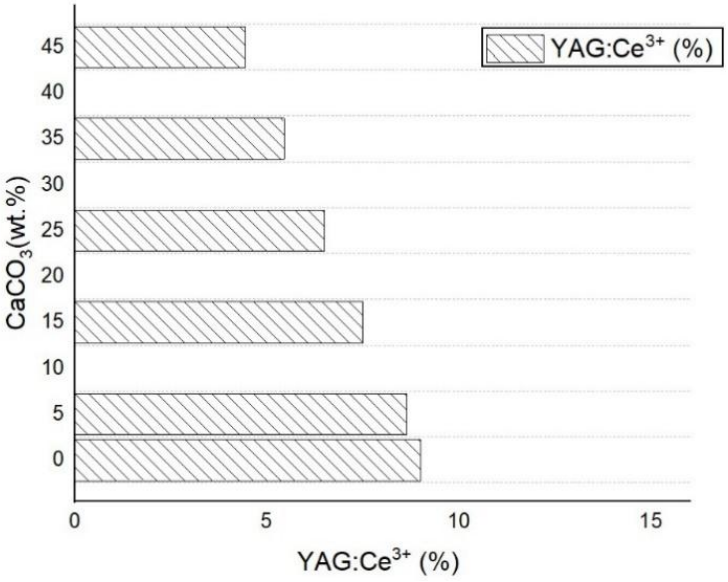


Figure 2: The amount of YAG:Ce^{3+} phosphor with different Eu-BCKPO phosphor concentrations

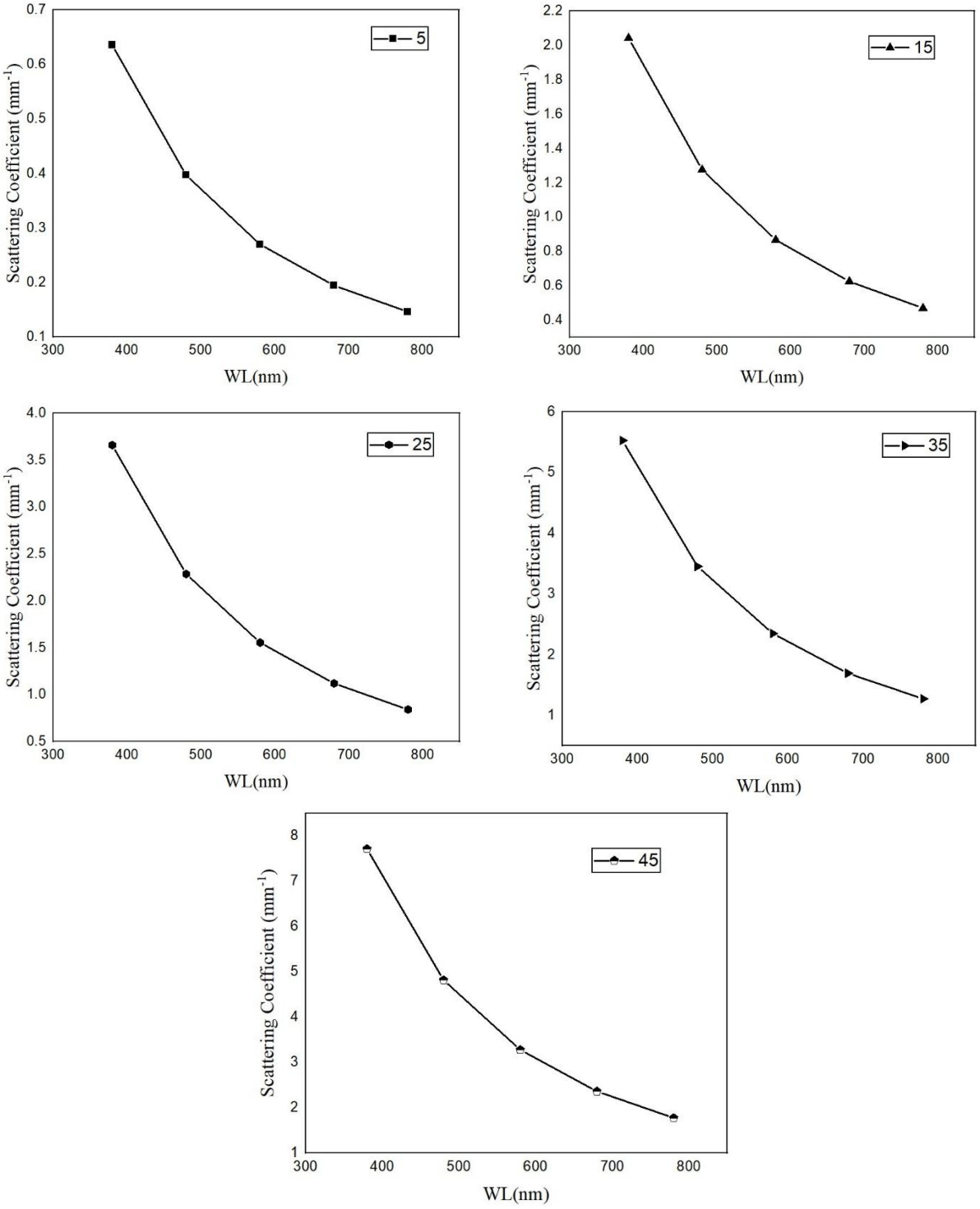


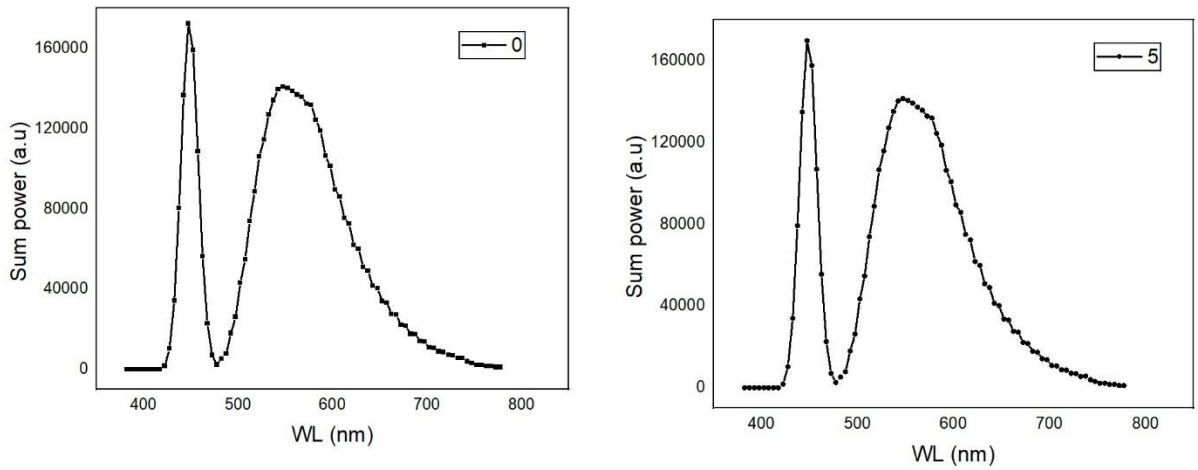
Figure 3: Scattering coefficients of light in LED with different Eu-BCKPO phosphor concentrations

The purpose of using Eu-BCKPO phosphor in the conventional LED structure is to promote the green emission strength in the luminescence of the generated light. With increasing concentration of Eu-BCKPO phosphor in the package, the green emission is gradually increase, while the blue emission intensity slightly declines. This can be seen in Figure 4. The increase in emission power in the green region (~540 nm) could be ascribed to the improved conversion efficacy induced by the scattering enhancement. Particularly, the Eu-BCKPO phosphor emission band includes the blue-to-green regions, thus it can absorb the blue light from the LED chip to convert and emits green light. In this way, the intensity of green-light power is

improved. Moreover, the intensity of blue light is also strong when the Eu-BCKPO phosphor is present. This implies that the back-scattered and re-absorbed blue light proportion is considerably reduced, contributing to enhance the lumen output of the LED light. The data in Figure 5 has confirmed this statement as the higher the doping amount of Eu-BCKPO phosphor, the more intense the lumen output of the LED light [27, 28].

Asides from the lumen efficiency of the LED, the color temperature consistency of the LED is achievable with the increasing Eu-BCKPO phosphor dosage. Figure 6 demonstrates the CCT values in the presence of Eu-BCKPO phosphor, in which Figure 6a and 6b show the angular CCT and deviation of CCT [29, 30], respectively. At the 0^o viewing angle, the CCT values is the highest, regardless of the Eu-BCKPO phosphor concentration. The highest CCT is collected at about 5200 K and gradually decreases as the concentration of the Eu-BCKPO phosphor increases but the CCT is still higher than 5000 K. Additionally, at other viewing angles, the CCT values are above 4800 K. Such findings indicate that the LED light is cool white light. In terms of the CCT consistency, the presence of Eu-BCKPO phosphor initiates noticeable fluctuation (see Figure 6b). Initially, the addition of 5-15 wt%. Eu-BCKPO phosphor induces the reduction of CCT deviation but not significantly. As the Eu-BCKPO phosphor dosage increases to 25 wt%, the CCT deviation suddenly rises. With higher doping Eu-BCKPO amount (~35 wt%), the CCT deviation significantly drops and reaches the lowest value. This means that the CCT deviation can be notably reduced with high Eu-BCKPO dosage of about 35 wt%. In other words, the Eu-BCKPO phosphor is potential to the enhancement of CCT uniformity of the LED light.

The above findings demonstrate that Eu-BCKPO phosphor is a promising material to solve the green downside while getting luminous flux and CCT uniformity of the LED light promoted. As mentioned, the Eu-BCKPO phosphor emission has weak red emission intensity, thus, the color rendering efficiency of the LED light with Eu-BCKPO phosphor is not as good as expected. The rendering performance with increasing Eu-BCKPO concentration can be observed in Figure 7. The color rendering index (CRI) in Figure 7a and color quality scale (CQS) in Figure 7b show gradual decline with increasing Eu-BCKPO phosphor dosage in the package. Therefore, to get the higher color rendition performance, the red spectra in the phosphor luminescence should be stimulated [31, 32]. On the other hand, the purpose of the work to examine whether the Eu-BCKPO phosphor can enhance the green emission intensity in the LED light luminescence is achievable.



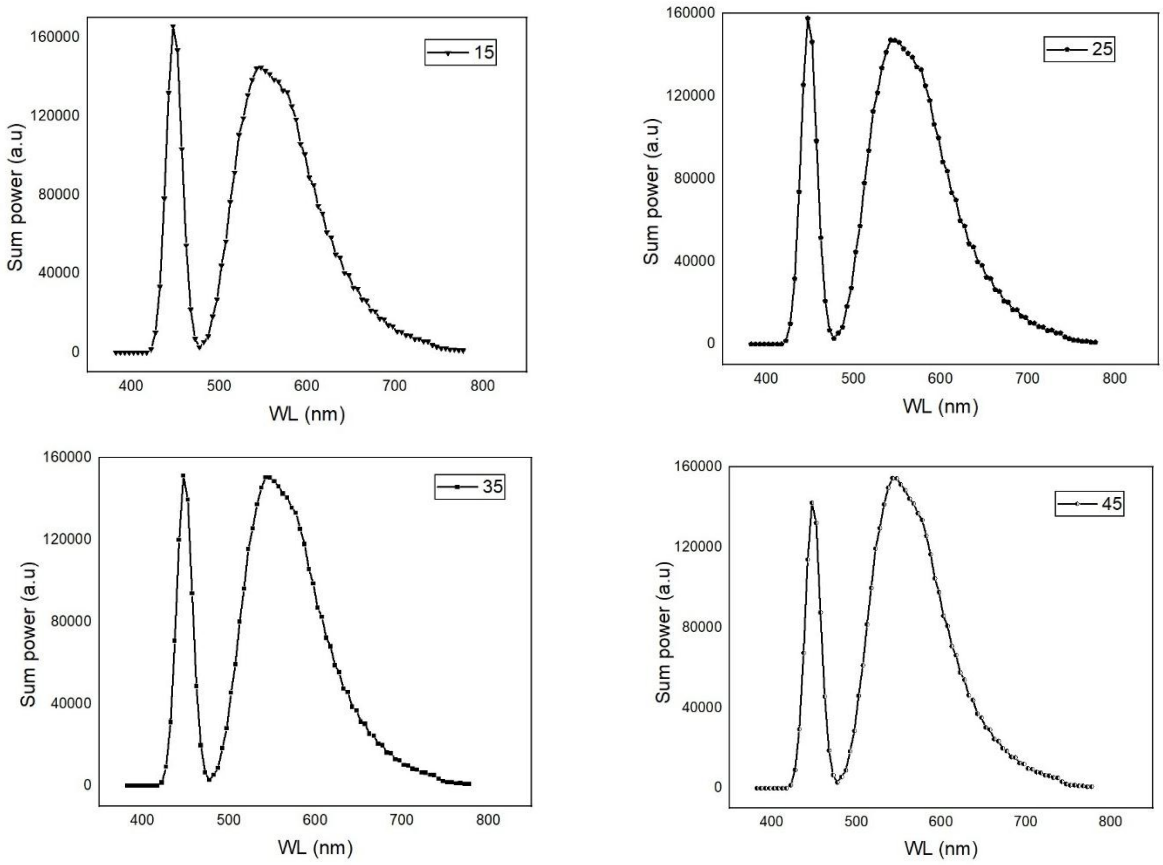


Figure 4: The total luminescence of the LED light in the presence of Eu-BCKPO phosphor

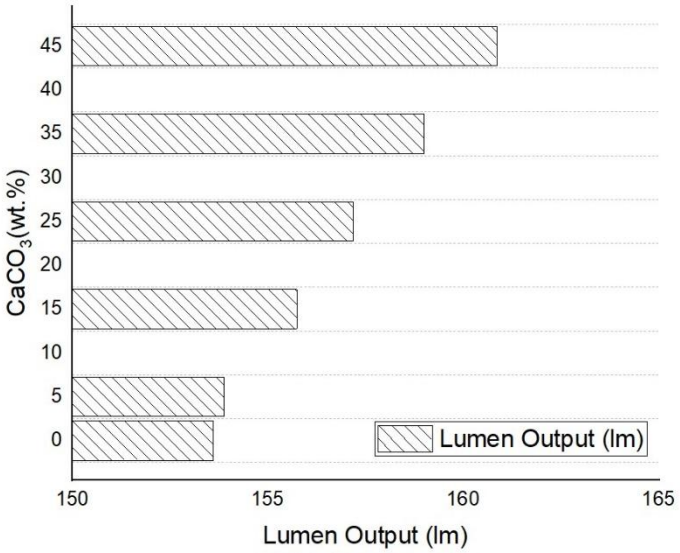
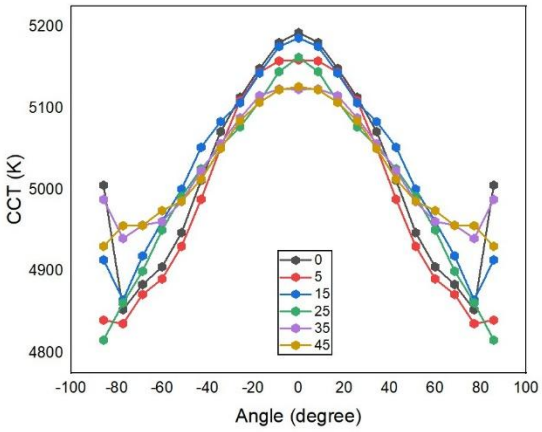
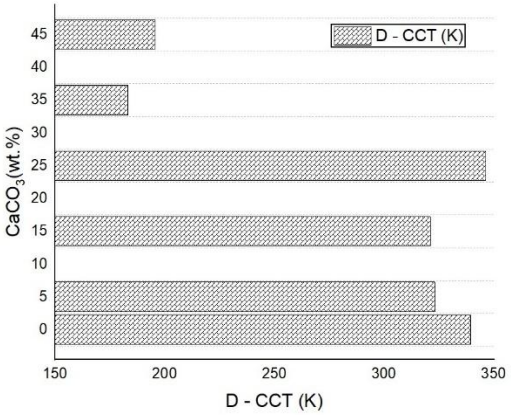


Figure 5: The lumen output of the LED light in the presence of Eu-BCKPO phosphor

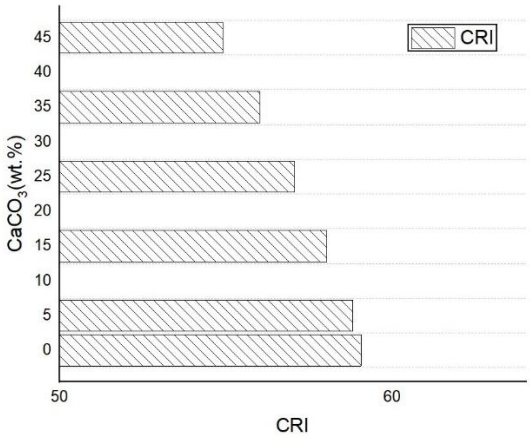


(a)

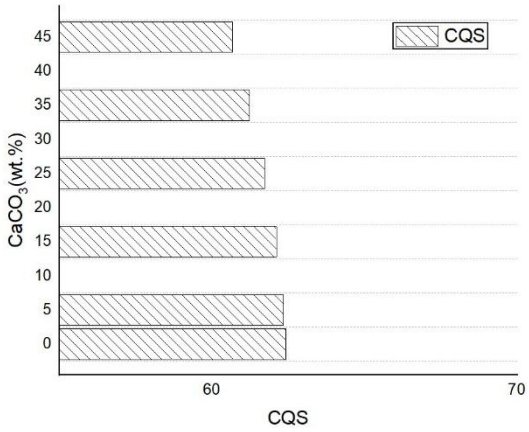


(b)

Figure 6: CCT properties of the LED light in the presence of Eu-BCKPO phosphor: (a) Angular CCT and (b) CCT deviation



(a)



(b)

Figure 7: Color rendition performance of the LED light in the presence of Eu-BCKPO phosphor: (a) CRI and (b) CQS

4 CONCLUSION

The Eu-BCKPO phosphor was prepared through the solid-phase reaction at 1250 degrees Celsius in this paper. The obtained Eu-BCKPO phosphor presents strong excitation band matching the near-ultraviolet excitation wavelengths. The phosphor also has board emission from blue to orange-red, with the peak positions at 443 nm, 460 nm, 481 nm, 500 nm, and 535 nm. The thermostability of the phosphor is adequate for being used in conventional LED light. The phosphor shows potential to bridging the green gap in the LED luminescence. The increase of Eu-BCKPO phosphor concentration in the package promotes the blue-light scattering and green-light conversion, leading to more intense green spectrum and LED lumen output. Additionally, the generated LED light in the presence of Eu-BCKPO phosphor is the cool white light with CCT varies within 4800-5200 K. Moreover, at 35 wt% of Eu-BCKPO, the lowest CCT deviation is obtained, denoting the ability to enhance the CCT uniformity of the phosphor. Nevertheless, due to the weak red emission of the Eu-BCKPO phosphor, the CRI and CQS gradually decrease as the phosphor doping concentration increases. It is essential to improve the red emission of the Eu-BCKPO phosphor to get the higher color rendering efficiency.

APPENDIX

Appendixes, if needed, appear before the acknowledgment.

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PHỐT PHO $BA_3CAK(PO_4)_3:EU^{2+}$ TĂNG CƯỜNG ÁNH SÁNG XANH CHO CÁC ỨNG DỤNG ĐÈN LED TRẮNG

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Tóm tắt. Phốt pho $Ba_3CaK(PO_4)_3:Eu^{2+}$ (Eu-BCKPO) được tổng hợp bằng phương pháp phản ứng trạng thái rắn ở nhiệt độ cao. Hợp chất phốt pho được tổng hợp có thể tạo ra ánh sáng trắng mát với hiệu suất lượng tử cao do sự thay thế vị trí của Eu^{2+}. Để kiểm tra tác động của phốt pho Eu-BCKPO lên dải phát xạ xanh và tổng thông lượng sáng của đèn LED thông thường, các lượng khác nhau của phốt pho Eu-BCKPO được kết hợp với chip xanh (465 nm) và phốt pho vàng YAG:Ce^{3+}. Lượng phốt pho Eu-BCKPO tăng lên hỗ trợ sự ổn định của nhiệt độ màu và cải thiện độ sáng của đèn LED được kiểm tra. Vùng bước sóng xanh trong quang phổ LED tổng thể cũng tăng lên với lượng pha tạp Eu-BCKPO cao hơn. Tuy nhiên, hiệu suất hoàn màu của ánh sáng trắng LED giảm xuống khi nồng độ phốt pho Eu-BCKPO tăng do thành phần màu đỏ không đủ. Do đó, Eu-BCKPO có thể được sử dụng trong chế tạo LED xanh hoặc kết hợp với vật liệu phát quang màu xanh và đỏ để tạo thành các thiết bị LED nhiều màu/trắng.
Từ khóa: $Ba_3CaK(PO_4)_3:Eu^{2+}$, Nhiệt độ màu, Diode phát quang, Vật liệu phát quang, YAG:Ce^{3+}

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