

Investigation on $\text{BaSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ Phosphor with Blue-Green Emission for Optical Enhancements of WLEDs

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ABSTRACT: $\text{BaSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ phosphor (BaEu phosphor) with color emission presented in blue-green spectral region is fabricated by applying the precursor of $\text{Ba}_2\text{SiO}_4:\text{Eu}^{2+}$ and a doping concentration of Eu^{2+} ion of 0.05. The peak emission intensity of the prepared BaEu phosphor is observed at 490 nm – 500 nm wavelength band. Under the InGaN LED light excitation of 460 nm, the phosphor show better luminescence-intensity peak than the original $\text{YAG}:\text{Ce}^{3+}$ yellow phosphor, with the same give dopant concentration of 5% mol. In addition, the phosphor exhibits small thermal quenching as the luminescence recorded at 150°C maintain at a level as high as 75% of the room-temperature luminescence. Furthermore, when being applied to a WLED, BaEu phosphor presents certain effects on the chromatic quality and luminous intensity, depending on the applied concentration. With 5%wt. concentration, BaEu phosphor is beneficial to the luminous flux of 3000 K WLED and color rendering features. Meanwhile, the concentration of 10% results in higher luminous intensity of 4000 K WLED, and better color uniformity for both preset correlated color temperatures of WLEDs.

KEY WORDS: WLEDs, $\text{BaSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$, Monte Carlo theory, Color homogeneity, Luminous flux

Date of Submission: 09-07-2026

Date of acceptance: 20-07-2026

INTRODUCTION

Since the outstanding features of white light emitting diodes (WLEDs) was reported in many earlier research papers, including the high endurance, longevity, non-mercury fabrication and energy efficiency, they have been recognized as one of the most promising sources of illumination for solid-state lighting technologies [1, 2]. The WLED using phosphor conversion materials, also called phosphor-converted WLED, is consider as a common LED type to generate white light [3-5]. In this structure, the phosphor is excited by the blue excitation from the LED chips, thus it is essential to select a phosphor material that provides good excitability in the blue wavelength of the LED chip [6-8]. $\text{MSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ phosphors, with M = Sr, Ba, Ca, which possess great luminescent characteristics and good thermal stability, have been extensively analyzed as a potential candidate for suitable phosphor material to benefit the WLED's lighting performances [9-11]. The mentioned studies shown that $\text{MSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ had high probability of emission tuneability in the range of about 490 – 585 nm under the blue excitation of 460 nm. This result can be adjusted and the high color rendering WLED could be recognized by varying the concentration of Eu^{2+} dopant ion and the type of M. Among the mentioned types of M, the Ba turned out to be the more appropriate one as $\text{BaSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ (shortened as BaEu) presented a narrow band of emission centered at 490 nm – 500 nm, which could offer a sufficient compensation of blue-green spectral energy that is shortage in the common yellow-phosphor-converted WLED package. This property also indicates that $\text{MSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ (M = Ba) phosphor has only one Ba site with highly symmetric surrounding in its crystal structure [12, 13]. Consequently, a phosphor-converted WLED with superior CRI was fabricated by utilizing the combination of this blue-green emitting BaEu phosphor and other phosphors with yellow, red, and green emissions [14, 15]. The investigation on the luminescence and crystal geometry of BaEu phosphor were first presented in 2005 by Li's research team [16]. The BaEu phosphor was prepared using the common solid-state reaction with the starting composition of BaCO_3 , Eu_2O_3 , SiO_2 , and Si_3N_4 . Nevertheless, in 2009, another report stated that the first prepared BaEu phosphor was impurity; and the authors also denied the accuracy of the previous report on the crystal structure of BaEu phosphor [17-19]. They showed that the crystal formation of BaEu occurred in an orthorhombic crystal system with space group $Cmcm$ and the cell parameters $a = 14.44 \text{ \AA}$, $b = 5.34 \text{ \AA}$, and $c = 4.83 \text{ \AA}$, and this result does not match with that from the study in 2005. The authors then

prepared the BaEu phosphor via the same method of solid-state reaction but using different starting composition of BaCO_3 and Si_3N_4 . However, the high possibility of contaminated BaEu phosphor was still reported due to the Si_3N_4 presence, and the single-phase BaEu phosphor could not be obtained as a result.

The single phase of the BaEu phosphor could be obtained by utilizing a precursor material of $\text{Ba}_2\text{SiO}_4:\text{Eu}^{2+}$ with various amount of the ingredients of the phosphor composition. Luminescence characteristics of the phosphor is included and the influences of the BaEu phosphor concentrations on the lighting features of WLED are also examined and discussed to provide a valuable reference for the selection of phosphor conversion material that meet the requirements for high-power WLED applications.

EXPERIMENTS

$\text{BaSi}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ (shortened as BaEu) phosphor with strong blue-green emission is prepared by utilizing $\text{Ba}_2\text{SiO}_4:\text{Eu}^{2+}$ phosphor as the precursor and with 0.05 Eu^{2+} doping concentration. The composition of the required phosphor includes BaCO_3 , SiO_2 and Eu_2O_3 . The procedure for BaEu preparation is started with mixing all the ingredients in stoichiometric ratios, and ended with the washing stage using 10% v/v nitric acid aqueous solution. After being well mixed, the attained mixture is heated at 1100°C in the gas flow of 5% H_2 and 95% N_2 for around three hours. As the first firing finishes, the $\text{Ba}_2\text{SiO}_4:\text{Eu}^{2+}$ precursor is obtained, and subsequently is ground into powder and blended with Si_3N_4 . Then, the second firing process that lasts six hours is carried out at $1200^\circ\text{C} - 1500^\circ\text{C}$ in the same reducing gas flow. The re-fired products, after being washed with nitric acid, subsequently encounters a 24-hour drying process at 120°C to get the final phosphor material for further investigations. The excitation and emission spectra of the BaEu phosphor is identified with a fluorescence spectrophotometer. The examinations of morphology and grain size of BaEu are performed with a SEM – scanning electron microscope. The crystal structure of the prepared phosphor is identified with a X-ray diffractometer (XRD) operated at 40 kV and 30 mA Cu K radiation. An oxygen/nitrogen analyzer together with an energy-dispersive X-ray spectrometer are utilized for the elemental investigation of the phosphor. The specific brand and model of the mentioned instruments are provided in Table 1. Besides, the temperature-dependent luminescent characteristic of BaEu phosphor is observed with a photomultiplier under a InGaN-based LEDs light excitation of 460 nm [20].

Table I. Models of phosphor-characterization instruments

Instruments	Models
Fluorescence spectrophotometer	F-4500, Hitachi, Japan
Scanning electron microscope	S-3000HXS, Hitachi, Japan
X-ray diffractometer	RINT2200, Rigaku, Japan
Oxygen/nitrogen analyzer	EMGA-550, Horiba
Energy-dispersive X-ray spectrometer	EMAX-7021H, Horiba

RESULTS AND ANALYSIS

When BaEu bluish-green emitting phosphor (B-GEP) is added into the phosphor configuration of the white LED structure, the changes in the concentration levels of the yellow phosphor $\text{YAG}:\text{Ce}^{3+}$ is observed, at two different correlated color temperatures (CCTs) of 3000 K and 4000 K, as displayed in Figure 1 (a) and (b), respectively. As can be seen the higher concentration of BaEu phosphor results in lower level of yellow phosphor concentration, especially at higher CCT of 4000 K. Besides, the concentration of yellow phosphor at 3000 K WLED shows insignificant differences between two concentrations of added BaEu. In this case, the decrease in concentration of yellow phosphor is also dependent on the particle size of the B-GEP. As demonstrated in Figure 1 (a), with $1\mu\text{m}$ particle size of BaEu phosphor, $\text{YAG}:\text{Ce}^{3+}$ concentration in regard to 10wt% BaEu shows high level, about 45%. As the particle size of BaEu increases, the yellow phosphor percentage correspondingly decreases. The lowest concentration of $\text{YAG}:\text{Ce}^{3+}$ can be observed when BaEu phosphor particle size of 4 – 6 μm . With bigger size of BaEu from 7 – 20 μm , the concentration of the yellow phosphor slightly increases and then being relatively stable, especially in the case of 10wt% BaEu concentration. In WLED having 4000 K CCT in Figure 1 (b), the changes in $\text{YAG}:\text{Ce}^{3+}$ with different concentration of this B-GEP is more easily noticed. Also, the particle size of BaEu considerably impacts the attained figures. The higher the concentration, and the smaller the particle size of the green phosphor, the lower the concentration of $\text{YAG}:\text{Ce}^{3+}$. In other words, as size of the applied B-GEP becomes larger, the concentration of yellow $\text{YAG}:\text{Ce}^{3+}$ phosphor increases, regardless of BaEu concentration. At 4000 K, $\text{YAG}:\text{Ce}^{3+}$

concentration bottoms out with the addition of 10%wt and 2 μm BaEu phosphor concentration and particle size, respectively. YAG:Ce^{3+} phosphor could yield high luminescence, yet its scattering is inferior, leading to low chromatic homogeneity and efficiency in light extraction [21-23], the addition of BaEu with small particle size and high concentration could promote the presence of scattering, and the reduction in light loss caused by inefficient light extraction. In addition to that, the concentration decrease of YAG:Ce^{3+} with the existence of BaEu phosphor can support the stability of the thermal properties as well as the pre-determined CCTs of the WLED. Thus, there is a high probability that using blue-green emitting BaEu phosphor encourages the improvement of chromatic features and luminous performances of the WLED. The following discussion will focus on the color rendering metrics, color uniformity, emission spectra and luminous efficacy of the WLED with the presence of BaEu phosphor in the package to examine this point.

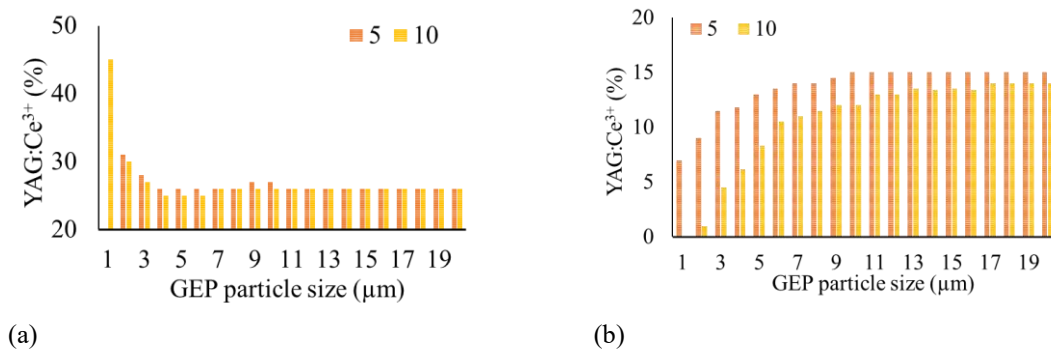


Figure 1: The change of YAG:Ce^{3+} concentration with different BaEu concentrations: (a) 3000 K; (b) 4000 K

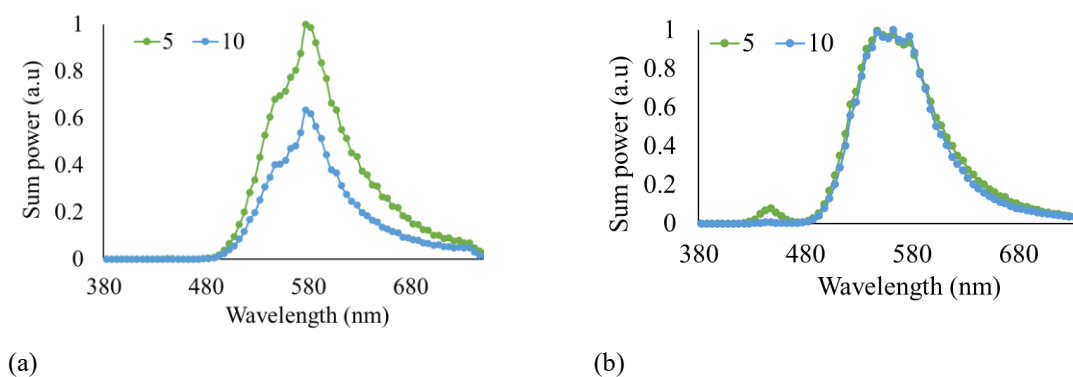


Figure 2: Power emissions of WLEDs with different BaEu concentrations at: (a) 3000 K; (b) 4000 K

The sum power of the phosphor-coated LED with two different concentrations, 5% and 10% wt., of BaEu phosphor is presented in Figure 2. The WLED emission power recorded at 3000 K and 4000 K shows that 5%wt BaEu leads to greater output of the emission energy. In particular, at the same concentration of 5% concentration of BaEu, the 3000 K WLED exhibits much stronger power in the wavelength range of about 500 nm – 700 nm. Meanwhile, the 4000 K package shows relatively identical result to that in the case of 10%wt. BaEu, except a small enhancement in 420 nm – 470 nm wavelength range. Additionally, the emission power centers of WLEDs having 3000 K and 4000 K CCTs with BaEu addition can be observed around 580 nm, and in 530 – 550 nm wavelength bands, respectively. These results indicate the stronger spectral power in the wavelengths of green, blue, yellow, and red lights. In other words, conversion efficiency and extraction efficiency, or luminous performance of WLEDs are enhanced with BaEu phosphor, especially, when being used at lower concentration. Plus, the results imply that the chromatic rendering of the white LED is probably boosted due to the improvement in color balance and red spectral energy along with the presence of BaEu phosphor.

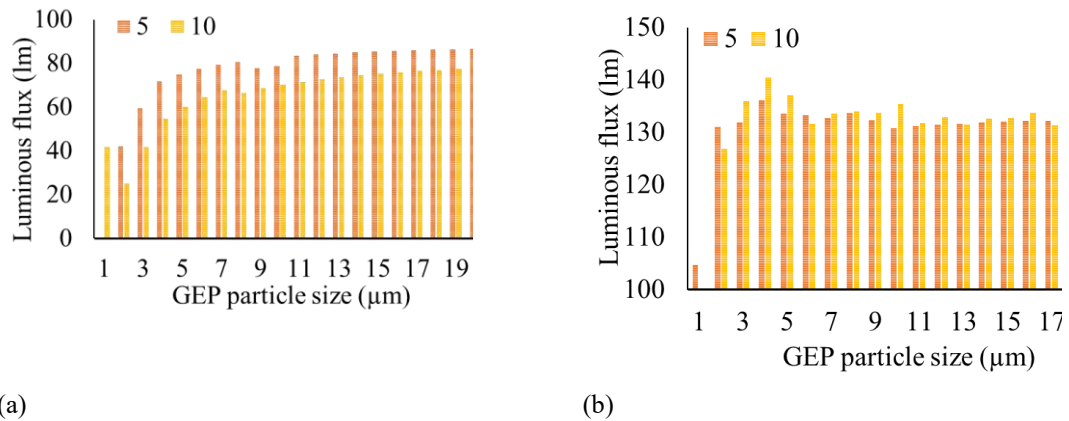


Figure 3: Luminous output of WLEDs with different BaEu concentrations at: (a) 3000 K; (b) 4000 K

The effects of BaEu blue-green phosphor on the luminous flux illustrated in Figure 3 demonstrates that 5%wt concentration is more beneficial to the WLED with lower CCT (3000 K). This means the lumen output of 4000 K WLED can be promoted with higher weight percentage of BaEu phosphor. As can be seen in Figure 3 (a), the utilized green-phosphor concentration at 5wt% results in stronger luminescence output, and the increasing particle size also elevate the intensity. This may be related to the power emission in Figure 2 and the fact that larger particle size of phosphor contributes greatly to stimulating the proportion of lights to be transmitted in more a straight direction. Therefore, the limitation on the trapped light and energy loss is set and consequently enhancing the emission intensity. On the other hand, the luminous flux of the WLED with 4000 K tends to be better with higher concentration of BaEu phosphor, see Figure 3 (b). At 4000 K CCT, 10wt% BaEu is more advantageous as the concentration of yellow phosphor concentration is lower, refer to Figure 1 (b). Though this improvement of luminous flux is not too considerable, in comparison with the 5%wt one, it is undeniable that BaEu is effective for the stronger luminous output of the WLED. Besides, that the luminous flux at 5wt% concentration of BaEu is somehow lower in the WLED having 4000 K might be ascribed to the enhancement in the blue emission region (see Figure 2 (b)), which also indicates that the color rendition might be better.

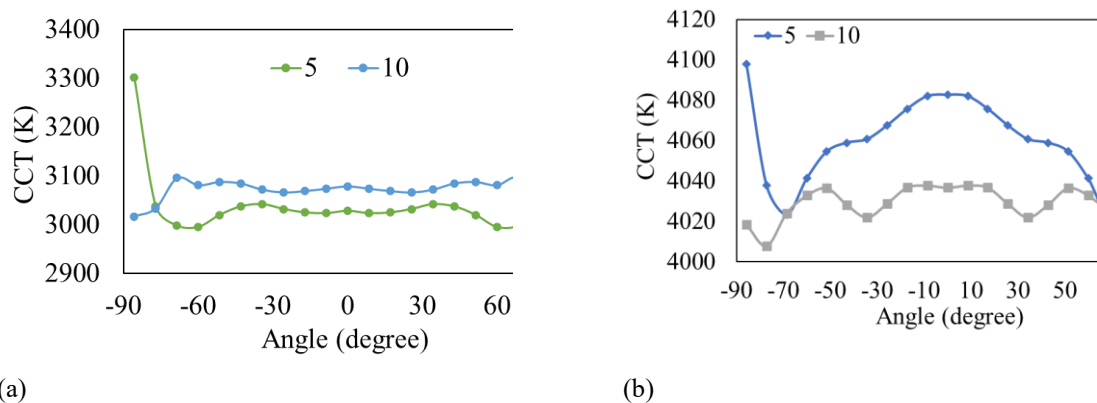


Figure 4: The CCT difference of WLEDs with different BaEu concentrations at: (a) 3000 K; (b) 4000 K

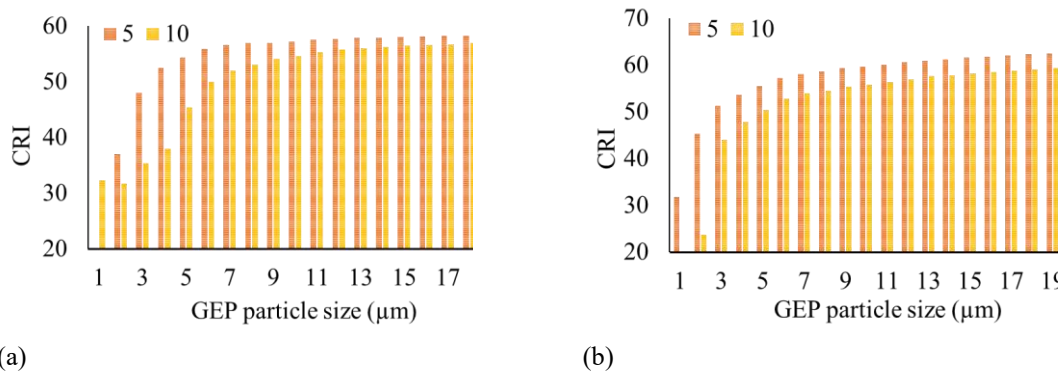


Figure 5: CRI values of WLEDs in regard to BaEu concentrations at: (a) 3000 K; (b) 4000 K

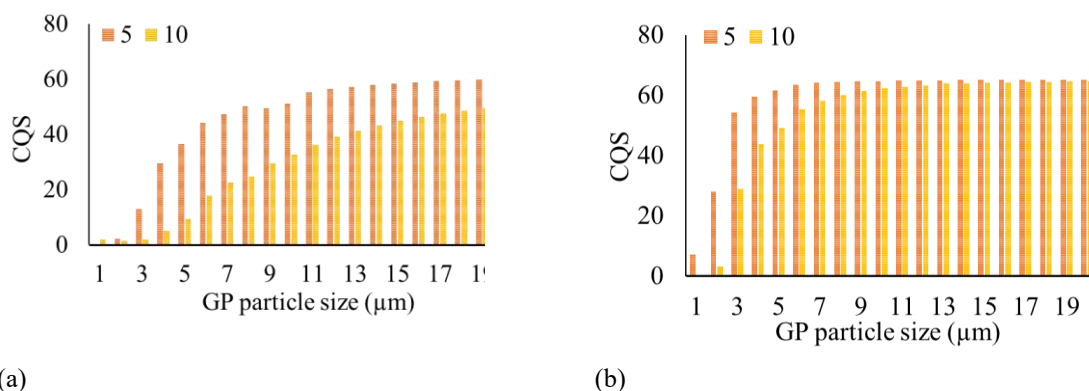


Figure 6: CQS values of WLEDs with different BaEu concentrations at: (a) 3000 K; (b) 4000 K

As all the WLEDs are not used to serve general illuminating purposes, the LED with high luminous intensity could be suitable for other applications, for example, backlighting for LCD – liquid crystal display technology. In terms of general lighting applications, it is quite challenging for the development of WLED sources to reach the “ideal” label. The reason may lean over the preference of human eyes since there is no perfect match between the response of the eyes to the observed color and emissions of the light sources. In other words, the color that the eyes recognized between the two light sources might be the same, yet their emissions can be distinct from each other. Thus, to effectively improve the evaluation of the WLED’s color performance, the human visual preferences should be included. Additionally, it is essential to determine the priority of the optical improvements for the WLED depending on the targeted lighting applications.

The color uniformity of a WLED can be evaluated by considering the deviations of chromaticity in the given viewing angle. The chromatic deviation could be calculated by the subtraction of maximum CCT – minimum CCT. The CCT deviation of the WLED with BaEu concentration is described in Figure 4, in which the viewing angle is set from -90 to 90 degree. At both preset CCTs, 10wt% BaEu phosphor results in smaller color variances. This implies that the color uniformity when using 10%wt BaEu is better than that with 5%wt BaEu. This might be the result of the light-scattering enhancement and the sufficient spectral-energy compensation of chromatic components, including blue, green, and red. The improved scattering probably gets light distributed in various direction, leading to the stimulation in light combing process, which is advantageous to the uniformity of emitted white lights. Moreover, the BaEu phosphor helps to promote the conversion efficiency of blue light, since it will convert those blue rays to green beams, once these blue radiations are absorbed by the green phosphor BaEu. Consequently, the green light component becomes greater in amount and contributed to heightening the color uniformity and luminous flux of the WLED, which is one of the most important functions of the BaEu phosphor in WLED structures. In addition to the uniform color property, the color rendering is another crucial aspect in the chromatic assessment of white-light color quality. The color rendering index (CRI) is extensively utilized in studying the color evaluation of white LED. However, the CRI has a limitation on the range of color samples, in other words it can only offer a small reproduction of color gamut. Besides, as mentioned above, for the adequate lighting quality of WLED, it is essential to consider the observer’s preference as one of the vital factors. Thus, the developed parameter for color rendering assessment called color quality scale – CQS has been proposed. By combining the CRI, color-coordinated property, and the

observer's preferences, CQS probably presents more improvement over the CRI [24, 25]. Thus, attaining high CQS is relatively difficult than producing high CRI. The CRI and CQS of the WLED with different concentration of BaEu examined at 3000 K and 4000 K are presented in Figure 5 (a), (b) and Figure 6 (a), (b), respectively. Generally, both CQS and CRI of the WLED at two different CCTs shows a minor decrease when using 10wt% BaEu concentration, compared to those in the case of 5wt% BaEu. This can be demonstrated with the side effect if excessive scattering enhancement which is the imbalance between the color components. Specifically, as the scattering is boosted with higher concentration and small particle size, the blue-light conversion will get more benefits. As a result, the green color emission will prevail over the others, causing a large difference in the rendered color. Thus, the color rendition is degraded in general. The highest values of CRI and CQS can be observed around 60 with 5wt% concentration and increasing particle size of BaEu phosphor.

CONCLUSIONS

The study shows that BaEu phosphor fabricated by using the precursor of Ba₂SiO₄:Eu²⁺ precursor and Eu²⁺ ion at 0.05 dopant concentration results in strong blue-green emission that peaks 490 nm – 500 nm. When being excited under 460 nm LED light, at the same dopant concentration of 5%, BaEu phosphor presents higher luminescent intensity than the YAG:Ce³⁺ one. Additionally, the examinations on BaEu phosphor when being added in phosphor package of a WLED display that by varying the phosphor concentration, the desired enhancement in certain optical properties can be accomplished. Particularly, lower concentration of BaEu (5%wt) is good for stimulating the luminous output of 3000 K WLED and color rendering features. On the other hand, with higher dopant concentration (10%wt.), BaEu phosphor benefits the lumen of the WLED having 4000 K CCT, and the color uniformity. It also recorded that the combination between the increasing particle size and lower concentration of BaEu could lead to the overall enhancements of both color and luminous performance of the white LED. Thus, the BaEu phosphor can be the potential to access the further development of phosphor conversion material for high-power WLED lights.

ACKNOWLEDGEMENT

The author wishes to express their gratitude to the Posts and Telecommunications Institute of Technology, Vietnam, for financial support for this research.

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