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Blue-emitting fluorophosphate phosphor to enhance color rendition of near-ultraviolet LED white light

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ABSTRACT

This work presents a novel blue phosphor, Na_2MgPO_4F: Eu^{2+} (NMPF: Eu) for violet 405-nm light-emitting diode (LED) devices. The compound is synthesized via a simple one-step sintering process using readily available precursors. Notably, NMPF: Eu exhibits highly efficient and thermally stable blue emission when excited by violet light. The NMPF: Eu is applied to produce a white LED device driven by a 405 nm LED chip and Y3Al5O12: Ce3+ (YAG: Ce) yellow phosphor. The impacts of NMPF: Eu phosphors are investigated by varying their particle size while maintaining consistent doping concentrations. Impressively, the LED prototype displays a substantial reduction in blue light emission while generating white light with enhanced color rendering and luminous properties. These results highlight the suitability and potential of NMPF: Eu as a promising phosphor for widening violet LED applications, especially in generating white light perceptible to the human eyes.

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1. INTRODUCTION

The continuous advancement of energy-efficient lighting technologies has led to the widespread adoption of light-emitting diode (LED) lights for various applications, such as general illumination, displays, and automotive lighting [1]–[3]. To achieve high-quality white light, LEDs often employ phosphor materials to convert part of their emission spectrum to complementary colors, typically blue light excitation to yellow or red-light emission. One of the most current ways to achieve the efficient white light for LED lighting systems is the utilization of InGaN blue-emitting LED chips combined with the yellow-emitting $Y_3Al_5O_{12}$: Ce^{3+} phosphor (YAG: Ce). However, this type of LED device is reported with low color rendering efficiency and cool white light owing to the intense blue-light emission [4].

Achieving a reduction in LED-chip blue emission intensity can be accomplished through a viable approach involves employing the combination of phosphor materials of blue, red, and green emissions and a 405-nm violet LED chip. The violet-LED generated white light exhibits improved color rendering index (CRI) and lumen performance compared to that from traditional blue-LED packages. Additionally, it reduces the likelihood of droop, enhancing overall LED performance. Furthermore, employing violet LEDs enables the covering over the near-ultraviolet (UV) and blue regions, creating a continuous and broader emission spectrum to improve the chromatic tunability [5], [6]. Nonetheless, under the violet excitation of 405 nm, the phosphor must exhibit a high photoluminescent quantum yield to address the stokes' loss issue. Moreover, the phosphors must demonstrate emission intensity degradation and chromaticity drift due to variations in temperature to deliver a consistent emission color perceptible to the average human eye.

Many studies have investigated and proposed potential green and red phosphors satisfying the posing requirements but analysis on effective phosphor under a 405-nm excitation is barely conducted. The most common blue phosphor in the market is $BaMgAl_{10}O_{17}$: Eu^{2+} phosphor. This phosphor was reported to exhibit efficiently high quantum yield under the 400-nm violet LED, making it suitable for plasma display technologies and domestic illumination. However, one significant limitation of $BaMgAl_{10}O_{17}$: Eu^{2+} lies in its susceptibility to hydrolysis, leading to the rare-earth ionic oxidation from Eu^{2+} to Eu^{3+}. Consequently, prolonged operation of this phosphor results in observable shifts in the emission wavelength and a subsequent decrease in emission intensity. While alternative blue phosphors like $Sr_3MgSi_2O_8$: Eu^{2+} (SMS: Eu) and $Ca_5(PO_4)_3Cl$: Eu^{2+} (CPC: Eu) exhibit strong absorption in the violet region, they suffer from specific shortcomings. Particularly, the SMS: Eu phosphor demonstrates poor thermal stability, and the CPC: Eu experiences reduced efficiency, making them less suitable alternatives in practical applications [7]–[10].

In this research paper, we report a novel blue phosphor Na_2MgPO_4F: Eu^{2+} (NMPF: Eu) that is well excited by violet LED chips. The synthesis of this compound involves a one-step sintering process using available precursors. A key feature of this phosphor is its high blue-emission efficiency and stability under violet illumination excitation. We demonstrated the application of NMPF: Eu in a LED device pumped by a 405 nm LED chip, resulting in the generation of white light with improved chroma rendering efficiency. The impacts of NMPF: Eu phosphors are monitored by varying its particle size while keeping its doping concentration fixed. Remarkably, the device exhibited enhanced color rendering properties with high luminosity with the utilization of bigger NMPF: Eu particle sizes. Collectively, these findings underscore the efficiency of the NMPF: Eu phosphors for the development of violet LED devices.

2. METHOD

The NMPF: Eu phosphor was produced using the solid phase reaction process with high purity (>99.9%) reacting agents. The specific elements and their origins are shown in Table 1. To start the synthesis, all reagents were blended stoichiometrically in an acetone medium by grinding in an agate mortar. The doping concentration of Eu was at 0.04 mol% and Na_2CO_3 was added with an additional amount of 7.5 wt%. This addition contributes to compensating for the evaporation during synthesis [11], [12]. Then, the initially mixed compound was subjected to a ball-milling grinding process for 100 minutes. The resulting powder was subsequently pressed into a pellet with a radius of 3 mm. Next, the pellet was put in the alumina crucible and the sintering process was carried out at 825°C for 8 hours in a reduced atmosphere ($5\%H_2$: $95\%N_2$). Afterward, the heated pellet was re-ground for further examinations. The luminescence of the NMPF: Eu phosphor and the produced white LED were collected using the instruments listed in Table 2.

Table 1. Eu^{2+}-doped Na_2MgPO_4F starting reagents

Reacting components	Bought from
Na_2CO_3	Sigma Aldrich
MgF_2	Sigma Aldrich
MgO	Sigma Aldrich
$NH_4H_2PO_4$	Acros Organics
Eu_2O_3	Alfa Aesar

Table 2. Luminescence determining tools

Characteristics	Tools
Phosphor luminescent spectrum	Horiba Fluoromax-4 fluorescence spectrophotometer (excitation source of a 150-W xenon lamp)
Temperature-dependent emission spectrum	PTI fluorescence spectrophotometer (excitation source of a 75-W xenon lamp) with a Janis VPF-100 cryostat for an 80-640 K temperature-controlled environment
Quantum yield	Labsphere spectralon-coated 150-mm-diameter integrating sphere and de Mello et al's method
LED luminous spectrum and performance	Avasphere-5-IRRAD spectrophotometer

3. RESULTS AND DISCUSSION

The NMPF adopts an orthorhombic crystal structure, classified under the Pbcn (No. 60) space group. Its structure is characterized by a robust and tightly interconnected backbone composed of NMPF units. Within this intricate unit, two crystallographically independent NaO_5F_2 polyhedra, centered on Na atoms, share faces with two highly distorted MgO_4F_2 octahedra [13]. Despite the isovalency between

Eu^{2+} and Mg^{2+}, it is anticipated that Eu^{2+} ions will substitute for one or both of the Na+ ions. This expectation arises from the fact that the ionic radius of Eu^{2+} closely matches that of Na^{+}, in contrast to Mg^{2+} [14]. This ionic substitution in the NMPF: Eu structure can contribute to the generation of blue light under the UV illumination. The excitation spectrum of the NMPF: Eu phosphor reveals notable peaks at approximately 340, 365, and 400 nm. Generally, the excitation peaks at around 340 nm and 365 nm are common with blue-emitting phosphors, but the NMPF: Eu phosphor shows ability to be excited at 400 nm, resulting in the production of blue light with a uniquely short stokes' shift. Consequently, the blue-emission minimization can be achieved for LED light, especially crucial for the LED applications focus on producing warm white light for human-visual comfort at night [15].

When using the NMPF: Eu phosphor in the LED fabrication, its particle size is altered, inducing the changes in the scattering factor of the package. We determined the scattering of light at different wavelengths from 380 nm to 780 nm and demonstrated the result in Figure 1. All wavelengths show increased scattering performance, both reduced scattering and scattering coefficients, in response to the enlargement of NMPF: Eu particle diameter. Moreover, the UV (380 nm) show the highest reduced scattering coefficients, indicating the higher UV-light utilization by the phosphor with large diameter. The improved scattering efficiency on the increasing NMPF: Eu particle size also contribute to induce the excitation light absorption effectiveness and re-emitted light scattering by the phosphor materials in the package. As a result, the color dispersion and reproduction can be enhanced.

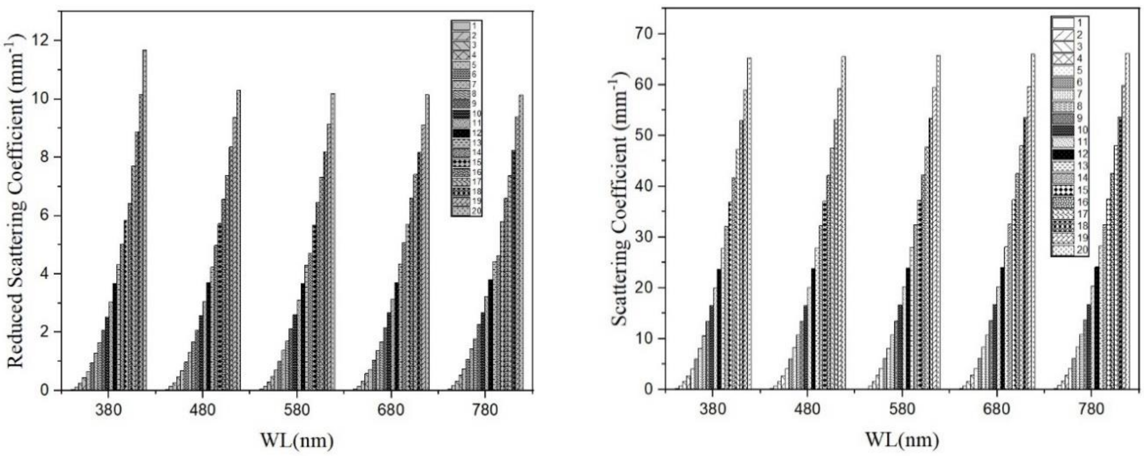

Figure 1. The scattering efficiency in the LED with different NMPF: Eu diameters from 1 µm to 20 µm

During the operation of the LED devices, the heat increase is unavoidable, which affects the color coordination. In an increasing temperature environment, from 300 K to 500 K, the NMPF: Eu phosphor exhibited a blue shift, attributed to the intensified higher energy peak and the low energy peak's quenching at elevated temperatures. Therefore, the NMPF: Eu phosphor proves to have remarkable chromatic stability within the typical examined temperature range in an LED device. This indicates that using the NMPF: Eu phosphor can improve the chromatic uniformity of the LED package [16].

The correlated color temperature (CCT) range and CCT delta assessments were carried out with different sizes of NMPF: Eu phosphor particles. The results are depicted in Figures 2 and 3, respectively. Overall, increasing the particle size of NMPF: Eu does not absolutely benefit the color uniformity as this somewhat induces the CCT delta, the average difference between the highest CCT and lowest CCT points of an angular CCT range. There is a notable fluctuation in CCT ranges of the LED when increasing the particle size of NMPF: Eu phosphor. When increasing the NMPF: Eu particle size an increment from 1 µm to 2 µm, the CCT delta bottoms out with a value of ~25 K. This means the color difference on the chroma scale is insignificant, resulting in the more satisfying color uniformity. As the NMPF: Eu particle size increases, the CCT delta also increases and peaks at ~225 K when NMPF: Eu diameter is 7 µm. Continuing enlarging the demonstrates a significant drop in CCT delta, but the values are still higher compared to that obtained with 2 µm NMPF: Eu particles. So, to achieve the best color uniformity, using the NMPF: Eu phosphor with a particle size of 2 µm seems to be an ideal option.

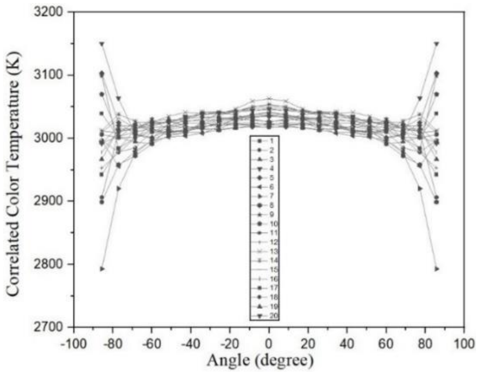

Figure 2. Angular CCT range of the LED emission with different NMPF: Eu diameters from 1 µm to 20 µm

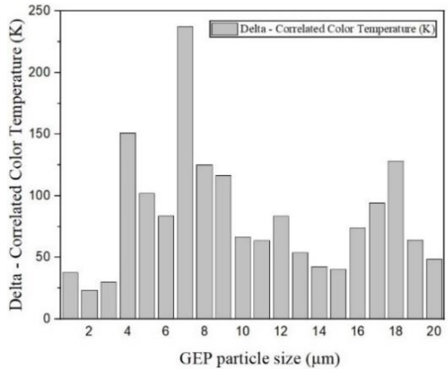

Figure 3. CCT delta of the LED emission with different NMPF: Eu diameters from 1 µm to 20 µm

The fluctuation and increased CCT delta could be ascribed to the changes in the transmission peak intensity as a function of altering NMPF: Eu diameters. The transmission peaks of the LED with different NMPF: Eu diameters from 1 µm to 20 µm are displayed in Figure 4. The difference when increasing the NMPF: Eu diameter from 1 µm to 2 µm is noticeable, especially in the region of 550-635 nm. With the particle size larger than 2 µm, the peak intensity does not alter much. Besides, the peak at 590 nm (yellow) is much more intense than that at the 450 nm (blue). This result indicate that the converted yellow light power is stronger than the blue one. The change in YAG: Ce concentration on the increasing NMPF: Eu granule diameters, shown in Figure 5, can demonstrate this phenomenon. When the bigger NMPF: Eu particles are added, given that the NMPF: Eu concentration is fixed, the concentration of YAG: Ce yellow phosphor increases. The highest of YAG: Ce amounts are recorded when NMPF: Eu particle size ranges from 5-7 µm. With NMPF: Eu particles larger than 7 µm, the YAG: Ce concentration slightly declines but is greater than that with 1-2 µm NMPF: Eu particles. The increase in YAG: Ce amounts promote its absorption and conversion of blue light emitted from NMPF: Eu particles, leading to the notable intensity in transmission power [17].

The luminous flux of the LED as a function of altering the NMPF: Eu diameters is subsequently shown in Figure 6. The increasing NMPF: Eu diameters is beneficial to the luminosity of the white LED, according from the demonstrated data. The luminous intensity gradually increases with the NMPF: Eu particle-size enlargement, reaching its peak when NMPF: Eu particle size is 10 µm. The decline in luminosity is also noticed with the diameter more than 10 µm, but the intensity is far more significant compared to the result in the case of 1 µm NMPF: Eu particle size. Notably, with larger particle sizes, the light transformation between blue and yellow or red-orange is amplified, owing to the broader phosphor sheet associated with larger particles. Consequently, the overall spectral energy decreases. Moreover, in the case of substantial particle size, the transformed light may experience rear-reflection, leading to a reduction in luminescent intensity and an increase in the CCT level [18].

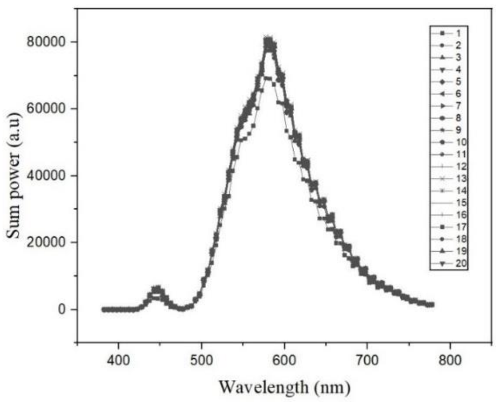

Figure 4. Transmission spectral power of the LED emission with different NMPF: Eu diameters (1-20 µm)

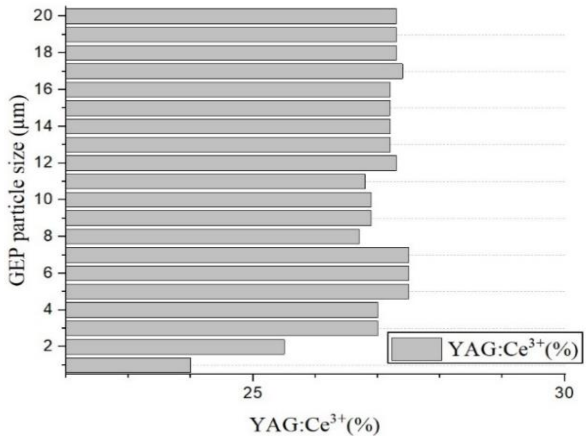

Figure 5. YAG: Ce of the LED emission with different NMPF: Eu diameters from 1 µm to 20 µm

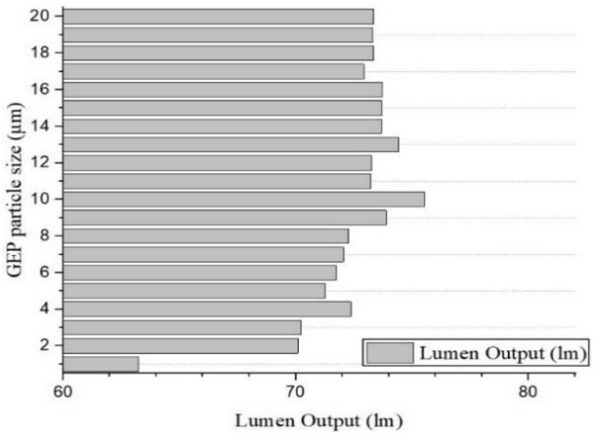

Figure 6. Luminous flux of the LED emission with different NMPF: Eu diameters from 1 µm to 20 µm

To evaluate how illumination conveys hues to our visual perception, the CRI has traditionally been employed, assessing illumination based on sunlight standards. While CRI has served as a longstanding indicator of hue quality and is suitable for older lighting technologies like incandescent and fluorescent lights, its limited coverage of only eight hue samples makes it inadequate for modern devices, especially LEDs, which encompass a broader range of hues. Consequently, CRI cannot accurately evaluate the chromatic performance of LED devices. To address this limitation, a more comprehensive index called the color quality scale (CQS) was introduced [19]–[21]. Unlike its predecessor, CQS incorporates multiple variations in chromatic quality, encompassing aspects such as hue, brightness, and saturation, enabling a more inclusive assessment of hue output. With these additional factors considered, CQS emerges as a much more appropriate and reliable index for evaluating light quality in white LED devices.

The resulted CRI and CQS in connection to the NMPF: Eu diameters of 1-20 µm are presented in Figures 7 and 8. Increasing the NMPF: Eu diameters generally promotes the rendering performance of the LED white light. The CRI increases when the NMPF: Eu diameter is enlarged from 1 µm to 2 µm. After that, the values remain quite stable regardless of using bigger NMPF: Eu particles. In terms of the CQS, the enhancement with larger NMPF: Eu particles is more notable. With the utilization of 13 µm NMPF: Eu particles, the best CQS value is obtained. The CQS value recorded in the case of 9 µm NMPF: Eu particles is in the second place, which is slightly lower than the peak. The notable CQS values with such NMPF: Eu particl sizes can be attributed to the increase CRIs and the better color coordination (low CCT delta). Thus, the larger NMPF: Eu particles can be applied to enhance the color rendering efficiency of the white LED package [22]–[25].

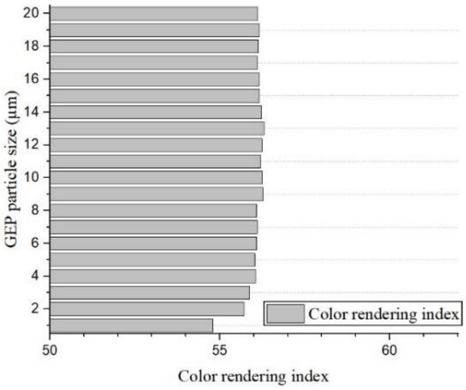

Figure 7. CRI performance of the LED emission with different NMPF: Eu diameters from 1 µm to 20 µm

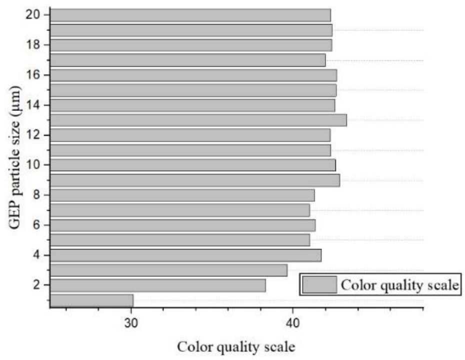

Figure 8. CQS performance of the LED emission with different NMPF: Eu diameters from 1 µm to 20 µm

4. CONCLUSION

This research introduces a novel blue phosphor, NMPF: Eu, specifically designed for violet 405 nm LED devices. The compound is synthesized through a one-step sintering solid-state reaction. Significantly, NMPF: Eu demonstrates highly efficient and thermally stable blue emission under violet light excitation. We developed a white LED device by combining this phosphor with a 405 nm LED chip and YAG: Ce yellow phosphor. Through a comprehensive investigation, we explored the impacts of varying NMPF: Eu phosphor particle sizes. The increasing phosphor particle size resulting in stronger scattering performance, enabling the improvement of CRI, CQS and luminous properties. Besides, the blue emission intensity within the white light range is lower than that of the yellow emission owing to the higher concentration of YAG: Ce phosphor, leading greater blue-light utilization by the yellow phosphor. These outcomes underscore the remarkable suitability and potential of NMPF: Eu as a promising phosphor for expanding violet LED applications. This research contributes to the advancement of energy-efficient LED lighting technology and opens new avenues for its practical implementation in various lighting scenarios.

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