RED PHOSPHOR COLOR-CONVERTED FILM WITH TIO_2 PRODUCING WHITE LIGHT EMITTING DIODES

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Abstract. *The $Na_2GdMg_2(VO_4)_3$ (NGMV) has proved to be an exceptional substrate for the doping of trivalent europium ions to produce a novel red phosphor. The paper presents NEuGMV red phosphors, synthesized via the conventional solid-state method, with a focus on evaluating their photoluminescence performance. The obtained results emphasize the latent applications of NEuGMV red phosphors in the context of warm white light emitting diodes (WLEDs). To further investigating the use of NEuGMV red phosphors in developing WLED lighting, a color-converted film, $NEuGMV@TiO_2$, is developed by incorporating TiO_2 scattering particles and NEuGMV with silicone gel. The relationship between $NEuGMV@TiO_2$ and the blue-LED-chip emitted light is explored, with particular attention to the impact of varying TiO_2 doping concentrations. Introduction of the $NEuGMV@TiO_2$ composite introduces a distinct 590-nm red emission peak in the produced light spectrum, effectively mitigating color variation. However, excessive TiO_2 content is observed to diminish the total lumen output due to intense scattered light, thereby elevating the likelihood of light loss. Nonetheless, optimal TiO_2 incorporation leads to the demonstration of $NEuGMV@TiO_2$ potential as a tunable red-luminescent layer within the WLED system.*

Keywords: *White LED; Lambert-Beer law; Color rendering index; Luminous efficacy.*

1. Introduction

In recent times, the significance of phosphor-transmuted white light emitting diodes (pc-WLEDs) has been magnified for their exceptional attributes, encompassing eco-friendliness, heightened luminescent efficacy, prolonged operational lifespan, and cost-effectiveness [1, 2]. Typically, commercially available pc-WLEDs are synthesized by integrating blue chips with $Y_3Al_5O_{12} : Ce^{3+}$ (YCeAG) yellow phosphors. Nevertheless, these configurations raise certain conspicuous inquiries, notably pertaining to the deficient red component, resulting in a low color rendering index and an elevated correlated color temperature (CCT) higher than 4500 K. An alternative approach for generating white light involves employing near ultraviolet (UV) chips in combination with three-color phosphor components (blue, red, and green) [3, 4]. Accordingly, the utilization of red phosphor endowed with remarkable luminescent characteristics to achieve the warmer and high-CRI white light. A multitude of endeavors have been documented in the exploration of innovative red phosphors, exemplified by $K_2TiF_6 : Mn^{4+}$. However, studies showed that Mn^{4+} primary emission range lies within 650–670 nm, a range with limited sensitivity to the human visual system [5,6]. In contrast, the rare earth ion Eu3+ presents itself as a promising alternative as a red-emitting center, primarily emitting within the 600–630 nm range, which aligns well with human visual sensitivity.

Despite its potential, Eu^{3+} experiences feeble absorption through $4f - 4f$ transitions due to spin-forbidden processes. This limitation can be effectively surmounted by the introduction of sensitizers, for instances, Bi^{3+} and Cu^{+}. Apart from that, the $VO_4^(- 3)$ ion in vanadate phosphor is also a potential sensitizer for Eu^{3+}, making this phosphor an excellent substrate for integrating trivalent europium. Additionally, the emission spectrum band of $VO_4^(- 3)$ cover 400-850 nm wavelength range, offering a feasible means of reinforcing the luminescent output of Eu3+ through energy transfer (ET). Within the realm of vanadate phosphors, the $Na_2GdMg_2(VO_4)_3$ (NGMV) turns out as an exceptional substrate for the doping of rare-earth ions owing to its the congruence of valences and closely matched ionic radii between Gd^{3+} and the trivalent rare-earth ions. This judicious choice holds the promise of facilitating robust rare-earth doping strategies, fostering an environment conducive to remarkable luminescent properties and enhanced performance. In light of this, the exploration of novel red phosphors incorporating the component $NGMV : Eu^{3+}$ (NEuGMV) holds substantial promise [7,8].

This study utilizes the red phosphors NEuGMV, synthesized using the conventional solid-state methodology. An examination of their photoluminescence performance forms the crux of our investigation. The findings underscore the latent potential of NEuGMV red phosphors in applications involving warm WLEDs [9–11]. Subsequently, we make a color-converted compound comprising NEuGMV, YCeAG yellow phosphor, TiO_2 scattering particles, and silicone gel to transform the blue-LED-chip emitted light. We named this compound as $NEuGMV@TiO_2$, hereafter. Among particles, TiO_2 was chosen for its combination of multiple qualities, including chemical consistency, photocatalytic capability and biocompatibility. It is capable of harnessing UV illumination and its chemical consistency provides resistance against degeneration when subject to severe conditions. It is also non-toxic and inexpensive, suitable for mass production and use. The addition of TiO_2 aims at enhancing the blue-light utilization of the phosphor compound. The work feasibly monitors the WLED's lighting performance through varying the doping concentration of TiO_2. The introduction of NEuGMV@TiO2 compound induces the emerge of 590-nm red emission peak in the generated light spectrum, which contribute to reduce the color variation. However, the increases of TiO_2 amount causes the total lumen output to decrease owing to excessive scattered light scattering, leading to high probability of light loss [12,13]. Yet, with proper amount of TiO_2, the $NEuGMV@TiO_2$ prove their capability as a tunable red-luminescent layer of the WLED.

2. Experimental

NEuGMV phosphor was synthesized using a high-temperature solid-state method. The concentration of Eu^{3+} was determined at 0.7. The high-purity initial constituents, comprising Na_2CO_3, $MgCO_3$, NH_4VO_3, Gd_2O_3, and Eu_2O_3, were meticulously weighed according to the stoichiometric proportions. Subsequently, the components were mixed by grinding with alcohol in an agate mortar for 30 minutes. The resulting mixtures underwent two controlled sintering phases at 500^oC and 750^oC ambient air conditions; each phase lasts 5 hours. Following sintering, the obtained compounds were re-ground for subsequent investigations. The phosphor's fluorescence properties were investigated at room temperature, involving emission spectra, excitation spectra, fluorescence decay curves, and temperature-dependent emission spectra (the temperature values were from 300 K to 570 K). These measurements were conducted utilizing an Edinburgh FS5 fluorescence spectrometer equipped with a 150W Xenon lamp, a nanosecond flashlamp (nf900), and a temperature control unit [14,15].

3. Results and Discussion

Monitoring the NEuGMV phosphor under the excitation wavelength of 356 nm, the collected emission spectrum showed broad coverage, including several peaks at 590 nm, 610 nm, 652 nm, and 705 nm, originated from the Eu^{3+} transitions from $^5D_0$ to $^7F_1$, $^7F_2$, $^7F_3$, and $^7F_4$, respectively. Among those peaks, the most intense one is located at 610 nm. Subsequently, the excitation band of the NEuGMV observed with the 610 nm emission also broad with several peaks centered at 356 nm, 394 nm, and 465 nm. The 356 nm excitation peak was resulted from the $VO_4^{(-3)}$ while the other two was induced from the Eu^{3+} transition from $^7F_0$ to $^5L_6$ and $^5D_2$, respectively. These results indicate that it is possible to excite our phosphor with n-UV (356 nm) or blue-LED chip (465 nm).

$$ETE = 1 - \frac{I_p}{I_a} \tag{1}$$

where the Ip and Ia indicate the $VO_4^{(-3)}$ emission intensities with and without Eu^{3+} dopant in the NEuGMV phosphor. The ETE with 0.7 Eu^{3+} concentration was about 89 %, proving the efficient energy transfer between $VO_4^{(-3)}$ and Eu^{3+} in the NEuGMV phosphor. In particular, upon excitation by near UV light, the $VO_4^{(-3)}$ ions undergo an energy transfer process, transitioning from the $^1A_1$ ground state to the $^1T_2/^1T_1$ excited states. Subsequent to non-radiative relaxation, these $VO_4^{(-3)}$ ions emit a green-yellow broad band of light, facilitated by transitions from the excited states of $^3T_2/^3T_1$ back to the ground $^1A_1$ state. Notably, a portion of the energy from the $VO_4^{(-3)}$ is effectively transferred to proximal Eu^{3+} ions, inducing the $^7F_0 \rightarrow {}^5D_0$ transition. As a result, a series of distinct emissions at wavelengths of 590 nm, 610 nm, 652 nm, and 705 nm was generated, corresponding to the Eu^{3+} transitions from $^5D_0$ to $^7F_1$, $^7F_2$, $^7F_3$, and $^7F_4$.

When the $NEuGMV@TiO_2$ compound was applied to make the pc-WLED package, it is expected to obtain the emission peaks in red region. Subsequently, the generated spectrum of the WLED in presence of $NEuGMV@TiO_2$ is displayed in Figure 1. It can be easily observed the presence of the narrow 450-465 nm and wide 500-600 nm bands. The emission peaks change with the variable TiO_2 concentration. At low 10 wt.% TiO_2, the shoulder peak at 465 nm and two sharper peaks at about 545 nm and 590 nm are exhibited, attributed to the blue LED chip, YCeAG yellow phosphor, and NEuGMV red phosphor. When TiO_2 concentration increases to 20 wt.% and more than that, the small peak at 525 nm is presented, while the 465 nm peak intensity decline significantly. In addition, not only does the blue peak intensity decrease but also the whole band 500-600 nm intensity. Such a decrease in spectral intensity leads to the reduction in lumen output for our WLED. Figure 2 displays our WLED's radiance with increasing TiO_2 amount in the $NEuGMV@TiO_2$ compound, and the higher the TiO_2, the lower the resulting luminosity [16]. Lumen output, which is used to judge visible brightness, cannot be directly controlled by TiO_2 since it does not discharge light. Regardless, its particle size would have a significant impact on the efficiency with which light may traverse and escape a material or device, particularly in situations that use phosphor samples. Tinier TiO_2 granules tend to scatter at lower wavelength levels, widening the light pathway inside the phosphor latticework. As a result, photonic transmutation efficiency will improve, increasing lumen output. Larger granules may spread with more uniformity across wavelength levels, helping illumination in escaping with low intrinsic absorptivity, benefiting high-powered devices. When grains get too small, excessive dispersion can limit light inside a setting, creating rear-reflection or illumination penalty and reducing luminosity. Granules of appropriate size would mitigate these drawbacks while maintaining considerable lumen proficiency. Refined granules outperform phosphor samples in terms of dispersion, resulting in a more homogeneous lighting sheet, avoiding brightness hotspots and improving lumen consistency. Unusual or consolidated granules may provide variable brightness, demanding extra phosphor to achieve an equivalent lumen. The amount of TiO_2 can alter thermic conductivity in compound situations. Improper thermic diffusion induced by large, incorrectly loaded granules may degrade phosphor samples, progressively reducing brightness. Tiny grains generally help to stabilize thermic state while retaining lumen production. The change in spectral peaks and transmission intensity could be ascribed to the scattering property influenced by variable TiO_2 concentration. The reduced scattering coefficients (RSC) for $NEuGMV@TiO_2$ under different TiO_2 dosages are shown in Figure 3. When the integrating amount of TiO_2 in the compound becomes higher, the RSC values increase regardless of the examined wavelengths. Additionally, the longer wavelengths demonstrate the better RSC, indicating that the phosphor compound can effectively scatter and redirect the converted lights. The induced blue radiance scattering encourages blue-ray absorptivity and conversion to longer light wavelengths of the $NEuGMV@TiO_2$ phosphor sheet, according to Mie scattering. In this way, the yellow and red phosphor can easily convert the incident light, resulting in more emission peaks of yellow-green and red regions [17–19]. However, with such a strong scattering effect, the blue emission reduces owing to strong absorption of the phosphors. Besides, the higher TiO_2 leads to the higher probability of re-absorption and even self-absorption of the phosphor material. Moreover, when amount of TiO_2 becomes higher, the thickness of the phosphor sheet or the particle density increases, leading to the deficient light extraction. In this case, we fixed the phosphor layer at 0.08 mm, so the increasing TiO_2 amount results in higher particle density in the phosphor sheet. Furthermore, this dense phosphor film possibility gives a rise to the color temperature of the WLED. Therefore, to reduce such problems, the YCeAG yellow phosphor concentration must be reduced on the increasing TiO_2 concentration (given that the NEuGMV amount is fixed), as demonstrated in Figure 4. Furthermore, in the evaluation of color rendering efficiency within the framework of the pc-WLED, we conducted an analysis encompassing both chroma quality scale (CQS) along with chroma rendition indicator (CRI), as depicted in Figure 5. Notably, the CQS serves as an expansive metric for gauging color reproduction,

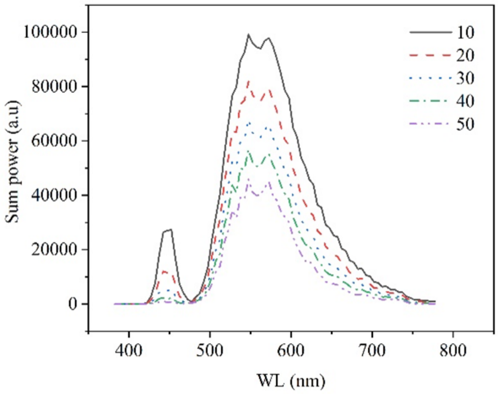

Fig. 1: The generated spectrum with variable TiO_2 amount in $NEuGMV@TiO_2$.

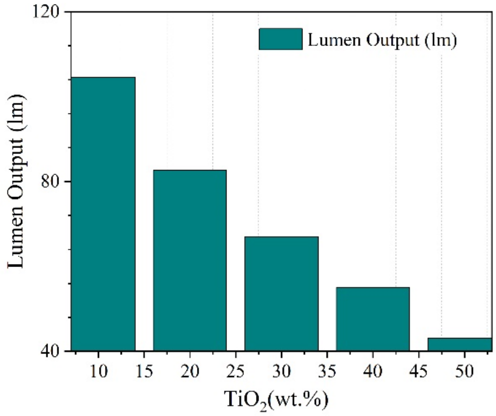

Fig. 2: Lumen output with variable TiO_2 amount in $NEuGMV@TiO_2$.

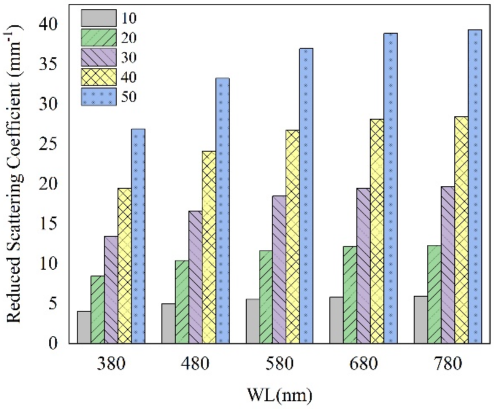

Fig. 3: Reduced scattering coefficients at corresponding wavelengths with variable TiO_2 amount.

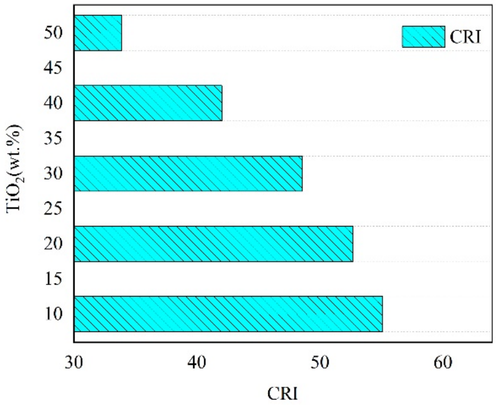

Fig. 4: YCeAG yellow phosphor concentration with variable TiO_2 amount.

surpassing the constraints of the conventional CRI approach. This particular assessment is of substantial value when appraising color fidelity in the context of solid-state lighting, particularly LEDs. The CQS accounts for an array of factors, including hue preservation, chroma enhancement, gamut area index, and gamut shape index, providing a nuanced and comprehensive evaluation of the color performance of a light source and serving as an invaluable tool for optimization [20–22]. As presented in Figure 5a for CQS and Figure 5b for CRI, both metrics exhibit a downward trend with increasing TiO_2 concentration from 10 wt.% to 50 wt.%. While the incorporation of $NEuGMV@TiO_2$ results in a relatively broadened spectral range spanning from blue to orange, the observed reduction in both CQS and CRI with elevated TiO_2 content may be attributed to an imbalance in color distribution [23, 24]. As demonstrated, the higher TiO_2 concentration leads to the diminishing proportion of blue color, and the light chroma gradually change from green to yellow and further to red.

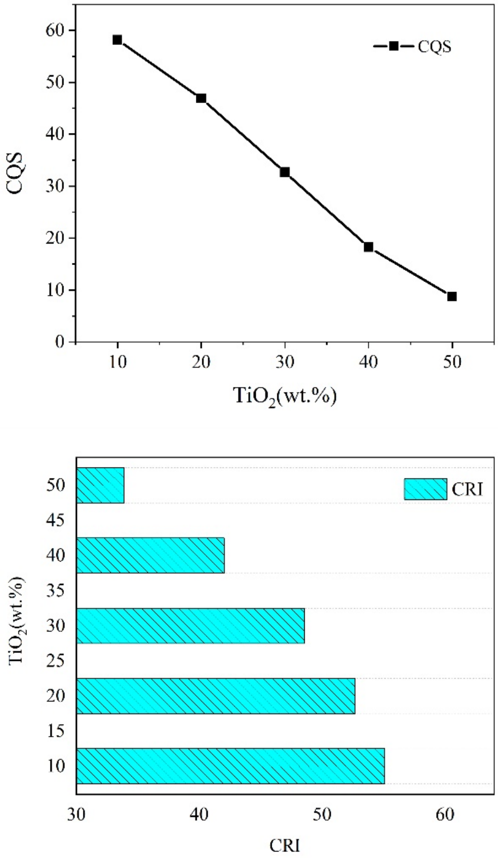

Fig. 5: The color rendering efficiency with different TiO_2 amounts in $NEuGMV@TiO_2$: (a) CQS and (b) CRI.

However, the introduction of NEuGMV@TiO2 contributes to achieving a warmer white light emission with a correlated chromatic temperature approximately within the range of 3875-4050 K, as illustrated in Figure 6 [25, 26]. The CCT span increases (75-208 K) when the TiO_2 concentration moves toward to 30 wt.%. However, as the TiO_2 continues increasing to 50 wt.%, the CCT range decline to 83 K, very close to the reference value (75 K with 0 wt.% TiO_2). Moreover, at the direct viewing angle, the CCT at 50 wt.% TiO_2 is lower than the reference CCT. These results indicate that $NEuGMV@TiO_2$ with 50 wt% TiO_2 helps to obtain lower CCT for warm WLED light. In addition to the general CCT range, the variation of CCT (delta-CCT) regarding variable TiO_2 is taken into account and exhibited in Figure 7. This delta-CCT value, indicative of color uniformity, reflects the disparity among the maximal and minimal CCT outcomes. Lower delta-CCT values correspond to enhanced uniformity. Although TiO_2 does not discharge illumination, its particle size may significantly influence how illumination is spread and viewed [27]. Tinier granules improve Rayleigh dispersion, allowing blue light to be dispersed more effectively. As a result, the CCT would increase, giving illumination a cooler color. Larger granules incline towards Mie dispersion, requiring less wavelength and producing warmer light due to less blue dispersion [28]. Greater dispersion via refined granules may prolong the optic route inside the phosphor sheet, allowing more blue illumination to be digested and released as yellow or red, lowering CCT and producing warmer illumination [29]. Refined granules improve rear-reflection, increasing light uniformity while eliminating hotspots. Uniform diffusion for smaller granules aids in achieving a homogeneous chroma, reducing chroma variation over the exterior. Inconsistent particle sizes can lead to a lack of spectrum balance and inconsistent CCT. The level of variation in CCT demonstrates a gradual increase upon the rising TiO_2 content, reaching a maximum at 30 wt.%. Beyond 30 wt.% of TiO_2, the delta CCT exhibits a noticeable decrease, with the lowest value collected at 50 wt.% TiO_2. Thus, the introduction of $NEuGMV@TiO_2$ also exhibits potential in attaining consistent color distribution within the WLED structure.

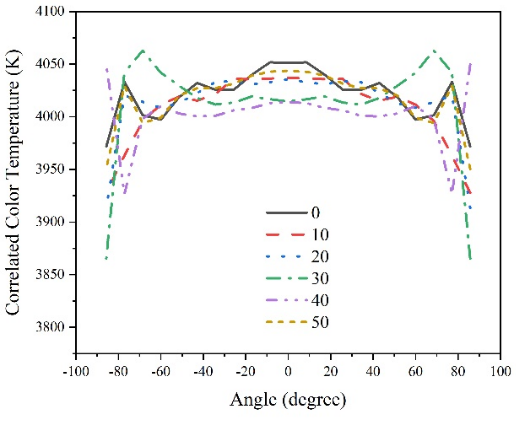

Fig. 6: CCT levels with various TiO_2 amounts in $GD@TiO_2$ compound.

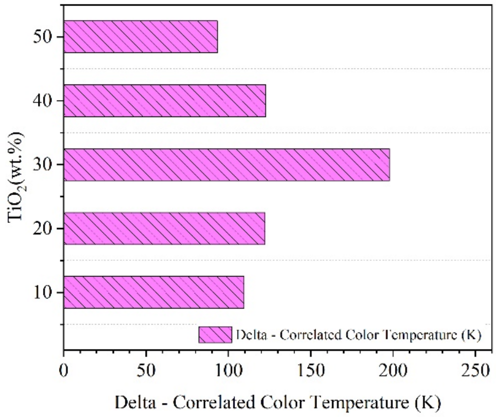

Fig. 7: CCT delta with various TiO_2 amounts in $GD@TiO_2$ compound.

4. Conclusions

In conclusion, this investigation harnessed the potential of NEuGMV red phosphors, synthesized through the conventional solid-state approach. The results illuminate the untapped prospects held by NEuGMV red phosphors, particularly within the realm of warm WLEDs. A color-converted compound named $NEuGMV@TiO_2$ was developed, encompassing NEuGMV, YCeAG yellow phosphor, TiO_2 scattering particles, and silicone gel, for a blue-pumped LED. The incorporation of TiO_2 contributes to achieving the enhancement in blue-light utilization through improving the scattering property of the phosphor sheet. Throughout this work, the WLED's lighting performance are examined using variable TiO_2 doping concentration. The introduction of the $NEuGMV@TiO_2$ compound instigated the emergence of a distinct 590-nm red emission peak, in addition to the 465 nm blue and 545 nm yellow-green peaks. However, higher TiO_2 content led to a decrease in total lumen output due to higher self-absorption of the phosphor materials. Nonetheless, the NEuGMV@TiO2 introduction helps accomplish the warmer white light emission and proves to be potential in attaining consistent color distribution. Overall, the $NEuGMV@TiO_2$ compound can act as a promising tunable red-luminescent layer within the WLED system, thereby offering new avenues for enhanced lighting technology.

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