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Effects of TiO_2 in graphene-quantum-dot film on lighting color uniformity of a white light-emitting diodes

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ABSTRACT

Improvement in color uniformity of white light-emitting diodes (WLED) is one of the imperative goals for high-quality solid-state illumination. The conventional WLED model with a single yellow phosphor $YAG:Ce3^{+}$ and blue LED chip does not provide good color distribution and often has a yellow-ring phenomenon. Using a diffusing layer to induce scattering light efficiency can address this issue for such a conventional WLED package. A diffusing layer comprised of graphene quantum dots (GDs) and TiO_2 (TiO_2@GD) is proposed to fulfill this goal. The TiO_2@GD composites prove to possess excellent biocompatibility, low toxicity, and thermal and chemical stability, holding great potential in high-power WLED production. By maintaining a constant GDs content of 10 wt%, the research explores the impact of varying TiO_2 doping concentrations on the lighting performance of the WLEDs via the mean of light scattering. The TiO_2@GD layer also induces a red-shift in the emitted light spectrum, contributing to a reduction in color variation. While a decline in luminosity and color rendering performance becomes evident with excessive TiO_2 content, the study underscores the potential of TiO_2@GD as a viable diffusing layer for LEDs to obtain improved angular uniformity of color distribution.

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1. INTRODUCTION

The immense attention has been given to the white light emitting diodes (LED) as it can be a highly promising future light source due to their remarkable attributes encompassing elevated lumen efficiency, energy-saving performance, extended operational lifespan [1], [2]. Prior attempts at achieving white light emission have typically relied on amalgamating separate dopants or multiple components, inevitably resulting in phase separation and undesirable color variation. In general, there are two researched principal approaches to generate white light. The first approach involves the direct incorporation of luminescent materials as the emitting component, necessitating meticulous fine-tuning of device architecture and technology. The second approach entails employing light-emitting substances as color conversion layers within the LED chip. This alternative method, focusing on the color conversion layer, offers relative simplicity. One of the materials used to fabricate the conversion layer in the LED package is the yellow-emission $YAG:Ce^{3+}$ phosphor. The luminescence power of this phosphor-converting white LED was good, but the color distribution was inferior, limiting the cross-field utilization of the devices [3]-[5]. To address the persistent color related temperature (CCT) non-uniformity, several methods were proposed, commonly including conformal dispensing phosphor or freeform lens. However, the large absorption of blue light, the inconvenience in adjusting the phosphor film breadth in conventional fabrication process, and lack of insight in wide-angle CCT distribution hinder the novelty of such approaches [6], [7]. Besides, the scattering pattern of the phosphor layer can contribute to manipulate the CCT distribution of the white light on target surfaces [8]. As a result, another approach introducing scattering stimulating factors was proposed. Integrating diffusing layer above the phosphor film and doping scattering particles into the phosphor layer were considered the simple and effective techniques [9], [10].

The use of scattering particles did show improvement but white LEDs (WLEDs) produced with organic materials, despite being cost-effective and amenable to large-scale production, suffer from inherent deficiencies pertaining to environmental stability and device longevity. Hence, the scattering-stimulated phosphor layer's performance would decline after extended and high-frequency operation. In this situation, using a diffusing layer comprised of good resistance and high efficiency materials can be the potential alternative. Intriguingly, semiconductor nanocrystals, notably semiconductor quantum dots, has emerged as a promising candidate owing to their exceptional quantum yield, narrow-bandwidth emission, and resistance to photobleaching, effectively circumventing the constraints associated with whole-organic-material-based WLEDs [11], [12]. Among various types of semiconductor quantum dots, graphene quantum dots (referred to as graphene dots or GDs) hold significant promise across diverse domains, including chemical sensing, catalysis, and bioimaging [13]. The major attribute for their wide-application domain is their diminished toxicity that often encounters in original heavy-metal doped quantum dots while holding remarkable biocompatibility and impressive photoluminescence (PL) and exceptional surface grafting capabilities. These properties position GDs as appealing candidates for integration into optoelectronic devices. Notably, the potential substitution of rare-earth phosphors in white LEDs emerges as a compelling application for GDs. However, the utilization of GDs in LED technology has been associated with certain drawbacks, notably limited luminance and insufficient current density [14]. Nevertheless, GDs exhibits certain limitations, such as a narrow bandgap, low efficiency electron and heat transfer properties akin to TiO_2 and ZnO semiconductors, and not having the capability to donate metal ions for biocidal applications [15], [16].

Thus, the synergistic amalgamation of at least two dissimilar nanomaterials, exemplified by TiO_2 and graphene-based materials, each endowed with distinct properties and complementary functionalities, presents a promising avenue for fulfilling the burgeoning demands of energy and biomedical applications [17], [18]. These hybrid materials, termed nanocomposites, entail the combination of two or more components, at least one of which possesses a nanoscale structure. Of particular interest, the integration of TiO_2 with GDs unveils novel prospects in cross-field application relating photocatalysis, attributed to the materials adaptable structure, remarkable mobility of charge carriers at ambient conditions, elevated thermal and electrical conductivities, robust chemical stability, and the extension of UV absorption into the visible wavelength range [19], [20]. Based on the achievements of previous studies, this paper demonstrates the diffusing TiO_2@GDs layer to achieve the goal of enhancing the color uniformity of the conventional WLED device. This study focuses on the effects of TiO_2 concentration in the diffusing layer to regulate the CCT distribution and lumen output of the WLED model originally built with YAG:Ce^{3+} phosphor and blue LED dies. The work monitors the WLED's lighting performance through varying the doping concentration of TiO_2 while GDs amount is constant at 10 wt%. With the variation of TiO_2 dose, the light scattering is regulated, impacting the light quality of the WLED. The introduction of TiO_2@GDs layer leading to the red-shift in the generated light spectrum, which contribute to reduce the color variation. Though the reduction of luminosity and rendered color performance is noticed when TiO_2 amount surpasses a certain level, the GD@TiO_2 prove their potential serving as a luminescent layer of the LED [21], [22].

2. METHOD

The TiO_2@GDs nanocomposite was prepared using the hydrothermal route [23]. The GDs was synthesized via the microwave method while the TiO_2 particles was bought from Degussa (Germany). The obtained microwaved GDs exhibits green-yellow emission. The luminescence data of the TiO_2@GDs were collected through ultraviolet-visible (UV-vis) spectroscopy as well as photoluminescence spectra. The scattering measurement and simulation was carried out with the Mie-scattering theory and MATLAB program. In the pursuit of our research objectives, the yellow-green GDs were prepared with the following process. Initially, we introduced 50-mg nanoscale graphite to a mixture comprising 40 mL of a HNO_3:H_2SO_4 (1:3) acid solution [24], [25]. Following this, a 2-hour session of ultrasonication was administered to the solutions. The resulting dispersion underwent a 4-hour reaction at 100 °C, facilitated by microwave irradiation. Subsequently, the dispersion was subjected to filtration through a microporous membrane with a pore size of 220 nm, and subsequent neutralization was executed using sodium carbonate. To achieve the desired consistency, an infusion of 160 mL of deionized water was incorporated, leading to the acquisition of the aqueous solution of GDs through dialysis utilizing a dialysis bag with a molecular weight cutoff of 1,000. Then, after modifying the GDs solution's pH level to 13.0, we took 15 mL of this solution to be microwaved at 200 °C for 8 hours. The dialysis employing a molecular weight cutoff of 1,000 was subsequently carried out, and the resulting aqueous GDs solution was meticulously stored for further examinations.

Then, the as-synthesized GDs was blended with 15 ml water and TiO_2 with different concentration was dispersed into the GDs solution. Then, the obtain solution was stirred in normal temperature condition for 4 hours. The obtained mixture was moved into a Teflon-sealed autoclave heated at 150 °C for 6 hours, followed by a 4,000-rpm centrifugation process for 5 minutes. The final product was dried 24 hours in a 50-degree-Celsius vacuum.

3. RESULTS AND DISCUSSION

The diffuse layer TiO_2@GD is used proposed to achieve the improvements in color distribution uniformity and lumen output of the conventional blue-pumped LED package. The TiO_2 content in the GD layer plays a crucial role in manifesting the light scattering performance of the layer, leading to changes and potential enhancements in color uniformity. However, the induced scattering can hinder the intensity of lumen output. Therefore, the selection of the proper TiO_2 amount is imperative to take advantage of light scattering properties while not sacrificing lumen performance significantly. To investigate the scattering influences of TiO_2 on the GDs layer emission and conversion performances, it is essential to grab the fluorescence properties of the synthesized GDs. The obtained GD samples, upon the 365-nm exciting spectrum, exhibited a wide emission profile centered around 445 nm, complemented by a relatively subdued peak at 575 nm. On the other hand, the excitation wavelength was extended to the range of 400-450 nm, a discernible reduction in GDs' fluorescent intensity was observed. Such findings present that GD samples can be excited with either near-ultraviolet or blue radiating source, indicating that the GD layer is applicable to the conventional blue-pumped white LED in this paper. The introduction of TiO_2 here not only improves the photocatalysis of the GD compound but also introduced the impacts on the scattering properties of the diffusing layer. The scattering data at different wavelengths are collected with varying TiO_2 concentration and demonstrated in Figure 1. The scattering at all light wavelengths increases, especially at longer wavelengths, as the integrated amount of TiO_2 is higher. This result indicates the capability of regulating the wide-dispersion of both incident and converted lights in the WLED. Besides, it seems that the yellow light will be scattered more, allowing greater events of light mixing between yellow and blue light rays to occur. As a result, the yellow-ring phenomenon can be effectively diminished. On the other hand, the concentration of yellow YAG:Ce^{3+} phosphor also changes when the amount of TiO_2 increase, as can be seen in Figure 2.

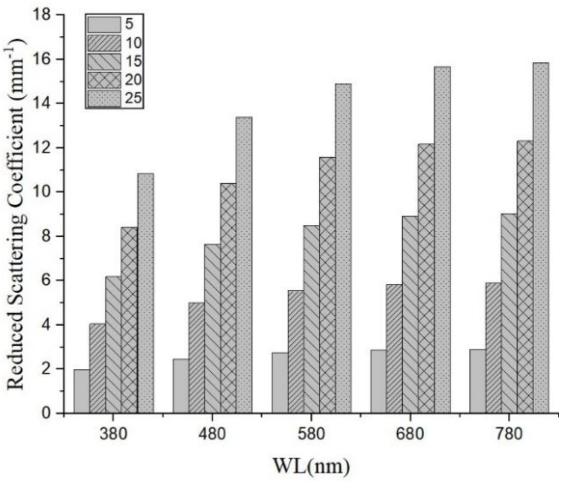


Figure 1. Scattering coefficients at corresponding wavelengths with various TiO_2 contents

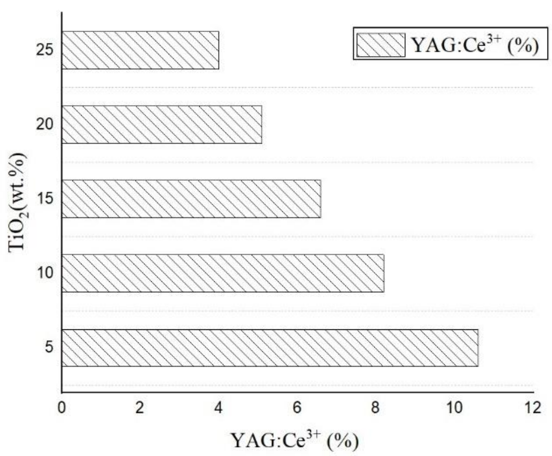


Figure 2. YAG:Ce^{3+} contents with various TiO_2 amounts

The decline in YAG:Ce^{3+} amount play a critical role in maintaining the target CCT range and reducing self-absorption effects since the scattering tends to be increasingly intense with the TiO_2 growing amount. Consequently, the resulted light emission spectrum of in the presence of TiO_2@GD compound changed with the variation of TiO_2 amount as shown in Figure 3. Overall, the emission of the WLED light included a narrow band of 445-465 nm and a broader band from ~500 nm to ~615 nm. Moreover, the eminent peaks at about 460 nm, 545 nm, and 594 nm are demonstrated. On the increasing of TiO_2 amount, the weaker emission peaks in green and red regions, at about 525 and 600 nm, are recorded in addition to the three existing peaks [26]-[28]. The 460-nm peak is originated from the blue-pumped LED, while the other peaks in the broad emission covering the green to orange wavelength can be attributed to the changed light scattering and absorption of the TiO_2@GD compound. The improved scattering with increasing TiO_2 in the compound results in the higher blue-light absorption by the GDs and the YAG:Ce phosphor, leading to the higher proportions of converted yellow-green light. Furthermore, the observed phenomenon of spectral redshift can be ascribed to the augmentation in GDs' absorption capabilities with the incident short-wavelength light. This intriguing effect becomes more pronounced when the concentration of TiO_2 within the GDs compound is heightened. In other words, the degree of absorption for shorter wavelengths surpasses that of longer wavelengths. Consequently, a substantial portion of the shorter-wavelength light undergoes absorption without emission, whereas longer wavelengths can readily traverse the resin due to their comparatively lower absorption coefficients [29], [30].

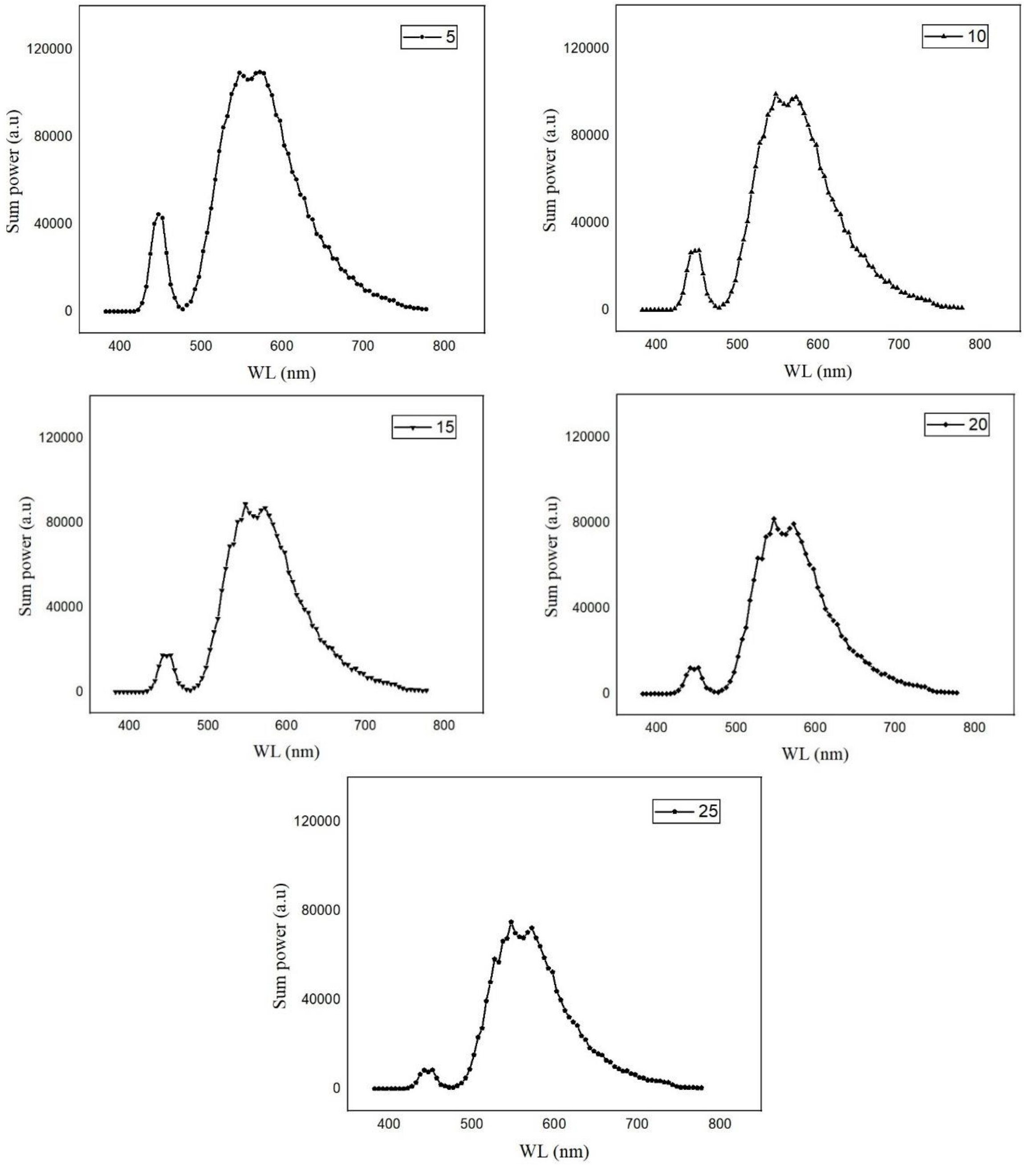


Figure 3. The emission spectrum with various TiO_2 amounts in GD@TiO_2 compound

However, as the concentration of TiO_2 increases, the emission peak intensity decreases (Figure 3), resulting in the decline of total lumen output. The lumen performance of the WLED with TiO_2@GD is shown in Figure 4, in which a continuous decline is demonstrated with the higher TiO_2. The decrease in light output is ascribed to the lower transparency of the color-conversion layer when the density of particles increases. Besides, such a declining transmission intensity indicates that backward illumination is stronger than the forward one. As a result, the light extraction is hindered, inducing light trapping and energy loss. Nevertheless, the TiO_2@GD contributes to achieving the warmer generated white light with the correlated chromatic temperature of around 3,900-4,050K, depicted in Figure 5. Additionally, the CCT distribution becomes smoother as TiO_2 weight percentage reaches 15 wt%. Beyond this TiO_2 amount, the large variation occurs between the center and the rear CCT distribution. Particularly, the CCT in $\pm 90^{\circ}$ is higher than that observed at 0° with 25 wt% TiO_2. This result shows that the light is well-dispersed in wider angles owing to the scattering property of the TiO_2@GD, but the direct light intensity (at 0°) is degraded. This finding matches the data of CCT variation (delta-CCT), as demonstrated in Figure 6. The delta-CCT, serving as a measure of color uniformity, represents the difference between the maximum and minimum CCT values. Lower delta-CCT values indicate superior uniformity. The CCT variation level gradual decrease with the TiO_2 concentration and bottoms out with 15 wt% TiO_2 before going up when TiO_2 concentration surpasses 15 wt%. Thus, GD@TiO_2 introduction is potential for achieving the consistent color distribution of the WLED. The TiO_2 content should be keep at around 15 wt% for the high performance of TiO_2@GD compound.

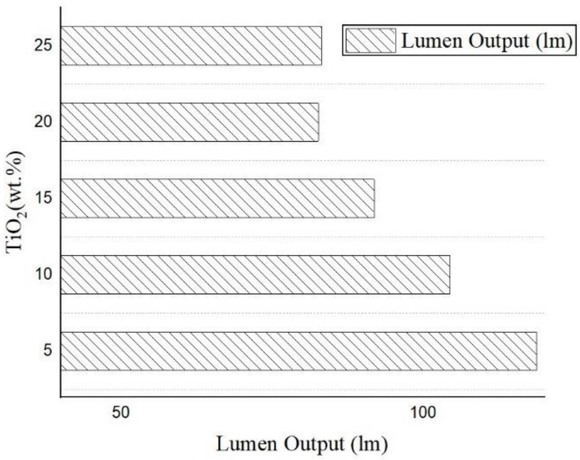


Figure 4. Lumen output with various TiO_2 amounts in GD@TiO_2 compound

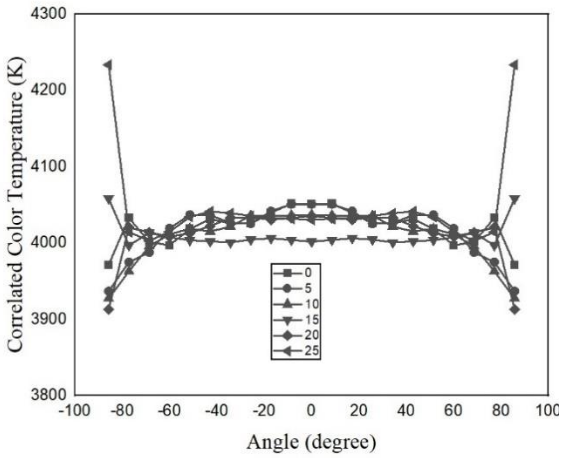


Figure 5. CCT levels with various TiO_2 amounts in GD@TiO_2 compound

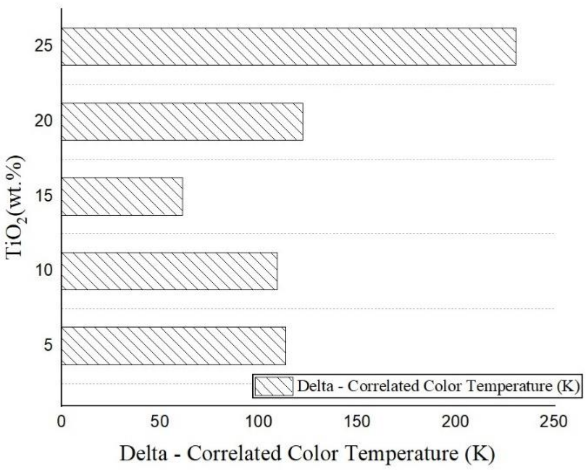
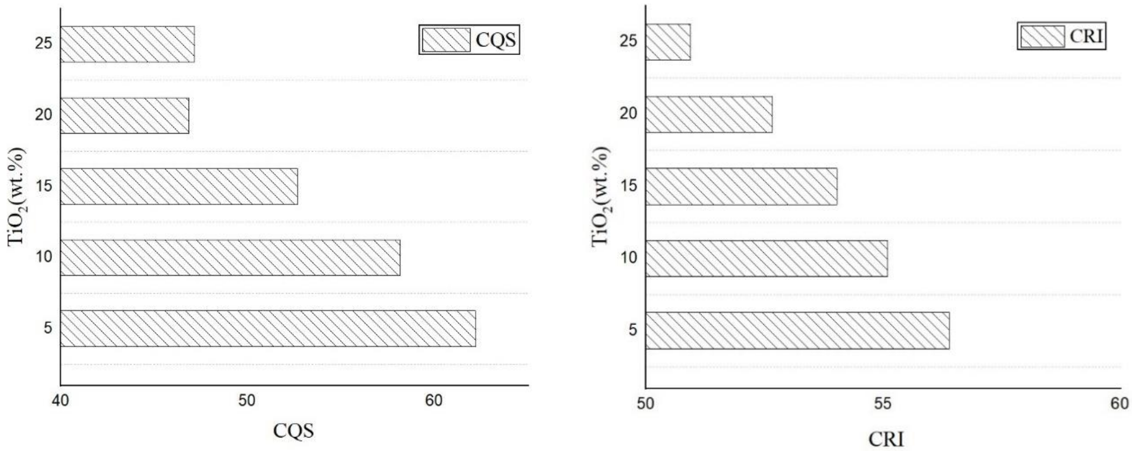


Figure 6. CCT delta with various TiO_2 amounts in GD@TiO_2 compound

In the assessment of the color rendering efficiency of WLED, we collected the data of color quality scale (shortened as CQS) and color rendering index (shortened as CRI), presented in Figure 7. Particularly, the CQS offers a comprehensive assessment of color reproduction, surpassing the limitations of traditional CRI. This metric proves particularly valuable in assessing color quality within solid-state lighting contexts such as LEDs. By accounting for numerous factors including hue preservation, chroma enhancement, gamut area index, and gamut shape index, CQS provides a nuanced understanding of a light source's color performance, serving as a valuable optimization tool. The CQS in Figure 7(a) and CRI in Figure 7(b) show the downward slopes as the TiO_2 concentration increases from 5 wt% to 25 wt%. Though the introduction of TiO_2@GD results in the relatively wide from blue to orange spectrum area, the decline in both CQS and CRI with high TiO_2 amount can be the consequence of imbalance color distribution [31]. Higher TiO_2 concentration in the compound, the amount blue color is lower than the yellow-green one. The orange-red amount is also insufficient for effective color reproduction.



(a) (b)

Figure 7. The color rendering efficiency of WLED with various TiO_2 amounts in GD@TiO_2 compound (a) CQS and (b) CRI

The findings in this study support our hypothesis of utilizing the scattering properties of TiO_2 to accomplish better scattering efficiency for the GD diffusing layer, by which the improvements in color uniformity of the white light. The increasing TiO_2 concentration leads to the stronger scattering of light at both short and long wavelengths. Among five concentration levels of TiO_2 used in the examination, 15 wt% is determined as optimal to obtain the smallest color deviation for the best color uniformity. However, the drawback is that the luminosity of the WLED declines with the same TiO_2 amount (15 wt%), which does not satisfy this study's initial goal. The increased backscattering effect and reabsorption by the phosphor and substrate can be attributed to this decrease in lumen output [32]. This result matches the findings in previous studies about light scattering effects on LED's transmission output. In short, the scattering by TiO_2 demonstrated in this study is beneficial to the light-distribution uniformity of the conventional white LED. This provides the manufacturers with another potential approach to enhance their white LED devices, especially ones utilizing quantum dots [33], [34].

However, the scattering is also significantly influenced by the particle size of the materials, and this parameter is not investigated in this paper. This study also does not reach the improvement in color rendering factors when increasing TiO_2 concentration. As a result, the study has just addressed a critical issue in efforts to enhance overall WLED's light color quality. Such limitations open other roads for further research in future, possibly combining TiO_2 particles with other materials to achieve a wider color spectrum while taking advantage of light scattering to uniformly disperse light components across viewing angles. Restructuring the LED's components such as the diffusing layer or excitation sources or varying the drive currents can be performed with the TiO_2 materials. With the findings in this paper, the scattering property of TiO_2 is an effective means of regulating the light performance of white LED. Thus, future works with suggested topics can contribute to extending the application of scattering materials, especially TiO_2, in various improved-quality LED devices.

4. CONCLUSION

This study has demonstrated the integration of graphene quantum dots (GDs) with TiO_2 scattering particles to craft a diffusing film, facilitating the transformation of emitted light from a blue LED chip. By manipulating the dosage of TiO_2, light scattering phenomenon was systematically controlled, thereby exerting a significant influence on the light quality of the WLEDs. Notably, higher amounts of TiO_2 lead to more robust scattering, particularly in longer wavelengths. This is evidenced by the emergence of pronounced emission peaks around 460 nm, 545 nm, and 594 nm, accompanied by two weaker peaks within the green and red regions at approximately 525 nm and 600 nm. Consequently, the TiO_2@GD diffusing layer demonstrates its capability of improving color uniformity and achieving warm white light for the LED model. Optimal color uniformity achieved with the TiO_2@GD composite is revealed with a TiO_2 content of around 15 wt%. However, escalating TiO_2 concentration decreases both emission peak intensity and color rendering performance.

The findings of this investigation can be used as a basis to understand the scattering impacts of TiO_2 content in TiO_2@GD diffusing layer, enriching the references of using TiO_2@GD nanocomposites in solid-sate lighting. Besides, further studies can focus on addressing the disadvantages of the TiO_2@GD in this research by optimizing the diffusing layer structure or morphologies to improve the luminosity and color rendering efficiency while keeping the stable CCT distribution uniformity. On the other hand, our study was performed with simple conventional WLED package, utilizing single phosphor YAG:Ce^{3+}. Since the utilization of multi-phosphor package in WLED fabrication has been popular, investigating the influences of the proposed TiO_2@GD layer incorporating with multiple phosphor materials can be a good topic to work on.

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