High Stability Carbon Dots Phosphor and Ultra-High Color Rendering Index White Light-Emitting Diodes

Yuxian Yan, Luqiao Yin, Huazhang Guo, Liang Wang, and Jianhua Zhang

***Abstract*—A new kind of carbon dots based phosphor is developed for light emitting diodes (LED) by adsorbing carbon dots to starch particles. The bonding mechanism is based on hydrogen bonding of surface functional groups of carbon dots and starch particles. Aggregation-induced quenching is avoided and the thermal stability of the carbon dots phosphor is enhanced. The carbon dots phosphor is used as color convert material for white LED devices. The white LED device fabricated has an ultra-high color rendering index of 93.9, almost pure white CIE coordinate at (0.332, 0.336) and moderate CCT at 5498 K. The photoluminescence intensity (PL) under constant temperature and alternative condition is both tested to study the thermal stability of the phosphor. The PL intensity shows no obvious change after 3 days of aging; only 4% of PL loss after 10 cycles of 25 °C /85 °C alternation. Based on its good PL properties and thermal stability, this carbon dots phosphor has great potential for white LED lighting.**

***Index Terms*—Carbon dots, thermal stability, white light-emitting diodes, color rendering index.**

I. Introduction

CARBON dots (CD) is a new kind of zero-dimension material, which characterized by its easy and variety synthesis method, low toxicity, wide application, and good biocompatibility [1]. The application of CD is widely studied, such as chemical catalysis [2], biomedical science [3], optoelectronics, energy storage [4], sensors (temperature [5], pH [6], fingerprint recognition [7]) and luminescent material. Recent researches of carbon dots are mainly focused on bio usage and sensor application, study about luminescent usage like photoluminescence properties, color convert materials are less concerned because of CD's low photoluminescence quantum yield (PLQY), compared to Cd-based, InP and perovskite quantum dots (QDs). However, these three kinds of QDs all characterized by their narrow full width at half maxi-ma (FWHM), thus they are ideal materials for flat panel display but not suitable for solid state lighting (SSL), in which field color rendering index (CRI), Commission Internationale de I'Eclairage (CIE) and correlated color temperature (CCT) is important [8]. Different from these QDs, CD have a wide FWHM [9]–[11] and can fill a wider range of spectrum area by a single kind of CD.

In order to apply CD to white light emitting diodes (WLEDs), it is better to synthesis CD powder or disperse CD solutions into solid state matrix, which is named as CD phosphor. The synthesized CD phosphor were further dispersed into different resins (mainly epoxy resin) or glues (silicone gel, PDMS) and applied onto LED chips. He and co-workers [12] synthesized a series of zinc borate/carbon dots composites (ZBH/yNCDs), the ZBH emits blue light and the composite emits yellow light, by using this single kind of composite phosphor, CCT controllable white LED is fabricated. Han's team [13] synthesis a white CD powder and dissolved into DMF (*N,N*-Dimethylformamide) and further dispersed into PVB (polyvinyl butyral), the WLEDs they fabricated have a CIE coordinate of (0.32, 0.31). Zhai *et al.* [14] synthesized green and red CD, they adsorb green CD to starch and red CD to PVP (polyvinyl pyrrolidone), these phosphors were dispersed into epoxy-silicon resin and applied on a blue LED chip, the WLEDs they made have a high CRI of 92, CIE coordinate of (0.33,0.33), and CCT of 5610. Wang *et al.* [15] use three color CD and PVP made CD phosphor and made R/G/B three kinds of PMMA (polymethyl methacrylate) films, the WLED they get has a really high CRI of 97, however the CIE coordinate was (0.3924,0.3912), which is too high to defined as "white". Sun and co-workers [16] com-bine blue CD phosphor and green/red polymer dot (PD) phosphor, the color parameters of the UV excited WLED are tunnelable: CCT from 2805 to 7786, CIE coordinate from (0.29,0.33) to (0.41,0.34) and CRI from 85 to 96. Zheng's team [17] wrapped CD with SiO_2, and dispersed the full color CD phosphor (from blue to red, 8 kinds of color) into epoxy resin, the WLEDs they fabricated have a CIE coordinate of (0.33,0.33), CRI at 80.4, another WLED devices have a CIE co-ordinate at (0.34,0.36) and ultra-high CRI of 97.4. Hu *et al.* [18] synthesized a hybrid material of polysiloxane embedded with Si-doped carbon dots, WLEDs fabricated with this hybrid phosphor have a CIE coordinate of (0.33,0.34), CRI of 91 and CCT at 6500 K. These articles show different dispersion method and WLED devices, however, the factors are not satisfied: not pure white CIE, too high CCK or too many kinds of CDs used in the device. Besides, little attention is focused on the thermal stability of the CD.

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In this work, a new kind of carbon dot based phosphor is synthesized by adsorbing yellow-green CD (yGCD) to starch particles, this starch carbon dot phosphor (s-yGCD) is further dispersed into epoxy resin and applied on LED devices. The white LEDs we fabricated has a high CRI of 93.9, pure white CIE coordinate at (0.33, 0.33) and moderate CCT at 5498 K, the thermal stability of the s-yGCD is also studied, it shows outstanding stability under 85 °C and cycles of 25 °C/85 °C.

II. Materials and Methods

A. Materials

2-amino-1-naphthol, ethylenediamine, absolute ethanol was purchased from Sinopharm Chemical Reagent Co., Ltd and used without further purification; starch was purchased from supermarket. 460 nm blue LED chips, reflector, epoxy resin, silicone gel (DOWSIL OE-6550) is purchased from the market for LED packaging.

B. Synthesis and Purification of yGCD

0.025 g of 2-amino-1-naphthol is dissolved into 10 ml of ethanol, then add 0.2 ml of ethylenediamine and disperse by ultrasonication; after 10 minutes of ultrasonic treatment, put the mixture into a 25 ml polytetrafluoroethylene reactor immediately and keep at 180 °C for 12 hours. After naturally cooling down to room temperature, the resulting solution was purified by dialysis for several days. Last, the yGCD powder could be obtained for further characterization after drying at 80 °C and the yGCD was dispersed into ethanol again for application of LEDs. The concentration of the yGCD solution is about 10 mg/ml.

C. Preparation of Starch-yGCD Phosphor

Weigh starch and yGCD with different mass ratio (20:1, 50:1, 100:1, 250:1, 500:1, 1000:1), disperse starch in the deionized water and keep stirring, then add yGCD solution into the starch water (yGCD are preserved in ethanol at a concentration of 10 mg/ml), after 24 hours of vivid stir, wash the mixture by centrifugation and ultrasonic cleaning for several times until the waste is clear, then dry the mixture by vacuum freeze-drying, at last, grind the powder through a 300-mesh sieve.

D. Fabrication of LEDs With s-yGCD Phosphor

The s-yGCD phosphor is dispersed at a mass ratio of 1:1 in the epoxy resin, in order to get evenly dispersed mixture, the same weight of ethanol is added and the mixture is kept stirring for at last 20 minutes; after that, the mixture is filled to the reflector in suitable amount, after vacuum defoaming for 5 mins at room temperature and curing for 30 mins in an 80 °C oven, the samples were placed at room temperature for 24 hours to fully cured. For white LED devices, silicon-coated CdZnSeS/ZnS phosphor [19] is mixed at a mass ratio of 1:2 in the silicone gel; after the s-yGCD is applied and cured in the oven, the CdZnSeS/ZnS mixture is applied on the device and curing for 30 mins in an 80 °C oven; finally, the device is placed at room temperature for 24 hours for fully cured.

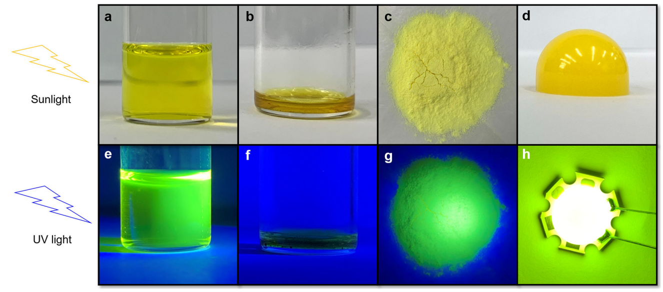

Fig. 1. (a) yGCD ethanol solution, (b) yGCD ethanol solution after vacuum, (c) s-yGCD phosphor, (d) s-yGCD phosphor/epoxy resin bulk under sunlight and under UV light (e), (f), (g), (h).

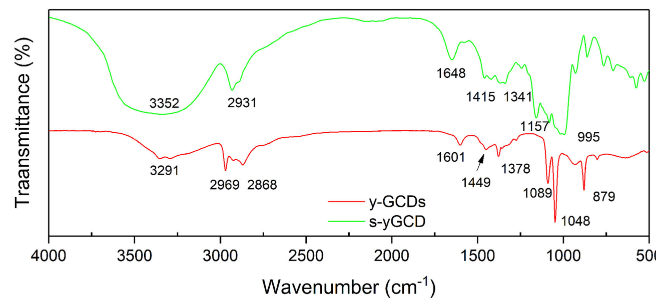

Fig. 2. FTIR spectrum of yGCD and s-yGCD.

E. Characterization

The Scanning electron microscope (SEM) picture is obtained by Zeiss Sigma 500. The Fourier transform infrared (FTIR) spectrum is obtained by a Fourier transform infrared spectroscopy (Thermo Fisher Nicolet iS5). The absorption (Abs) characteristics and photoluminescence (PL) characteristics of the s-yGCD phosphor were measured by a fluorescence spectrophotometer (HTACHI 4500) and a fluorescence spectrometer (U-3900H) respectively. The photoluminescence properties (PL) of the s-yGCD phosphor LEDs were measured by an integrating sphere test system (HASS-2000). Thermogravimetric Analysis (TGA) line and Differential scanning calorimetry (DSC) line are obtained by TA Q200. Other fluorescence images and photos were taken by a Nikon digital camera.

III. Results and Discussion

A. Characterization of s-yGCD Phosphor

The adsorption mechanism of yGCD is simple: the yGCD contains a lot of hydroxyls on the surface, thus can be easily adsorbed on the starch particle through hydrogen bonding. In Fig. 1, different forms of yGCD under sunlight and UV light are shown. Fig. 1(a) and (e) is yGCD solution, after remove 80% of ethanol under vacuum [Fig. 1(b) and (f)] an obvious quench is observed, which is caused by aggregation-induced quenching (AIQ) [20], [21]. Fig. 1(c) and (g) shows the s-yGCD phosphor under sunlight and UV light, the phosphor glows green light, representing that quenching is avoided. Fig. S1 shows the SEM picture of s-yGCD phosphor, the particle size of the phosphor is about 8 μm. Fig. 1(d) and (h) shows the picture of a fluorescent bulk made with our s-yGCD phosphor and epoxy resin.

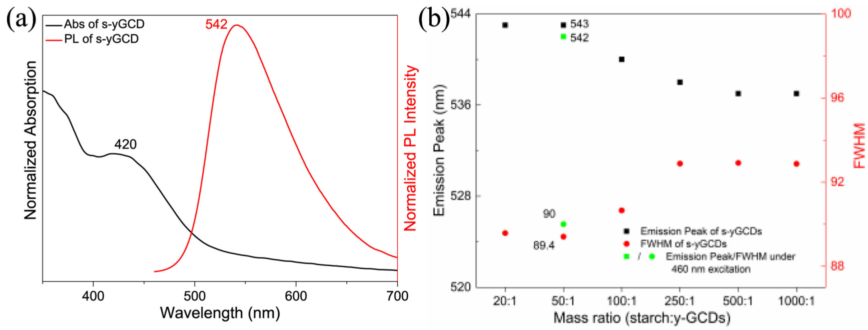

Fig. 3. (a) UV-visible absorption and photoluminescence spectrum of s-yGCD phosphor, (b) emission peak and FWHM of different mass ratio of s-yGCD film under 365 nm excitation, the green dot/square is taken under 460 nm excitation.

Fig. 2 shows the FTIR spectrum of yGCD and s-yGCD. From the red line the characteristic peaks of yGCD can be seen: the weak absorption peak at 3291 cm^{-1} is attributed to stretching vibration of (-OH) and N-H; the bands at 2969 and 2868 cm^{-1} represent the stretching of C-H [22]; the band at 1601 cm^{-1} belongs to stretching vibration of C = C; the bands at 1449 and 1378 cm^{-1} is attributed to stretching vibration of C-N and C-O-C [23]; the two peaks at 1089 and 1048 cm^{-1} be-longs to the stretching of C-O; the peak at 879 cm^{-1} belongs to the out-of-plane deformation vibration of C-H [24]. The green line shows the FTIR spectrum of s-yGCD, the wide and strong peak at 3352 and 2931 cm^{-1} is attributed to (-OH) and C-H stretching; the series of peaks between 1415 and 1341 cm^{-1} are attributed to C-OH/CH2 bending and C = O stretching vibration; the peaks at 1157 cm^{-1} is attributed to C-O/C-C stretching vibration. The adsorption of yGCD is mainly based on hydrogen bonding of carboxyl/amino groups(yGCD) and hydroxyl groups(starch), thus the intensity of -OH (3352 cm^{-1}) and C-C(1157 cm^{-1}) has apparently increased, this is probably the reason why our s-yGCD phosphor shows good thermal reliability in the following researches.

B. Photoluminescence Properties of s-yGCD Phosphor

In order to test the photoluminescence (PL) properties of s-yGCD phosphor, different mass ratio of s-yGCD phosphor were dispersed in the epoxy resin and cure in the mold, the samples for PL property tests were 1mm thick film.

In Fig. 3(a), the PL and Abs spectrum belongs to 50:1 s-yGCD phosphor, it can be seen that the s-yGCD phosphor has a good absorption for wavelength under 480 nm. The PL spectrum was taken under 460 nm excitation, which is the emitting wavelength of our blue LED chips used in WLEDs fabricating. The emission peak and FWHM of 50:1 s-yGCD phosphor is 542 nm and 90 nm respectively, showing in green in Fig. 3(b). For Fig. 3(b), all the emission peak and FWHM were taken under 350 nm excitation, which is the most suitable excite wavelength for the phosphor. From Fig. 3(b), it can be noticed that 20:1 and 50:1 s-yGCD phosphor has the same emission peak and FWHM. The peak emission of s-yGCD phosphor has a slight red shift from 537 nm to 543 nm as the mass ratio decreases, this is caused by the in-creased reabsorption of s-yGCD phosphor [25], [26]. The FWHM decreases as the mass ratio decreases, this is due to the

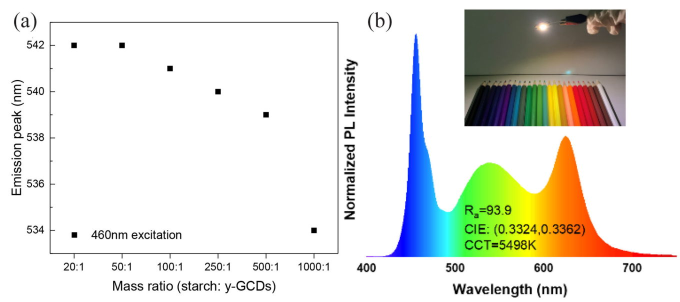

Fig. 4. (a) PL spectrum of s-yGCD phosphor under 460 nm excitation, (b) PL spectrum and color paraments of WLED, inset is the picture of working state.

property of starch itself: starch is easy to absorb moisture in the air and aggregate, as the mass ratio decreases, the more surface area of starch particles is covered by the yGCD, thus the chance of aggregation also decreased.

The on-chip performance of the s-yGCD phosphor is also tested. The PL spectrum of different mass ratio s-yGCD phosphor is tested first: the phosphor is dispersed at a mass ratio of 1:1 in the epoxy resin, the mixture is filled to be flush with the reflector and cured. The emission peak of the blue LED chips we use is 460 nm, and the results are shown in Fig. 4(a). The trend of emission peak under 460 nm excitation is similar to 365 nm excitation, the difference between 460 nm excitation and 365 nm excitation is minor, s-yGCD phosphor shows good potential for both UVLED based and blue LED based white LED devices. Fig. 4(b) shows the spectrum of the WLED device we fabricated, inset is the working state under a driving current of 30 mA. In order to get a high CRI, we use silicon-coated CdZnSeS/ZnS phosphor as the red source, the synthesis and characterization of this material is described as previously shown [19]. The spectrum of color sources is shown in Fig. S2-S4. The WLED we fabricated has a perfect PL performance: almost pure white CIE co-ordinate of (0.332, 0.336), ultra-high CRI of 93.9 moderate CCT at 5498 K, and luminous efficacy of 49.9 lm/W.

C. Thermal Stability of s-yGCD Phosphor

The thermal stability of s-yGCD phosphor is studied and some interesting phenomena is noticed. The stability under constant temperature is tested first, the s-yGCD phosphor devices is aged under 40 °C and 85 °C for 6 hours, the results are shown in

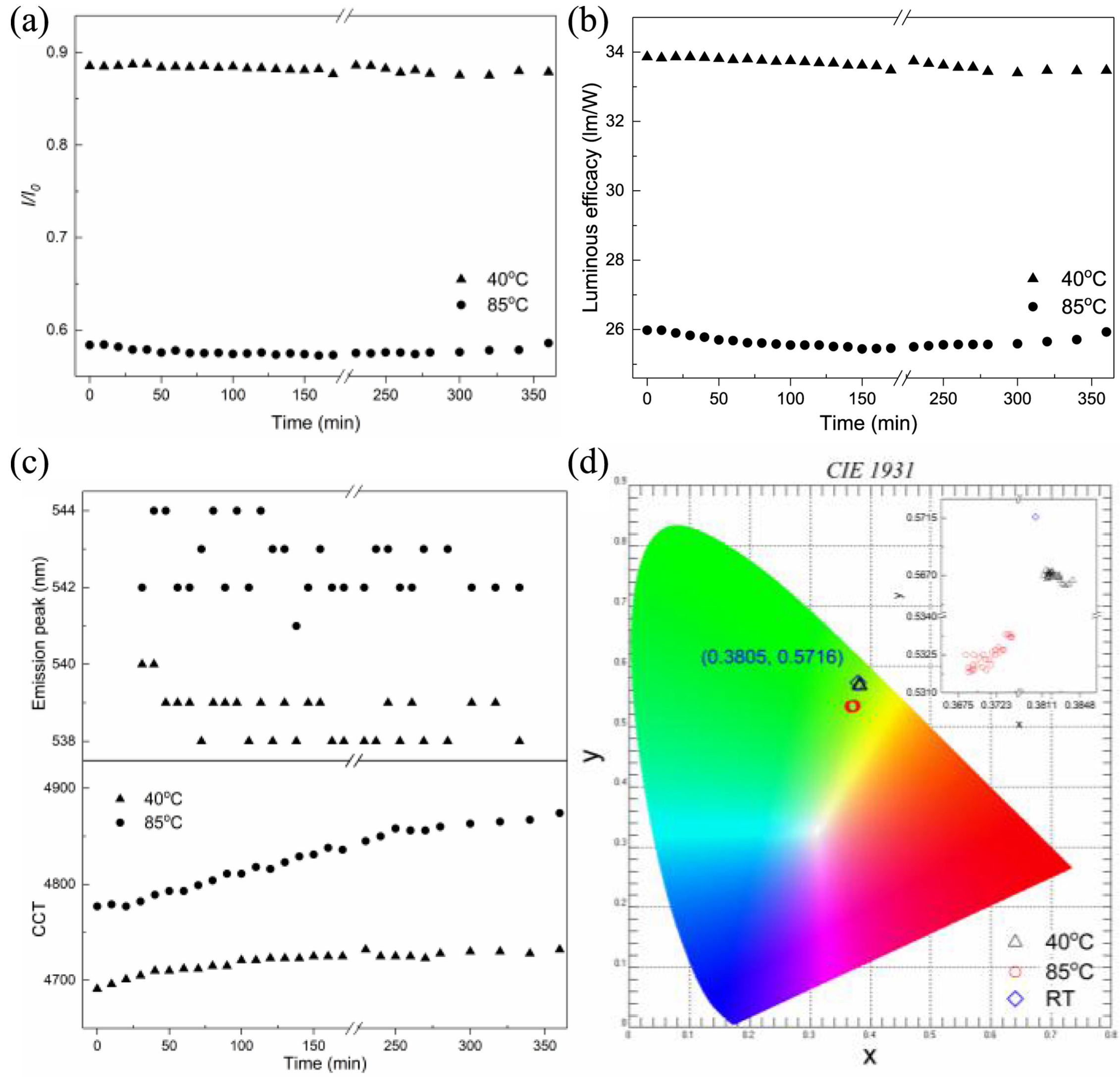

Fig. 5. (a) I/I_0, (b) Luminous efficacy, (c) Emission peak and CCT as a function of time, and (d) CIE coordinate change during the ageing, inset is the partial zoom.

Fig. 5. Fig. 5(a), (b) and (c) shows the changes of relative PL intensity (I/I_0), luminous efficacy, wavelength, and CCT during the ageing (I represent for PL intensity at different time under 40 °C and 85 °C, I_0 represents for PL intensity under room temperature (RT)). The change of PL intensity is hard to tell, it stabilized at 88% and 58% at 40 °C and 85 °C, compared to RT; the change of CCT reflect the change of PL intensity: the increase of CCT is caused by the decrease of PL intensity, the tested device is excited by 460 nm blue LED chips, as the PL intensity decreases, the intensity of blue light increases, thus the CCT increased. The emission peak and luminous efficacy also appeal good stability under both 40 °C and 85 °C: the emission peak vibrate between 538 nm and 540 nm under 40 °C, between 540 nm and 544 nm under 85 °C, the emission peak under room temperature is 537 nm. Fig. S5(a) and (b) show the change of I/I_0 and luminous efficacy during 3 days, no obvious changes were observed after the several hours. Fig. 5(d) shows the changes of CIE coordinate, the CIE coordinate under RT is (0.3805,0.5716) and the stability under 40 °C and 85 °C is also perfect, the change

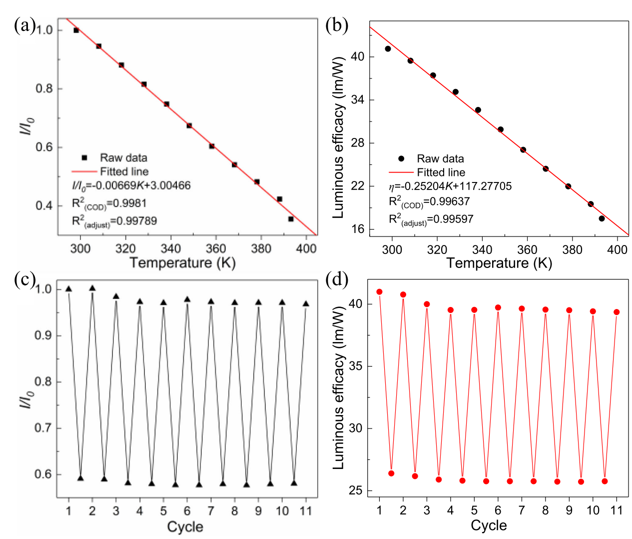

Fig. 6. (a) I/I_0 and (b) luminous efficacy as a function of temperature, (c) I/I_0 and (d) luminous efficacy under 25 °C/85 °C cycles.

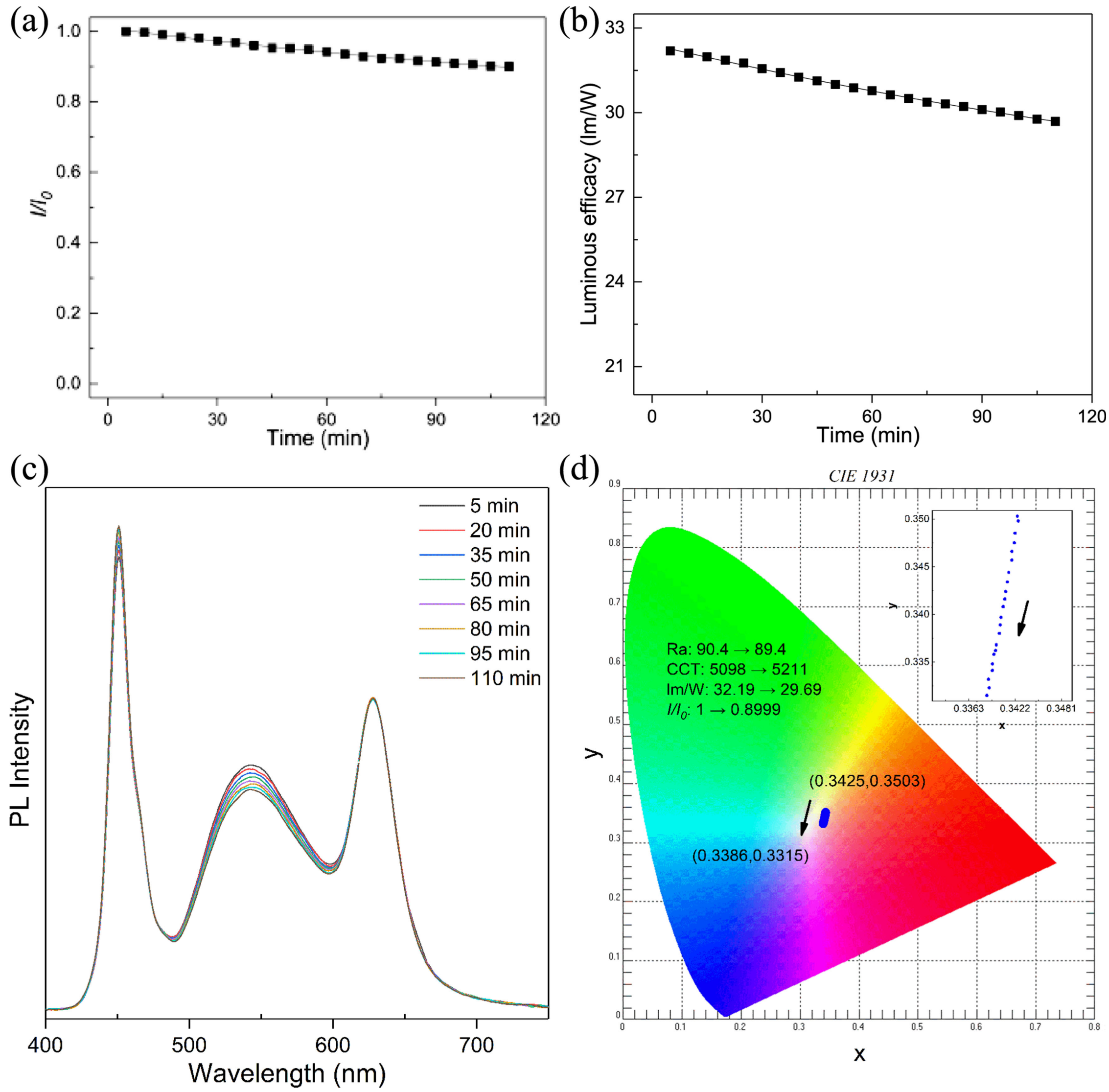

Fig. 7. (a) I/I_0, (b) luminous efficacy as a function of time, (c) PL spectrums of WLED device during ageing, and (d) CIE co-ordinate change during the ageing, inset is a partial zoom.

can be told from the inset, it is obvious that the distribution is more scattered under 85 °C.

Since s-yGCD phosphor shows good stability under constant temperature, the stability under a changing temperature is studied: the device is fixed on a heating plate to control the working temperature. The change of relative PL intensity and luminous efficacy between 25 °C and 120 °C is shown in Fig. 6(a) and (b): the red line in the figures is fitted line, shows that the relative PL intensity (I/I_0) and luminous efficacy is linearly related to temperature (K); here, I represents for PL intensity under different temperatures, I_0 represents for PL intensity under RT and η for luminous efficacy. The $R_{2(\mathrm{adjust})}$ for the fitted line is 0.99789 and 0.99597 respectively, meaning that our fitting is reasonable. This shows that s-yGCD phosphor is a potential material for temperature sensors, especially range from 25 °C to 85 °C. The stability under alternation temperature between 25 °C and 85 °C is also studied and the results are shown in Fig. 6(c) and (d). The data is collected under the following conditions: the heating rate is 20 °C/min, 10 minutes of stable time before test, 10 cycles are carried out in total. It can be seen that the I/I_0 and luminous efficacy has an apparent drop after the first cycle and kept stable during the remaining cycles, showing that the s-yGCD phosphor has a good stability under an alternating condition. Fig. S6 shows the I/I_0 change during a cycle of 25 °C/120 °C. The obvious PL loss is owning to the high temperature of 120 °C, which is higher than the Glass transition temperature (T_g) of the yGCD (Fig. S7 and S8). In order to confirm the lineal relationship of is attributed to s-yGCD phosphor, the thermal stability of pure blue LED chip under alternative temperature is tested and the results are shown in Fig. S9. Fig. S9(a) shows that the relationship of I/I_0 and temperature is lineal. However, Fig. S9(b) shows no lineal relationship between luminous efficacy and temperature.

The stability under constant excitation is studied at last, a white LED device fabricated as mentioned before is tested for 110 minutes under a driving current of 50 mA, the temperature is controlled at 25 °C, results are shown in Fig. 7. Fig. 7(a) and (b) shows the changes of PL paraments, the PL intensity of s-yGCD phosphor (peak at 542 nm) drops for 10% after

110 minutes of ageing and the luminous efficacy of the WLED device drops from 32.19 lm/W to 29.69 lm/W. Fig. 7(c) shows the PL spectrums of the device during working state, the three peaks are attributed by blue LED chip, s-yGCD phosphor and CdZnSeS/ZnS phosphor respectively, the peak intensity of s-yGCD phosphor has an apparent drop during the ageing while the other two only changed a little, this implies that the stability of s-yGCD phosphor under excitation state needs improvement. The CIE coordinate in Fig. 7(d) also shows a blue shift due to the attenuation of s-yGCD's PL intensity, while the CRI only changes a little from 90.4 to 89.4.

IV. Conclusion

In summary, s-yGCD phosphor is prepared by adsorbing yGCD to starch through hydrogen bonding, this dispersion method perfectly deals with the aggregation-induced quenching. The synthesized phosphor has good photoluminescence performance and thermal stability: the WLED device fabricated by our s-yGCD phosphor has an ultra-high CRI of 93.9, pure white CIE coordinate of (0.332,0.336), and moderate CCT at 5498 K; the s-yGCD phosphor shows perfect stability under both 25 °C and 85 °C, the PL loss is less than 5% after 3 days of aging; the PL loss under alternative thermal condition is 4% and shows good lineal relationship to temperature. We propose that the s-yGCD phosphor shows potential for temperature sensors and solid-state luminescence relate fields.

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