Precipitating tunable-emission CsPb(Cl/Br)\(_3\) QDs in boro-germanate glass for wide-color-gamut liquid crystal displays

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ABSTRACT

Embedding CsPbX\(_3\) (X = Cl, Br, I) perovskites QDs in inorganic glass matrix via in-situ crystallization has been regarded as a feasible strategy to improve their stability against water, heat, and light irradiation. However, the tunable-emission range of these CsPbX\(_3\)-nanocrystals-embedded glasses is still narrow, insufficient to cover a wide color gamut. Herein, a simple synthetic method was developed in this study to obtain the tunable-emission CsPb(Cl/Br)\(_3\) quantum dots (QDs) in boro-germanate glass (CsPb(Cl/Br)\(_3\) QDs@glass) by controlling the molar ratio of PbBr\(_2\)/PbCl\(_2\). With the increase of the PbBr\(_2\) concentration from 1.6 to 3.2 mol%, the emission wavelength of CsPb(Cl/Br)\(_3\) QDs@glasses can be shifted from 448 to 485 nm, and the corresponding photoluminescence quantum yield (PLQY) increases from 0.61 to 24.43%. In addition, the CsPb(Cl/Br)\(_3\) QDs@glass exhibits impressive promotion of thermal stability, photostability, and water resistance due to the excellent protective function of the glass matrix. As a proof-of-concept experiment, a LED device was fabricated by employing the optimal CsPb(Cl/Br)\(_3\) QDs@glass with a commercial 375 nm UV chip, yielding an intense cyan light. All the results indicate that the investigated CsPb(Cl/Br)\(_3\) QDs@glasses may find promising applications as color converters in wide-color-gamut liquid crystal displays (LCDs).

1. Introduction

Inorganic cesium lead halide (CsPbX\(_3\), X = Cl, Br, I) perovskite quantum dots (QDs) have attracted tremendous attention of late in view of their prominent properties, including their high photoluminescence quantum yield (PLQY), narrow full width at half maximum (FWHM), bright and tunable emissions covering the entire visible spectral region, and high defect tolerance [1–4]. With these outstanding features, CsPbX\(_3\) perovskite nanocrystals have been recognized as promising materials for application in many optoelectronic fields, such as photovoltaics, low-threshold lasers [5–8], photodetectors [9–11], and displays [12–14]. Especially, the continuously and tunable spectra region from blue to green to red (covering the whole visible spectrum) of CsPbX\(_3\) QDs has greatly met the demands for the maximum accessible color gamut of the next-generation liquid crystal displays (LCDs), encompassing 140% color space of NTSC [15]. Thus, all inorganic CsPbX\(_3\) QDs emitters have great applied value in the next-generation ultraviolet-light-emitting-diode-(UV LED)-pumped red-green-blue (RGB) backlight displays. The ionic structure and large surface energy of perovskite QDs, however, result in their instability when exposed to water, air, heat, and light irradiation. Thus, these problems have limited their practical application.

In the past few years, many coating methods were reported to enhance the stability of CsPbX\(_3\) nanocrystals, such as surface modification using thick poly(maleic anhydride-alt-1-octadecene) (PMAO) [16,17], amine [18], and crown ether [19]; incorporation of perovskite quantum dots (QDs) into silica [20,21] or polymethyl methacrylate (PMMA) [14]; the use of isostructured or heterostructured core–shell perovskite nanocrystals [22]; and atomic layer deposition [23]. Zhang et al. reported a facile one-pot approach to synthesizing CsPbBr\(_3\)@SiO\(_2\) core-shell nanoparticles by coating CsPbX\(_3\) nanocrystals with SiO\(_2\) on a single-particle level. Offering the benefit...
of protecting the SiO₂ shell, the product shows much higher long-term stability in humid air compared to the uncoated CsPbBr₃ nanocrystals [21]. This method is quite interesting, however, as the core/shell heterostructures will definitely induce structural defects and limit the optoelectronic performance of perovskite nanocrystals. Yang et al. obtained an ultrastable CsPbBr₃ composite using an effective postsynthetic strategy by coating CsPbBr₃ nanocrystals with a thick (~25 nm) PMAO polymer [17]. The coated CsPbBr₃ composite maintains more than 90% of its initial emission intensity under 144 h continuous ultraviolet illumination. Upon immersion in water for 24 h, the treated sample maintains 60% of its initial PL intensity. The lifetime of this type of organic polymer material, however, is relatively short due to its efflorescence and aging. In summary, the aforementioned methods can improve the material’s moisture stability to some extent, but the material’s long-term thermal stability and photostability still hinder their practical application. In 2016, Liu et al. precipitated the conventional II-VI and IV-VI QDs in glass, and first precipitated the CsPbBr₃ QDs in phosphate glass with preferable stability [24]. After that, many perovskite nanocrystals, such as CsPbI₃, CsPbBr₂, CsPbI₂Br, and CsPb₂Br₃, have also been successfully precipitated in glass through heat treatment [25–29]. Precipitating CsPbBr₃ QDs and CsPb(Br/I)₃ nanocrystals in glass makes them relatively mature by manipulating the appropriate molar ratio of raw materials like Cs₂CO₃, PbBr₂, and NaBr/NaI. A clear and effective method of embedding the CsPb(Cl/Br)₃ QDs in glass, however, has yet to be developed. In addition, it is still hard to determine the exact ratio of Cl/Br for the CsPb(Cl/Br)₃ QDs@glass because of the interfaces in the glass matrix.

In this study, an efficient synthesized method was developed by fixing the NaCl content and controlling the relative concentration of PbBr₂/PbCl₂, and a series of CsPb(Cl/Br)₃ QDs@glass samples were obtained. The microstructure and luminescent properties of CsPb(Cl/Br)₃ QDs@glasses were further investigated in detail. By increasing the PbBr₂ concentration, the emission peak can be shifted from 448 to 485 nm, and the CsPb(Cl/Br)₃ QDs@glass will reach the highest PLQY of 24.43% when the PbBr₂ content is 3.2 mol%. In addition, water resistance, cycle thermal stabilization, and photostability of the prepared samples were also performed. Finally, a proof-of-concept LED device was fabricated by combing the optimal CsPb(Cl/Br)₃ QDs@glass sample and a commercial 375 nm UV chip, yielding an intense cyan light with excellent optoelectronic performance.

2. Experiment section

2.1. Raw materials of the synthesis CsPb(Cl/Br)₃ QDs@glass samples

Cesium carbonate (Cs₂CO₃, 99.9%), lead(II) bromide (PbBr₂, 99.0%), boron oxide (B₂O₃, 98%), zinc oxide (ZnO, 99%), and strontium carbonate (SrCO₃, 99%) were purchased form Aladdin, and germanium dioxide (GeO₂, 99.99%), lead(II) chloride (PbCl₂, 99.0%), and sodium chloride (NaCl, 99%) were purchased from Macklin. All the above reagents were used as received, without further treatment.

2.2. Method and characterization

The CsPb(Cl/Br)₃ QDs@glass samples were synthesized through a melting–quenching method and subsequent crystallization (heat treatment). The precursor glasses were designed with 40.4GeO₂–36.2B₂O₃–3.2ZnO–5.3SrO–8.5Cs₂O–xPbBr₂–3.2NaCl (x = 1.6, 2.4, 3.2 in mol%) molar compositions. 20 g of each of these raw materials was weighed precisely, and then the materials were ground thoroughly in a dried agate mortar for mixing. Subsequently, the mixtures were transferred into corundum crucibles and sintered in a muffle furnace at 1000 °C for 30 min under the atmospheric environment. Then the melts were poured into pre-heated graphite molds and held for 4 h to release the inner stress. As the melts cooled down to room temperature, the precursor glasses (PGs) were obtained. Finally, all the PGs were heated at 460 °C for 10 h in a muffle furnace for in-situ crystallization. Thus, a series of tunable-emission CsPb(Cl/Br)₃ QDs@glass specimens were obtained.

For X-ray diﬀraction (XRD) analysis, a Rigaku Ultima-IV powder X-ray diﬀractometer was used with Cu Kα radiation (λ = 1.5405 Å) at 40 kV and 40 mA. A high-resolution transmittance electron microscope (HR-TEM) images of CsPb(Cl/Br)₃ QDs@glass with a 2.4 mol% PbBr₂ content. The absorption spectra were recorded on a Cary 5000 UV-vis-NIR spectrophotometer, and a Hitachi FL7000 fluorescence spectrophotometer was used for recording the room-temperature photoluminescence excitation (PLE) and emission (PL) spectra. The temperature-dependent PL spectra within 25–200 °C were recorded with a Hitachi FL7000 fluorescence spectrophotometer equipped with an Oxford OptiataDNI2 nitrogen cryostat. Moreover, the decay curves of the specimens were recorded using an FSL1000 (Edinburgh,
Britain). The room-temperature PLQY values of the samples were measured using a barium-sulfate-coated integrating sphere attached to the FSL1000. The electroluminescence spectra of the LED device were recorded using the FL7000 fluorescence spectrophotometer at a forward current.

3. Results and discussion

The XRD patterns of the obtained CsPb(Cl/Br)₃ QDs@glasses are shown in Figure 1. Several weak diffraction peaks could be carefully observed in the broad hump of the glass matrix, indicating the formation of crystalline phases in the glasses. When the PbBr₂ concentration is increased from 1.6 to 3.2 mol%, the corresponding diffraction peak gradually shifts towards the low-angle side because of the lattice expansion of the QDs induced by the partial substitution of the smaller Cl⁻ ions by larger Br⁻ ions. The average size of the CsPb(Cl/Br)₃ QDs is difficult to estimate from the width of the diffraction peak according to the Scherrer equation due to the weak diffraction intensity. These CsPb(Cl/Br)₃ QDs@glasses are still further studied via TEM.

In the TEM image (Figure 2a), distinct boundaries between the crystalline phase and the glass matrix can be clearly observed, showing that the CsPb(Cl/Br)₃ QDs were well precipitated in the glass matrix. It is worth noting that the QDs inside the glass exhibited a near-spherical shape morphology with an 8–15 nm size range, different from the cubic shape of colloidal QDs previously prepared through the wet-chemical method. This phenomenon was also reported in other literatures [30]. To further verify the nanoparticles, the interplanar spacing of an individual CsPb(Cl/Br)₃ particle with well-resolved lattice fringes was calculated to be 2.82 Å (Figure 2b), locating the 2.9150–2.7940 Å range (representing the (200) standard planes of CsPbBr₃ and CsPbCl₃, respectively).

A series of highly transparent CsPb(Cl/Br)₃ QDs@glass specimens were synthesized by controlling the relative PbBr₂/PbCl₂ content. To further investigate the optical properties of the above specimens, the absorption spectra of such specimens were recorded with the precursor glasses and the heat-treated specimens (solid lines) (Figure 3). Before being heat-treated, all the glass samples showed almost the same absorption spectra, as shown by the dash lines in Figure 3. Thus, it was concluded that the absorption edge located at ~ 323 nm could be attributed to the precursor glass because the absorption spectra did not change with the addition of PbBr₂. After heat treatment, the absorption edge shifted from ~ 442 to ~ 477 nm with the increase in PbBr₂ concentration from 1.6 to 3.2 mol%. Such an obvious red shift indicates that the CsPb(Cl/Br)₃ nanocrystals are effectively precipitated in precursor glass. The red shift of the absorption edges also indicates that the ratio of Cl/Br in CsPb(Cl/Br)₃ nanocrystals is effectively regulated. This can also be reflected by the change in the samples’ body color. Under 365 nm excitation, the CsPb(Cl/Br)₃ QDs@glass with 1.6 mol% PbBr₂ showed a broad photoluminescence band centered at 448 nm, indicating that there is no phase separation during the heat treatment, except for precipitating CsPb(Cl/Br)₃ QDs. When the PbBr₂ content increased from 1.6 to 3.2 mol%, the peak wavelength shifted from 448 to 485 nm. Tunable emission from blue to cyan is obtained (Figure 4a). Meanwhile, all the samples showed a small FWHM in the 17–23 nm range. Under 365 nm excitation, the PLQY increased from 0.61 to 24.43% with the increase in PbBr₂ content from 1.6 to 3.2 mol% (Figure 4c). Under 365 nm excitation, the bi-exponential fitting curves can favorably describe the characterization of the specimens [31].

\[ I(\tau) = A_1 e^{-\tau/\tau_1} + A_2 e^{-\tau/\tau_2} \] (1)

Then the average lifetime of each specimen can be calculated using the following equation:

\[ \tau_{ave} = \frac{\int I(\tau) dt}{I_0} \] (2)

where \( I_0 \) is the peak intensity (normalized as 1) and \( I(\tau) \) is the relative intensity. Based on the above two equations, the average lifetime of each specimen was calculated to be 12.1 ns for \( x = 1.6 \text{ mol\%} \), 16.4 ns for \( x = 2.4 \text{ mol\%} \),

![Figure 1. XRD patterns of the CsPb(Cl/Br)₃ QDs@glass samples with different PbBr₂ contents (from the bottom to the top: \( x = 1.6, 2.4, \) and 3.2 mol%). The pattern at the bottom represents the standard diffraction peaks of cubic-structured CsPbCl₃ (JCPDS NO. 18-0366), and the pattern at the top represents the standard diffraction peaks of the cubic-structured CsPbBr₃ (JCPDS NO. 54-0752).](image-url)
and 18.4 ns for \( x = 3.2 \text{ mol\%} \), respectively. The evaluated lifetime value was in the nanosecond scale as it was close to those in the previous literatures [23,25]. The lifetimes exhibited the same trend with the increasing relative concentration of PbBr\(_2\) as that of the PLQY, which was due to the decrease in the trap density of the CsPb(Cl/Br)\(_3\) QD structure. This phenomenon has also been previously observed for the colloidal CsPbX\(_3\) QDs [15,32,33].

Thermal stability, photostability, and water resistance are three key factors for evaluating the potential application of the prepared CsPb(Cl/Br)\(_3\) QDs@glass. A thermal-cycle experiment was carried out to assess the optical performance of the sample at a high temperature. Figure 5a exhibits a relative symmetrical change in the heating/cooling cycle process. For further quantification, at 100°C, the integrated intensity was about 20% of that at room temperature. After further increasing the temperature up to 200°C, the enhanced non-radiative transition of excitons led to almost complete fluorescence quenching. Meanwhile, the peak wavelength showed a blue shift from 484 to 465 nm, and the FWHM broadened from 23 to 40 nm when the temperature increased from 25 to 200°C (Figure 5c), which was caused by the thermal expansion and electron-phonon interactions. Fortunately, the integrated PL intensity of CsPb(Cl/Br)\(_3\) QDs@glass can be retained at ~90% of the original one after it undergoes a heating/cooling cycle process at 200°C. After undergoing one more heating/cooling procedure, the luminescent intensity of the sample was still ~88% of the initial value. Compared with the case of colloidal QDs, the thermal stability of CsPb(Cl/Br)\(_3\) QDs@glass obviously improved because the dense amorphous structure of glass can effectively prevent the interaction of CsPb(Cl/Br)\(_3\) QDs with water and oxygen at a high temperature. After exposure to UV light for 12 h, very little PL degradation (less than 3%) appeared for the CsPb(Cl/Br)\(_3\) QDs@glass \((x = 3.2 \text{ mol\%})\) (Figure 6). Upon immersion in water for 48 h, the PL emission intensity for the CsPb(Cl/Br)\(_3\) QDs@glass maintained ~100% of its initial PL intensity (Figure 7). All the results indicate that the decomposition of CsPb(Cl/Br)\(_3\) QDs exposed to water and UV light could be prevented by precipitating them in a glass matrix.

To further investigate the potential application of the CsPb(Cl/Br)\(_3\) QDs@glass, a LED device was fabricated with CsPb(Cl/Br)\(_3\) QDs@glass \((x = 3.2 \text{ mol\%})\) and a commercial 375 nm LED chip. The CsPb(Cl/Br)\(_3\)
Figure 4. (a) PL spectra of the CsPb(Cl/Br)3 QDs@glass samples with different PbBr2 contents (x = 1.6, 2.4, 3.2 mol%). (b) Peak wavelength and FWHM variations with increasing PbBr2 content. (c) PLQY and (d) decay curves of the CsPb(Cl/Br)3 QDs@glass samples with different PbBr2 contents (x = 1.6, 2.4, 3.2 mol%).

Figure 5. (a) Two-dimensional temperature-dependent PL spectra of CsPb(Cl/Br)3 QD@glass (x = 3.2 mol%) obtained through a heating/cooling cycle process. (b) Temperature-dependent integrated intensity of the PL spectra obtained via a two-time heating/cooling cycle at 200°C. (c) Peak wavelength and FWHM variations with different temperatures in the whole heating/cooling experiment.
QDs@glass has strong absorption in the 323–477 nm range, which well matches the emission of UV or the blue LED chip. Under driving with a 20 mA forward current, the LED device gives intense cyan light (inset of Figure 8) with (0.2491, 0.4196) color coordinates. When the forward current increased from 20 to 140 mA, there was almost no change in the shape of the EL spectra, except for the intensity. All the results indicate that the CsPb(Cl/Br)₃ QDs@glasses have great potential application as light convertors in wide-color-gamut LCDs.

4. Conclusions

In summary, an efficient method involving the modification of the molar ratio of PbBr₂/PbCl₂ was used to synthesize a series of CsPb(Cl/Br)₃ QDs@glasses. With increased PbBr₂ content, the obtained CsPb(Cl/Br)₃ QDs@glasses exhibited tunable optical performance by shifting the peak wavelength from 448 to 485 nm with a narrow band. When the PbBr₂ content was 3.2 mol%, the CsPb(Cl/Br)₃ QDs@glass had 24.43% PLQY and presented good stability due to the protection of the dense structure and the inert environment provided by the glass matrix. As a proof-of-concept experiment, a light-emitting diode (LED) device was fabricated using the optimal CsPb(Cl/Br)₃ QDs@glass and an ultraviolet (UV) LED chip, which gives intense cyan light. All the results indicate that controlling the ratio of PbBr₂/PbCl₂ can help obtain tunable-emission CsPb(Cl/Br)₃ QDs in boro-germanate glass for UV-LED-pumped red-green-blue (RGB) backlight displays.

Disclosure statement

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