

Ultrahigh Photoluminescence and Environmentally Stable K₂CsPbBr₃ Quantum Dots in Situ Crystallized in Zinc Borosilicate Glass

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Abstract

All-inorganic CsPbBr₃ perovskite quantum dots (PQDs) have attracted considerable attention for solid-state lighting, displays and optoelectronic devices on account of their high photoluminescence quantum yield, narrow emission linewidth and tunable bandgap. Nonetheless, the poor stability of CsPbBr₃ PQDs under humid and high-temperature conditions gives rise to pronounced degradation of their optical properties, thereby limiting their long-term applications. In this work, K⁺-doped CsPbBr₃ PQDs embedded in zinc borosilicate (ZBS) glass were fabricated through a two-step high-temperature solid-state route to improve their environmental and thermal stability. K⁺ incorporation markedly elevated the photoluminescence performance of the CsPbBr₃ PQDs. Furthermore, the ZBS glass matrix, owing to its exceptional mechanical robustness, provided effective protection against thermal degradation and moisture attack, thereby substantially heightening the stability of the embedded CsPbBr₃ PQDs. Remarkably, the PQDs retained an average particle size of ~ 2.3 nm even after calcination at 600 °C. In particular, the K_{0.2}Cs_{0.8}PbBr₃ sample exhibited a ~ 17 times reinforcement in PL intensity in contrast to the undoped sample, while maintaining a stable emission peak at 521 nm. Meanwhile, the bandgap increased from 2.34 to 2.36 eV, while the carrier lifetime was prolonged from 17.5 to 17.7 ns. On top of that, the sample retained approximately 98% of its initial PL intensity subsequent to immersion in deionized water for 42 days and exhibited nearly unchanged PL performance in the aftermath of storage in air for 70 days. This work provides a simple and effective strategy for developing high-performance and highly stable perovskite quantum dot-based luminescent glass materials.

1. Introduction

Inorganic CsPbBr₃ perovskite quantum dots (PQDs) are promising for LEDs [1], lasers [2], and photodetectors [3] owing to their high photoluminescence quantum yield (PLQY), narrow emission linewidth, tunable bandgap, and high color purity. [4–6] Nevertheless, for one thing, CsPbBr₃ is highly sensitive to moisture, oxygen and heat, rendering it susceptible to structural degradation and fluorescence quenching during prolonged operation and thereby severely limiting its practical applications.[7–9] For another, the intrinsic defects that readily form in the CsPbBr₃ crystal lattice facilitate nonradiative recombination, thereby lessening the photoluminescence (PL) efficiency.[10, 11] For this reason, it is impressive to develop effective strategies that simultaneously enable defect passivation and reinforced environmental stability to synergistically improve the PL efficiency and long-term stability of CsPbBr₃ PQDs, which has emerged as a pivotal research focus for their practical applications.

To date, phosphate,[12][13] silicate[14, 15] and tellurite glass[16, 17] matrices have been extensively explored for the fabrication of CsPbBr₃ PQDs glasses, ultimately triggering substantially reinforced moisture resistance and thermal stability. For instance, an elevated melt-quenching strategy enabled the direct crystallization of CsPbBr₃ PQDs within a phosphate glass matrix, yielding bright green emission under 350 nm excitation.[18] On top of that, encapsulating CsPbBr₃ PQDs within a stable and dense

TeO₂-based glass matrix enabled the material to retain approximately 90% of its initial PL intensity in the aftermath of immersion in deionized water for 120 h.[19] In comparison with conventional phosphate silicate and tellurite glass systems, zinc borosilicate glass [20–25] offers a more balanced combination of high chemical durability, exceptional hydrolytic resistance, superior mechanical strength, reinforced thermal stability and low cost, while circumventing the high density, unaffordable cost and comparatively constrained chemical durability of TeO₂-based glasses.[26] Simultaneously, the incorporation of ZnO helps regulate the glass network and tailor its crystallization behavior, thereby creating a favorable microenvironment for the uniform precipitation and growth of CsPbBr₃ PQDs.[27–31] Aside from that, previous studies have shown that partially substituting Cs⁺ with K⁺ effectively strengthens the PL performance and stability of CsPbBr₃ PQDs. This reinforcement primarily originates from the ability of K⁺ doping to optimize the crystal lattice and passivate both bulk and surface defects,[32–38] thereby lessening nonradiative recombination centers and elevating the PL efficiency and environmental stability of CsPbBr₃ PQDs.

For this reason, KCsPbBr₃ PQDs were fabricated through in situ crystallization in a zinc borosilicate glass matrix. Attributable to the confinement and protection provided by the glass matrix, the KCsPbBr₃ PQDs maintained an average particle size of ~ 2.3 nm in the aftermath of calcination at 600°C and displayed intense green emission centered at 521 nm. It is particularly noteworthy that subsequent to heat treatment at 600°C, the PL intensity of the K_{0.2}Cs_{0.8}PbBr₃ sample was ~ 17 times higher than that of the undoped CsPbBr₃ sample. Aside from that, the KCsPbBr₃ PQDs glass phosphor retained approximately 98% of its initial PL intensity subsequent to a 42-day immersion in deionized water, while its luminescence performance remained nearly unchanged after 70 days of storage in air, thereby underscoring its exceptional environmental stability. As evidently demonstrated by this work, the synergistic integration of ion doping and glass matrix confinement offers a beneficial strategy for simultaneously strengthening the PL performance and environmental stability of CsPbBr₃ PQDs, paving the way for the development of high-performance PQDs glasses for solid-state lighting and display applications.

2. Experimental Section

2.1 Materials.

All chemicals were of analytical grade and used as received in the absence of further purification. Cesium bromide (CsBr, 99.9%, A.R.), lead bromide (PbBr₂, 99.9%, A.R.), and potassium bromide (KBr, 99.9%, A.R.) were purchased from Aladdin (China) and used as precursors for the synthesis of perovskite quantum dots. Silicon dioxide (SiO₂, A.R.), boric acid (H₃BO₃, G.R.), and zinc oxide (ZnO, A.R.) were also obtained from Aladdin (China) and served as glass-forming reagents.

2.2 Synthesis of Zinc Borosilicate Glass

Zinc borosilicate (ZBS) glass was prepared by adopting a conventional melt-quenching method. SiO_2 (A.R., Aladdin), H_3BO_3 (G.R., Aladdin), and ZnO (A.R., Aladdin) were weighed in accordance with the nominal composition of $37.5\text{B}_2\text{O}_3\text{-}37.5\text{SiO}_2\text{-}25\text{ZnO}$ (mol%). The raw materials were thoroughly mixed by grinding in an agate mortar for 30 min to ensure compositional homogeneity. The resulting mixture was subsequently transferred into an alumina crucible and melted in a muffle furnace at 1500°C for 120 min to obtain a homogeneous glass melt. In the aftermath of melting, the glass melt was rapidly quenched in deionized water to relieve internal stress. Finally, the obtained glass was ground into zinc borosilicate (ZBS) glass powder for the subsequent synthesis of PQD glass.

2.3 Synthesis of KCsPbBr_3 PQDs Glass

As illustrated in Fig. 1., the prepared ZBS glass powder was mixed with the precursors for K^+ -doped CsPbBr_3 PQDs, including cesium bromide (CsBr , 99.9%, A.R., Aladdin, China), lead bromide (PbBr_2 , 99.9%, A.R., Aladdin, China), and potassium bromide (KBr , 99.9%, A.R., Aladdin, China). The precursor mixture and ZBS glass powder were blended at a mass ratio of 1:2 and thoroughly ground in an agate mortar for 30 min to ensure homogeneous dispersion. The resulting mixture was subsequently transferred into an alumina crucible and heat-treated in a muffle furnace at 600°C for 120 min to induce the in situ crystallization of K^+ -doped CsPbBr_3 PQDs within the glass matrix. In the aftermath of heat treatment, the product was reground and sieved through a 300-mesh sieve to obtain powders with a uniform particle size for subsequent structural, optical, and PL characterizations.

2.4 Characterizations

The phase structure of the samples was characterized by X-ray diffraction (XRD, Shimadzu XRD-7000s) by employing Cu K α radiation (40 kV, 20 mA) with a 2θ range of $10\text{--}60^\circ$ and a scan rate of $5^\circ/\text{min}$. The particle size and lattice spacing of the samples were analyzed by transmission electron microscope (TEM) and high-resolution transmission electron microscope (HRTEM). Elemental distribution analysis of K-doped CsPbBr_3 glass powder was performed by utilizing an Oxford X-MaxN 80T IE250 energy-dispersive X-ray spectrometer (EDS) with an accelerating voltage of 200 kV. The photoluminescence (PL) and photoluminescence excitation (PLE) spectra of the samples were measured by employing a Hitachi F-7000 fluorescence spectrophotometer at room temperature under a scanning rate of 240 nm/min , which was equipped with a 200 W Xeon lamp. The UV-visible absorption spectra were measured by utilizing a Shimadzu UV-3600i Plus spectrophotometer. The fluorescence decay curves were obtained on the same FLS 1000 system equipped with a femtosecond pulsed laser (EPL), by adopting 365 nm excitation and monitoring at 520 nm.

3. Results and discussion

3.1 Phase Composition and Crystal Structure

The crystal structure of the KCsPbBr₃ PQDs glass samples heat-treated at 600°C for 2 h was characterized by X-ray diffraction (XRD), and the corresponding patterns are presented in Fig. 2a. As revealed in the XRD patterns, all four samples exhibit a broad amorphous diffraction halo, which is characteristic of the glass matrix, suggesting that the ZBS glass retained its amorphous nature in the aftermath of heat treatment. As revealed in Fig. 2a, the undoped CsPbBr₃ sample exhibits several distinct diffraction peaks at $2\theta = 21.5^\circ, 24.2^\circ, 26.3^\circ, 30.4^\circ$ and 30.6° . These peaks can be well indexed to the standard diffraction pattern of CsPbBr₃ (PDF No. 18–0364), confirming the formation of the CsPbBr₃ crystalline phase. With the augmentation of K⁺ doping concentration, the diffraction peak located at $2\theta = 21.5^\circ$ gradually shifts toward higher diffraction angles. Because the ionic radius of K⁺ is smaller than that of Cs⁺, the partial substitution of Cs⁺ by K⁺ in the CsPbBr₃ lattice induces lattice contraction, resulting in lowered lattice parameters and a decreased interplanar spacing (*d*). In line with Bragg's law:

$$2d\sin\theta=n\lambda$$

1

A decrease in the interplanar spacing (*d*) gives rise to an elevation in the diffraction angle (2θ) to satisfy the diffraction condition. Consequently, the diffraction peak shifts toward higher diffraction angles. This peak shift provides clear evidence that K⁺ ions were successfully incorporated into the CsPbBr₃ lattice, resulting in lattice contraction. [39 Fig. 2b. illustrates the crystal structure of cubic CsPbBr₃PQDs, in which Cs, Pb, and Br atoms are represented by red, green and cyan spheres, separately. Subsequent to the introduction of K⁺ ions, the crystal structure remained in the cubic phase, suggesting that partial substitution of Cs⁺ by K⁺ did not alter the crystal symmetry.

Figure 2 illustrated the typical TEM and HRTEM images of KCsPbBr₃ PQDs glass calcined at 600°C. The inset showed the size distribution histogram of KCsPbBr₃ PQDs. As evidently demonstrated in the above picture, PQDs with an average particle size of ~ 2.4 nm displays an even distribution in the glass matrix in Fig. 2a, which aligns with the results of the previous XRD pattern. The high-resolution electron microscope image in Fig. 2b further confirms the presence of CsPbBr₃ and Cs₄PbBr₆ in KCsPbBr₃ PQDs glass. The lattice fringe spacing of 2.938 Å and 2.676 Å corresponds to the (002) crystal plane of cubic CsPbBr₃ crystal and the (404) crystal plane of the Cs₄PbBr₆ crystal, separately. As a consequence, PQDs can still distribute uniformly on the glass substrate in the form of less than the Bohr radius (~ 7 nm) under high temperature calcination and emit uniformly green light.

Figure 3a presents a typical TEM image of the K_{0.2}Cs_{0.8}PbBr₃ PQDs glass subsequent to heat treatment at 600°C for 2 h. As illustrated in the image, dark nanocrystals are uniformly distributed near the edge of the glass particle, suggesting the successful in situ formation of K_{0.2}Cs_{0.8}PbBr₃ PQDs within the glass matrix. Figure 3b depicts the particle size distribution of the K_{0.2}Cs_{0.8}PbBr₃ PQDs glass. As evidenced by the TEM image and particle size analysis, the PQDs exhibit uniform dispersion within the glass matrix with an average diameter of ~ 2.3 nm. The observed particle size aligns with the XRD results, further

confirming the formation of uniformly distributed nanosized $K_{0.2}Cs_{0.8}PbBr_3$ PQDs in the glass matrix. The high-resolution transmission electron microscopy (HRTEM) image depicted in Fig. 3c further confirms the presence of $CsPbBr_3$ PQDs within the $K_{0.2}Cs_{0.8}PbBr_3$ quantum dot-embedded glass. The well-resolved lattice fringes reveal the high crystallinity of the embedded PQDs. The inset displays well-resolved lattice fringes of an individual $CsPbBr_3$ PQDs. The measured lattice fringe spacing is approximately 2.930 Å, which can be indexed to the (002) crystal plane of cubic $CsPbBr_3$. As demonstrated in Fig. 3d, the selected-area electron diffraction (SAED) pattern exhibits distinct diffraction rings that can be indexed to the (-202) crystal plane of cubic $CsPbBr_3$. In comparison with the TEM and HRTEM results, these observations demonstrate that the $K_{0.2}Cs_{0.8}PbBr_3$ PQDs remain uniformly dispersed within the glass matrix. It is paramount to note that it has an average particle size below the exciton Bohr diameter (approximately 7 nm) subsequent to heat treatment at 600°C. Such nanoscale confinement is beneficial for maintaining their exceptional green photoluminescence.[40]

Figure 3. (a) Typical TEM image of $K_{0.2}Cs_{0.8}PbBr_3$ quantum dot-embedded glass subsequent to heat treatment at 600°C for 2 h. (b) Particle size distribution of the embedded $K_{0.2}Cs_{0.8}PbBr_3$ PQDs. (c) HRTEM image of $K_{0.2}Cs_{0.8}PbBr_3$ PQDs glass subsequent to heat treatment at 600°C for 2 h; the inset shows the lattice fringes of a $CsPbBr_3$ PQDs. (d) SAED pattern of the $K_{0.2}Cs_{0.8}PbBr_3$ PQDs glass.

Figure 4a and b present the high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) images of the $K_{0.2}Cs_{0.8}PbBr_3$ glass. The embedded quantum dots exhibit uniform image contrast without discernible regions of reinforced or lowered intensity, suggesting the absence of significant heavy-element segregation or phase separation and suggesting exceptional compositional homogeneity. Afterwards, STEM-EDS elemental mapping was performed to further verify the incorporation of K^+ into the $CsPbBr_3$ PQDs. As demonstrated in Fig. 4c, d, e and f, the spatial distributions of Br, Cs, K, and Pb closely coincide with the particle morphology, demonstrating a homogeneous distribution of all constituent elements throughout the PQDs in the absence of detectable elemental enrichment or segregation. Notwithstanding the fact that the K^+ signal is relatively weak on account of its low concentration, it is uniformly distributed across the entire particle, providing direct evidence for the successful incorporation of K^+ into the $CsPbBr_3$ PQDs. In comparison with the XRD results, these observations further confirm that K^+ is incorporated into the $CsPbBr_3$ crystal lattice.

3.2. Photoluminescence Properties of PQDs Phosphors

Figure 5a depicts the photoluminescence excitation (PLE) and PL spectra of $KCsPbBr_3$ PQDs glass subsequent to heat treatment at 600°C for 120 min. The $CsPbBr_3$ glass heat-treated at 600°C for 120 min exhibits a green emission peak at 521 nm under an excitation wavelength of 365 nm, which is characteristic of $CsPbBr_3$ PQDs. With the augmentation of K^+ doping concentration, no conspicuous shift in the emission peak position is observed. In particular, the PL intensity first increases and subsequently decreases with the augment of K^+ doping concentration. The maximum PL intensity is

achieved at a K doping concentration of 0.2 mol%. In comparison with the undoped CsPbBr₃ glass, the PL intensity of the sample with 0.2 mol% K doping is reinforced by approximately 17 times. With the emission wavelength fixed at 521 nm, the excitation spectra measured over the wavelength range of 280–480 nm demonstrate that the optimal excitation peak remains at approximately 365 nm for all samples.

Figure 5b depicts the UV–Vis absorption spectra of the CsPbBr₃ and K_{0.2}Cs_{0.8}PbBr₃ PQDs glasses subsequent to heat treatment at 600°C for 2 h. Both samples exhibit a distinct absorption peak at approximately 510 nm, which is the characteristic absorption peak of CsPbBr₃. After K⁺ doping, the absorption edge shifts slightly toward shorter wavelengths, suggesting a negligible elevation in the optical band gap. This result aligns with the slight lattice contraction caused by the smaller ionic radius of K⁺ in contrast to Cs⁺. Figure 5c reveals the association between $(\alpha h\nu)^2$ and the photon energy ($h\nu$) for the CsPbBr₃ and K_{0.2}Cs_{0.8}PbBr₃ quantum dot-embedded glasses subsequent to heat treatment at 600°C for 2 h. The optical band gap energy (E_g) of the KCsPbBr₃ quantum dot-embedded glass was determined from the $(\alpha h\nu)^2$ versus $h\nu$ plot in accordance with the following equation:

$$(\alpha h\nu)^2 = A (h\nu - E_g)$$

2

where A refers to a proportionality constant, α defines the absorption coefficient, h is Planck's constant, ν symbolizes the photon frequency, and E_g denotes the semiconductor band gap. The E_g values of the CsPbBr₃ and K_{0.2}Cs_{0.8}PbBr₃ PQDs glasses heat-treated at 600°C were determined to be 2.34 and 2.36 eV, separately. The negligible increment in the band gap further confirms that K⁺ doping induces subtle adjustments to the crystal lattice, thus giving rise to corresponding changes in the electronic band structure of the material.[39]

Figure 3e reveals the time-resolved PL decay curves of the CsPbBr₃ and K_{0.2}Cs_{0.8}PbBr₃ PQDs glasses subsequent to heat treatment at 600°C for 2 h. The decay curves were fitted by adopting the following biexponential equation:

$$I(t) = A_1 \exp\left(\frac{-t}{\tau_1}\right) + A_2 \exp\left(\frac{-t}{\tau_2}\right)$$

3

where $I(t)$ represents the emission intensity, A is a constant, and τ denotes the decay lifetime. In contrast to the undoped CsPbBr₃ sample, both the fast and slow decay lifetimes of the K_{0.2}Cs_{0.8}PbBr₃ sample are prolonged to some degree. To be specific, τ_1 increases from 15.2 to 15.4 ns, τ_2 increases from 127.9 to 130.2 ns, and the average lifetime increases from 17.5 to 17.7 ns. As demonstrated by the prolonged lifetime, K⁺ doping effectively reduces the density of defect states in the material and suppresses the

nonradiative recombination of charge carriers, thereby heightening the luminescence efficiency.[39, 40] This result aligns with the reinforced PL intensity.

3.3. KCsPbBr₃ PQDs Glass Powders for Environmental Stability

To further evaluate the environmental stability of the K_{0.2}Cs_{0.8}PbBr₃ PQDs glass, the samples were immersed in deionized water or exposed to ambient air at 28 ± 2°C and a relative humidity of 66 ± 5%. As depicted in Fig. 6a, the sample immersed in deionized water retained ~ 98% of its initial PL intensity. On top of that, no conspicuous shift in the emission peak was observed after immersion for 42 days. The above phenomena jointly demonstrated exceptional water stability. As illustrated in Fig. 6b, the PL intensity of the sample exposed to air exhibited almost no noticeable decay throughout the test. Subsequent to the above steps, no substantial shift in the emission peak was observed after 70 days of air exposure, suggesting the exceptional air stability of the K_{0.2}Cs_{0.8}PbBr₃ PQDs glass. The outstanding environmental stability is primarily attributed to the dense glass matrix, which effectively inhibits the diffusion of moisture and oxygen. Consequently, the CsPbBr₃ PQDs embedded within the glass are protected against environmentally induced degradation of their structure and optical properties.

4. Conclusions

To sum up, KCsPbBr₃ PQDs ZBS glass powders were successfully developed via a two-step high-temperature solid-state method. As evidently suggested by the XRD results, K⁺ was incorporated into the CsPbBr₃ crystal lattice. Attributable to the protective effect of the ZBS glass matrix, the KCsPbBr₃ PQDs maintained a stable particle size of ~ 2.3 nm subsequent to heat treatment at 600°C. In particular, the K_{0.2}Cs_{0.8}PbBr₃ sample prepared at 600°C exhibited the best PL intensity, with a PL intensity ~ 17 times higher than that of the undoped sample, while its emission peak remained at approximately 521 nm. Simultaneously, the optical band gap increased from 2.34 to 2.36 eV, and the TRPL lifetime was slightly prolonged to 17.7 ns, suggesting elevated luminescence efficiency. Aside from that, the K_{0.2}Cs_{0.8}PbBr₃ glass retained ~ 98% of its initial PL intensity after immersion in deionized water for 42 days. No remarkable changes in either the PL intensity or emission peak position were observed after storage in ambient air at 28 ± 2°C and a relative humidity of 66 ± 5% for 70 days. This study provides a new strategy for developing high-performance and long-lived perovskite quantum dot-embedded glass luminescent materials for potential applications in solid-state lighting, displays, and optoelectronic devices.

Declarations

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contribution

Yuxin Yan (Data curation; Investigation; Methodology; Software; Validation; Writing-original draft; Writing-review & editing) Ailing Zou (Conceptualization; Data curation; Formal analysis; Funding acquisition; Project administration; Resources; Supervision) Yunan Lin (Supervision; Validation; Visualization) Haohao Cao (Validation; Visualization) Zheng Wei (Validation; Visualization) Guorui Li (Validation; Visualization) Yanjie Zhang (Formal analysis; Funding acquisition; Resources; Supervision) Jingjie Yu (Formal analysis; Funding acquisition; Resources; Supervision)

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Figures

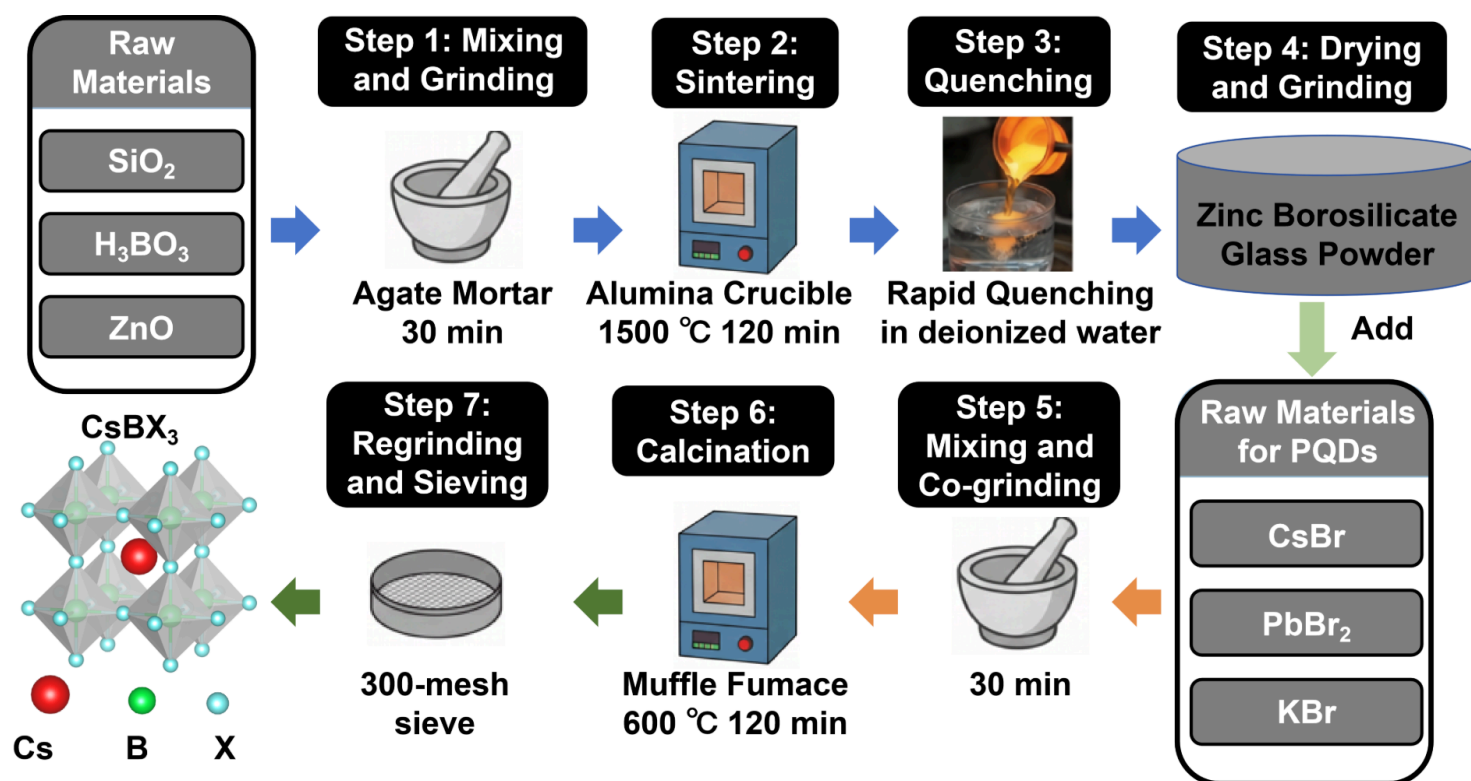


Figure 1

Schematic illustration of the synthesis process for KCsPbBr₃ PQDs embedded ZBS glass powder.

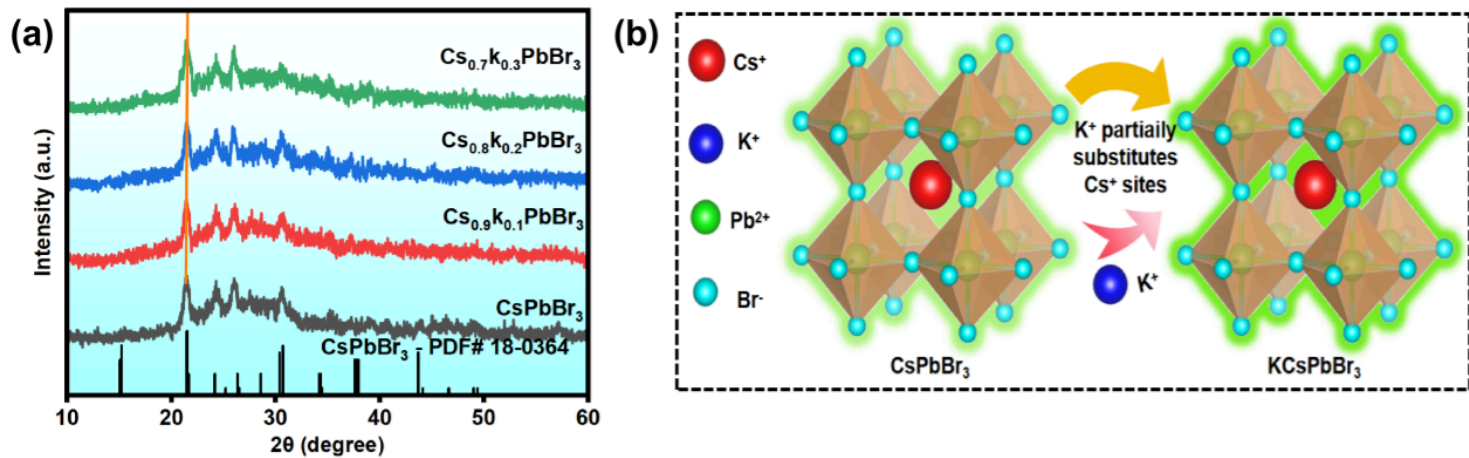


Figure 2

(a) XRD patterns of KCsPbBr₃ PQDs ZBS glass subsequent to heat treatment at 600 °C for 2 h. (b) Schematic crystal structure of cubic KCsPbBr₃ PQDs, illustrating the incorporation of K⁺ into the CsPbBr₃ lattice while preserving the cubic perovskite framework.

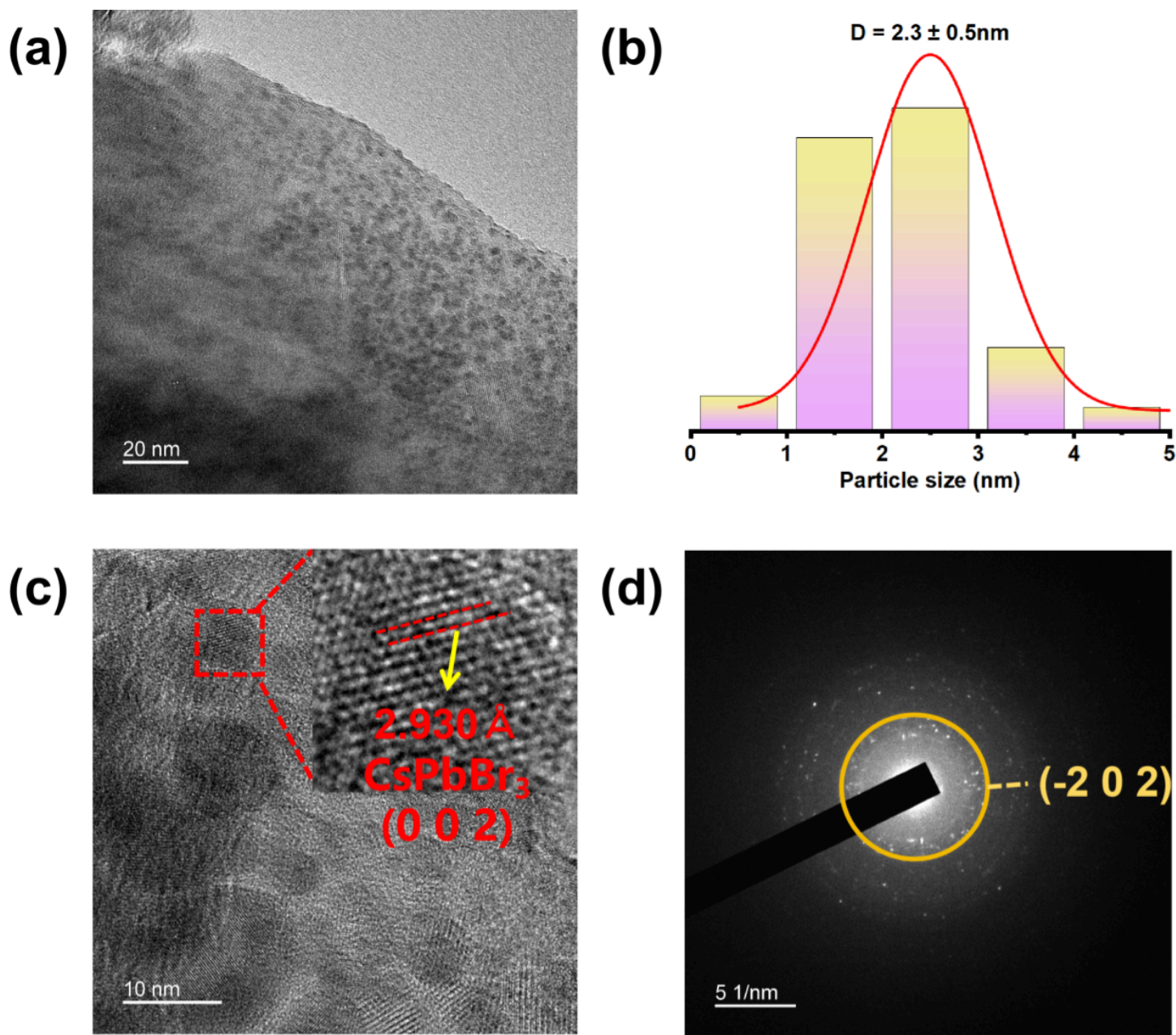


Figure 3

(a) Typical TEM image of $K_{0.2}Cs_{0.8}PbBr_3$ quantum dot-embedded glass subsequent to heat treatment at 600 °C for 2 h. **(b)** Particle size distribution of the embedded $K_{0.2}Cs_{0.8}PbBr_3$ PQDs. **(c)** HRTEM image of $K_{0.2}Cs_{0.8}PbBr_3$ PQDs glass subsequent to heat treatment at 600 °C for 2 h; the inset shows the lattice fringes of a $CsPbBr_3$ PQDs. **(d)** SAED pattern of the $K_{0.2}Cs_{0.8}PbBr_3$ PQDs glass.

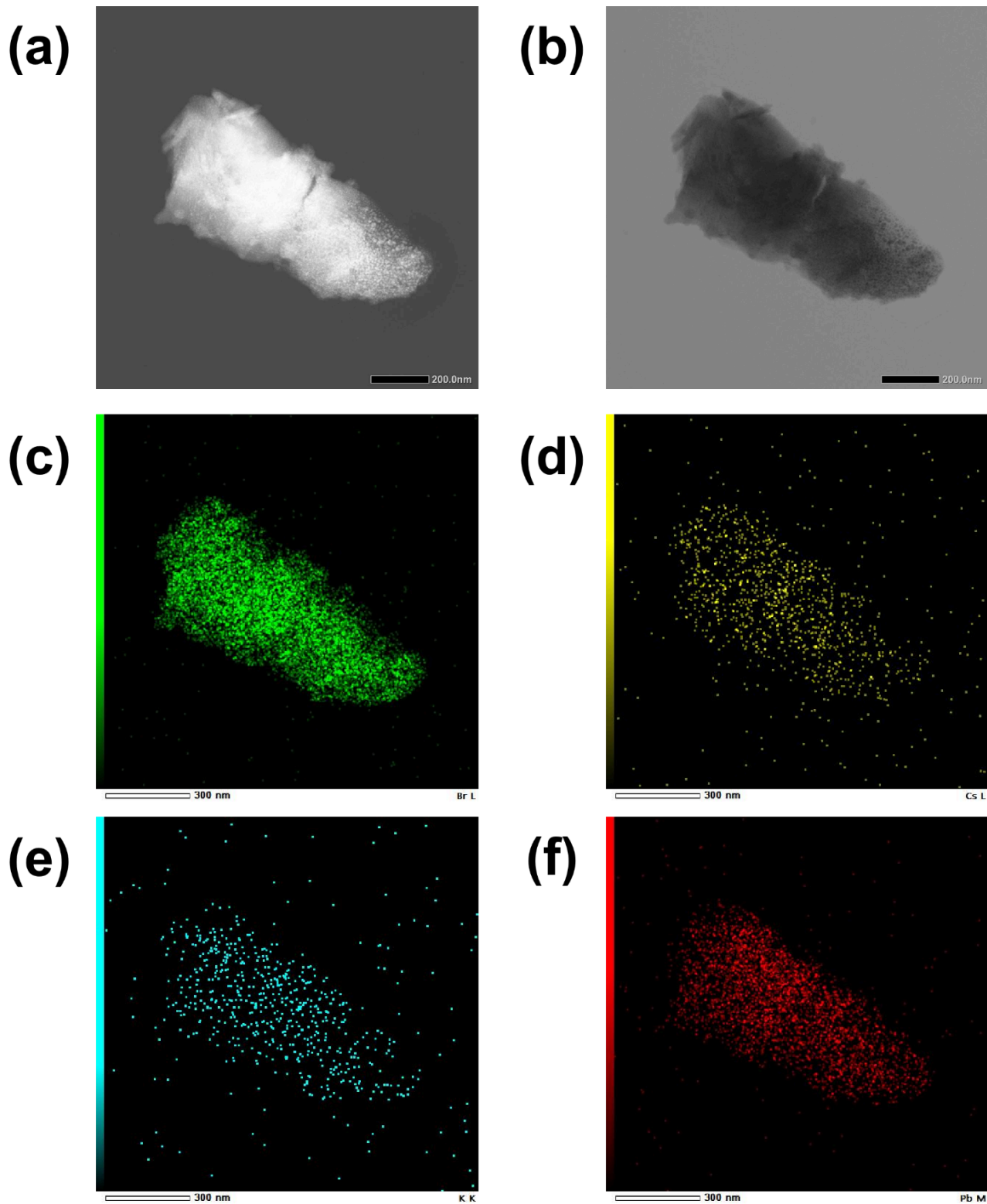


Figure 4

(a) Bright-field STEM image of the $\text{K}_{0.2}\text{Cs}_{0.8}\text{PbBr}_3$ glass in the aftermath of heat treatment at 600 °C for 2 h. **(b)** HAADF-STEM image of the same sample. **(c–f)** STEM-EDS elemental mapping images of Br, Cs, K, and Pb, separately.

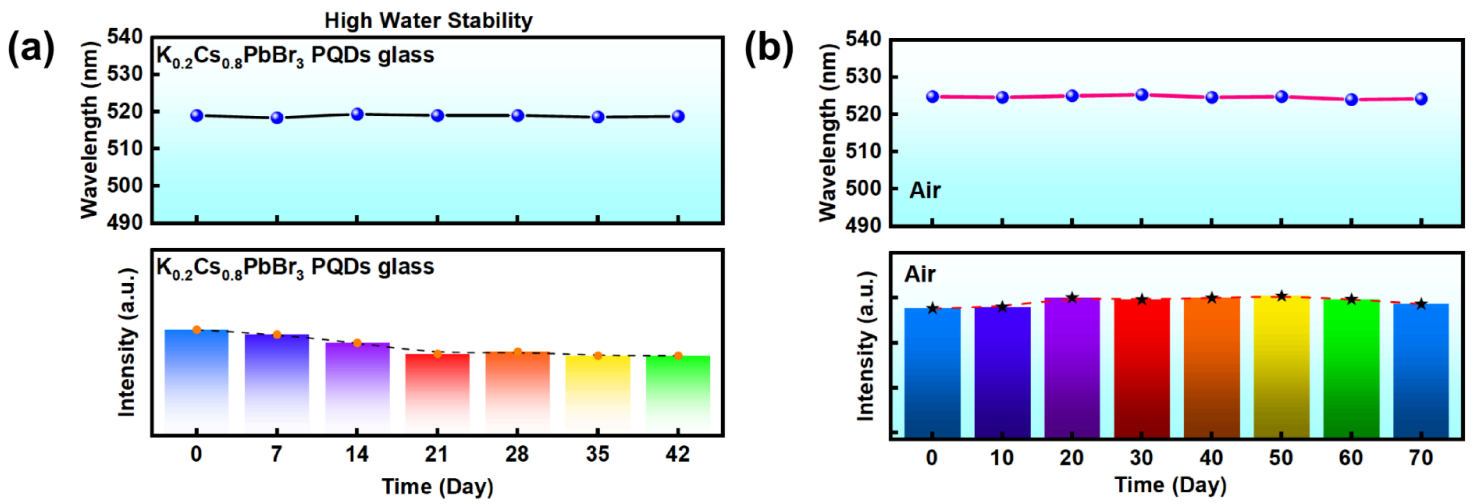


Figure 5

Environmental stability of the $K_{0.2}Cs_{0.8}PbBr_3$ PQDs glass under different storage conditions. (a) Evolution of the emission peak position and PL intensity as a function of immersion time in deionized water. (b) Evolution of the emission peak position and PL intensity as a function of storage time under ambient conditions (28 ± 2 °C and relative humidity of $66 \pm 5\%$).

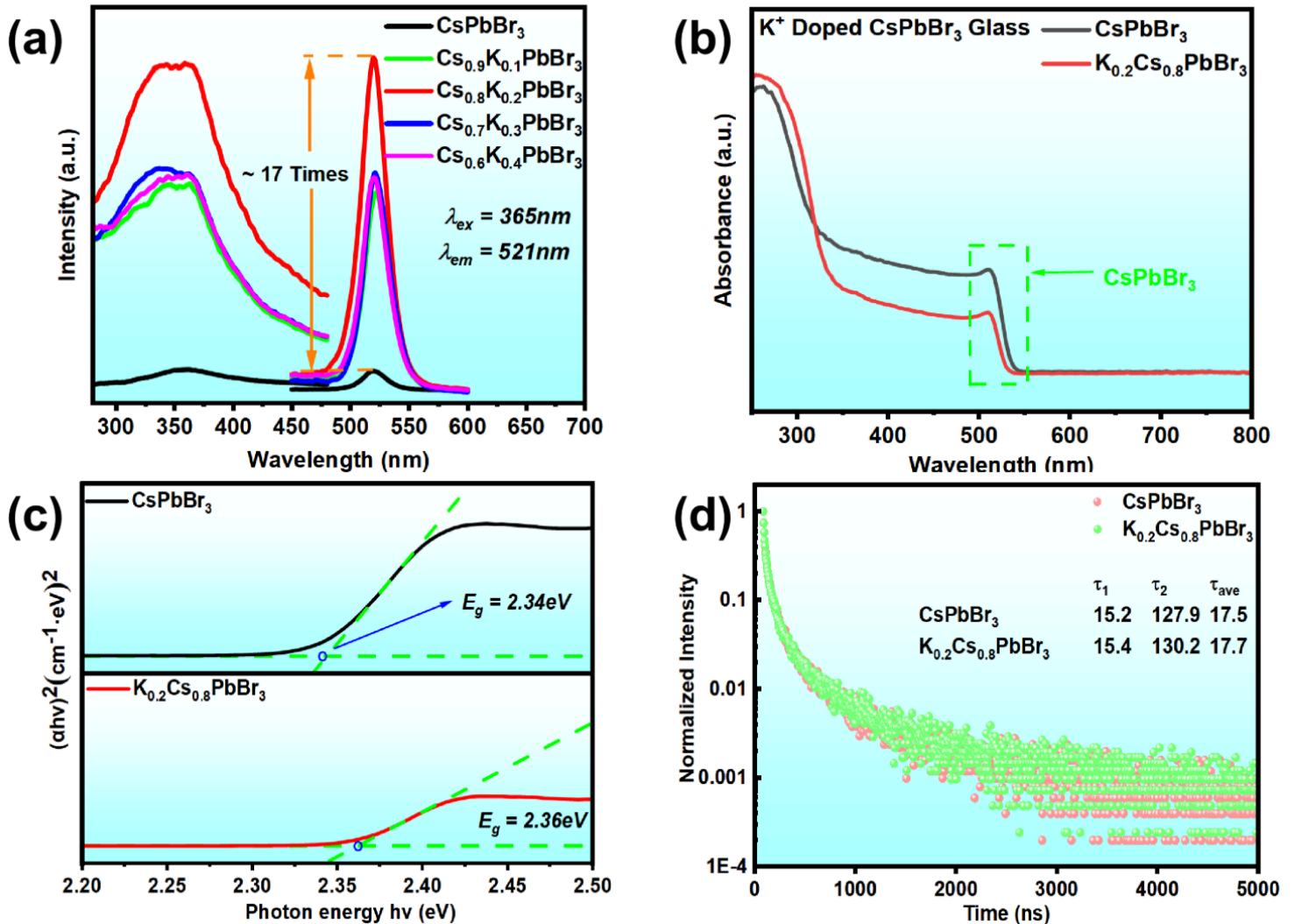


Figure 6

(a) PL and PLE spectra of KCsPbBr₃ PQDs glasses heat-treated at 600 °C, measured at an excitation wavelength of 365 nm and an emission wavelength of 521 nm, separately. **(b)** UV–Vis absorption spectra of the kCsPbBr₃ PQDs glasses heat-treated at 600 °C. **(c)** Tauc plots of $(ah\nu)^2$ versus photon energy ($h\nu$) for the KCsPbBr₃ PQDs glasses heat-treated at 600 °C. **(d)** Time-resolved PL decay curves of the samples heat-treated at 600 °C, measured under excitation at 365 nm and monitored at 521 nm.