

Research Article

Designable and highly stable emissive CsPbl₃ perovskite quantum dots/polyvinylidene fluoride nanofiber composites

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Abstract: All-inorganic CsPbI₃ perovskite quantum dots (QDs) with efficient pure red emission are broadly researched for applications as next-generation light emitters. Their optical stability against humidity and water, on the other hand, is a great barrier that needs to be significantly enhanced. In this study, polyvinylidene fluoride (PVDF) nanofibers (NFs) were synthesized by electrospinning and CsPbI₃ QDs were effectively composited into the interior of the PVDF NFs matrix by dip-coating. The CsPbI₃ QDs/PVDF NFs composites exhibited a high red photoluminescence (PL) quantum yield of $\sim 11\%$ with the PL peak slightly blue shifted compared with the bare QDs. Due to the high hydrophobicity, porosity, and excellent thermal insulation of the PVDF NFs and uniform dispersion of the QDs in the NFs network, the composite membrane demonstrated excellent water, thermal, and UV light resistance. In addition, the CsPbI₃ QDs/PVDF NFs composites were revealed to exhibit high designability, flexibility, and repeatability, indicating significant potential for different light-emitting applications.

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1. Introduction

Highly luminescent all-inorganic CsPbX₃ (X = Cl, Br, I) perovskite quantum dots (QDs) have been generally employed as an extremely competitive alternative in solid-state lighting, lasers, photodetectors, displays, and visible light communication as a result of their unique optical properties [1-3]. Due to the ionic nature of the perovskites, surface dangling bonds, and uncoordinated atoms, bare CsPbX₃ QDs displayed poor stability at high temperature and ambient conditions, leading to severe photoluminescence (PL) quenching and limiting their further development [4,5]. In comparison to bromide and chloride analogs, red-emissive CsPbI₃ with a relatively modest tolerance factor is thermodynamically unstable at room temperature, which can be easily transformed to the non-perovskite orthorhombic phase induced by humidity or water [6,7]. Currently, many attempts are being devoted to improving the stability of CsPbI₃ QDs, including ligand treatment on the surface and encapsulation with polymers or porous templates.

The surface chemistry of CsPbX₃ perovskite QDs is essential for surface modification and improvement of PL quantum yield (PLQY) and stability. The strategies for stabilizing the surface of CsPbI₃ QDs by changing ligand functional groups can massively improve the stability of CsPbI₃ QDs in air or water [8-13]. During the synthesis process, incorporation of 2-hexyldecanoic

acid with two short branched chains to replace oleic acid with long chains and using ZrO₂ coated CsPbBr₃ NCs can maintain their colloidal stability and contribute to efficient radiative recombination [14,15]. Moreover, a nanometer-thin lithium fluoride interlayer allowed surface tension relief within the microscopic device structure, enabling robust integration of high-efficiency light-emitting devices with self-aligned photolithography [16]. Recently, combining CsPbBr₃ QDs with a polymeric matrix to produce luminous composites displayed boosted stability and enhanced PL performance [17–21]. Despite numerous studies on CsPbBr₃/polymer composites, only a few attempts have been focused on CsPbI₃ [22–24]. In addition, the interfacial interaction between the QDs and polymers and PL properties is also massively influenced by the structure of the polymeric matrix.

One-dimensional nanofibers (NFs) offer the unique characteristics of high porosity, bending elasticity, and stretchability [25]. Electrospinning is an effective and low-cost method to produce multifunctional NFs from a variety of materials. Furthermore, the electrospun NFs may be easily produced as membranes to facilitate their applications. The composites of CsPbX₃ perovskite nanocrystals inside electrospun polymer NFs have been observed to exhibit both strong emissions and excellent stability [26–28]. Polyvinylidene fluoride (PVDF) polymer has outstanding hydrophobicity and thermal and mechanical stability [29], making it a suitable option for the production of CsPbI₃ QDs/PVDF NFs to improve the stability of CsPbI₃ QDs.

In this study, PVDF NFs membranes were fabricated by electrospinning and CsPbI₃ QDs were then evenly dispersed inside the NFs structure utilizing a dip-coating approach. The obtained CsPbI₃ QDs/PVDF NFs composite membrane showed almost identical excellent PL emission properties as that of the CsPbI₃ QDs. The composites demonstrated remarkable stability in air and water, retaining about 80% of the original PL intensity for 3 days in water because of the high hydrophobicity of the PVDF NFs. The composites were also discovered to have high stability under high temperature and UV light illumination. In addition, the composites were shown to have high designability, flexibility, and repeatability for light-emitting applications.

2. Experimental

2.1. Materials

Polyvinylidene fluoride (PVDF, Mw~400,000, Macklin), *n*-hexane (\geq 99%, Macklin), ethyl acetate (\geq 99%, Macklin), acetone (99%, Sinopharm), *N*,*N*-dimethylformamide (DMF, 99.5%, Sinopharm), cesium carbonate (Cs₂CO₃, 99.9%, Alfa Aesar), lead iodide (PbI₂, 99.9%, Alfa Aesar), oleylamine (OLA, 80-90%, Alfa Aesar), oleic acid (OA, Alfa Aesar), and 1-octadecene (ODE, >95.0%, Alfa Aesar) were used as received without further purification.

2.2. Synthesis of CsPbl₃ QDs

Cesium oleate (Cs-oleate) precursor was prepared by mixing 0.407 g of Cs₂CO₃, 1.25 mL of OA, and 20 mL of ODE in a 50 mL 3-neck flask, degassed in vacuum at 120 °C for 1 h, and heated at 150 °C at nitrogen atmosphere until full reaction of Cs₂CO₃ with OA. Then, 5 mL of ODE, 0.5 mL of OLA, 0.5 mL of OA, and 0.087 g of PbI₂ were added into a 25 mL 3-neck flask, degassed in vacuum at 120 °C for 1 h, and heated at 165 °C at nitrogen atmosphere for 10 min. After that, 0.4 mL of Cs-oleate precursor preheated at 120 °C was injected into the solution, which was cooled down to room temperature after 5 s. Finally, the solution was centrifuged at 9,000 rpm for 5 min. The obtained precipitate was washed with 1 mL of *n*-hexane and 3 ml of ethyl acetate, and dispersed in 4 mL of *n*-hexane to obtain the CsPbI₃ QDs solution.

2.3. Preparation of CsPbl₃ QDs/PVDF NFs composites

The precursor solution was prepared by dissolving 10 wt% PVDF in a DMF and acetone mixture (mass ratio of 6:4) at 80° C for 3 h, which was then stocked in a syringe with a stainless-steel

needle of 0.25 mm. The electrospinning process was carried out with an applied voltage of 12 kV at a fixed collection distance of 18 cm. The obtained PVDF NFs were dipped in the CsPbI₃ QDs solution for 12 h, washed with *n*-hexane, and finally dried in vacuum to obtain the composite membranes.

2.4. Characterizations

X-Ray diffraction (XRD) patterns were collected by an X-ray diffractometer (Bruker D8 Advance). The PL spectra were measured using a spectrofluorometer (Zolix OmniFluo900) with an excitation wavelength of 470 nm. For temperature-dependent PL measurement, the films were kept at each temperature point for 25 min before testing. The absorption and transmittance spectra were collected using an UV-Vis spectrometer (Shimadzu UV-3600). The morphology and elemental mapping analysis were carried out using a scanning electron microscopy (SEM, JSM-7800F) and a transmission electron microscopy (TEM, JEM-2100F). Fourier transform infrared (FTIR) spectra were recorded using a spectrophotometer (Thermo Scientific Nicolet-iS5).

3. Results and discussions

Schematic representation of the electrospinning setup and synthetic route of CsPbI₃ QDs/PVDF NFs composites is demonstrated in Fig. 1(a), (b). It is discovered that the as-synthesized PVDF NFs membrane was transparent, but after the incorporation of colloidal CsPbI₃ QDs, the composite membrane became semi-transparent (Fig. 1(c)). Upon UV light irradiation, the composite membrane presented bright red PL emission (Fig. 1(d)).

The morphology of the CsPbI₃ QDs synthesized by hot-injection method was investigated by TEM. The particle size of the QDs ranged from 7–13 nm, with an average value of about 10 nm (Fig. 2(a) and Fig. S1). From the high-resolution TEM (HRTEM) image (Fig. 2(b)), the QDs displayed an interplanar distance of d = 6.2 Å, corresponding to the (100) interplanar spacing of cubic phase α -CsPbI₃ [30]. The XRD pattern of the QDs matched with the simulated α -CsPbI₃ (Fig. 2(c)). The characteristic 2θ peaks at 14.2° and 28.3° can be clearly observed, corresponding to the (100) and (200) lattice planes of the α -CsPbI₃, respectively. The typical UV–Vis absorption and PL spectra of the bare CsPbI₃ QDs were displayed in Fig. 2(d), with the emission peak located at 692 nm. These findings demonstrated that the as-synthesized CsPbI₃ QDs were of cubic perovskite phase.

The as-prepared NFs showed straight and randomly oriented morphology (Fig. 3(a)). The diameters ranged from 800–2000 nm with an average value of about 1.49 µm (Fig. S2). Large gaps were obviously observed among the electrospun PVDF NFs, indicating the high porosity of the NFs. The energy-dispersive X-ray spectroscopy (EDS) results showed that a limited quantity of CsPbI₃ QDs was loaded on the surface of the PVDF NFs (Fig. S3), indicating that the majority of CsPbI₃ QDs were located inside the PVDF NFs matrix. The TEM images of the nanocomposites also verified that the $CsPbI_3$ QDs were evenly dispersed in the PVDF NFs network (Fig. 3(b), (c), and Fig. S4). The average size of the CsPbI₃ QDs in the composite membrane was almost identical to that of the bare QDs, proving that the CsPbI₃ QDs in the PVDF NFs membranes did not agglomerate. Compared to the in situ formed perovskite/polymer composites, the homogeneity of the QDs in the nanocomposites is considerably enhanced. The XRD patterns of the PVDF NFs and composite membranes are shown in Fig. 3(d). The composite membrane showed a sharp peak at 20.4°, corresponding to the (110) lattice plane of β -PVDF [31]. In addition, a small wide peak at 14.1° was also observed, corresponding to the (100) lattice plane of α -CsPbI₃. The optical transmittance spectra are illustrated in Fig. 3(e). The transmittance of the composite membrane decreased significantly in the region of 400-800 nm with a clear absorption edge as compared with the PVDF NFs membrane, which is due to the absorption of CsPbI₃ QDs in the PVDF NFs [32,33]. From the FTIR spectra (Fig. 3(f)), both of the samples showed CF₂ stretching vibration at 1182 cm^{-1} , PVDF crystalline phase vibration at



Fig. 1. (a) Electrostatic fiber spinning setup. (b) Schematic synthetic route of the CsPbI₃ QDs/PVDF NFs composites. Photographs of (c) the PVDF NFs and CsPbI₃ QDs/PVDF NFs composite membranes and (d) the composite membrane under UV irradiation.

 1072 cm^{-1} , the amorphous phase at 870 cm^{-1} , and CH₂ variable angle vibration at 1400 cm^{-1} . This suggests that the CsPbI₃ QDs were physically adsorbed onto the PVDF NFs without the formation of chemical bonds. The vibration peaks at 2856 and 2922 cm⁻¹ of the nanocomposites were due to the C-H bond between OA and OLA [34]. The above findings evidently indicated that the CsPbI₃ QDs were successfully composited into the interior of the PVDF NFs matrix [35].

The optical characteristics of the CsPbI₃ QDs/PVDF NFs composites were further analyzed. The PL peak of the as-synthesized composites was located at 688 nm (Fig. 4(a)). This minor PL blue shift was also observed in previous reports [36]. The PLQY of both the bare CsPbI₃ QDs film and composite membranes were about 11% (Fig. S5), with no significant decrease in PLQY after loading CsPbI₃ QDs into PVDF NFs. This is a great advantage over the composite films fabricated by thermal or UV polymerization which would deteriorate the perovskite QDs with a lower PLQY.

The stability of the composites in air and water was then investigated. The PL peak position and intensity remained constant after 7 days of storage in air (80% relative humidity), which confirmed that the nanocomposites were extremely stable in ambient conditions. After 60 days of storage in air, the PL intensity was still 45% of its original value. Furthermore, after 3 days of direct immersion in water, the PL intensity of the composite membrane maintained 80% of the original value (Fig. 4(b)). The variation of PL intensity of the composite membranes as a function of the aging time in air and water was summarized in Fig. 4(c) and Table S1. In addition, the PL peak of the nanocomposites shifted from 688 to 683 nm after 60 days in air and to 681 nm after 7





Fig. 2. (**a**, **b**) TEM images of the bare CsPbI₃ QDs. (**c**) XRD pattern of the typical CsPbI₃ QDs. (**d**) Absorption and PL spectra of the CsPbI₃ QDs dispersed in *n*-hexane.



Fig. 3. (a) SEM image of the PVDF NFs. TEM images of (b) the CsPbI₃ QDs/PVDF NFs composites and (c) the typical CsPbI₃ QDs embedded in the PVDF NFs. (d) XRD patterns, (e) transmittance spectra, and (f) FTIR spectra of the PVDF NFs and CsPbI₃ QDs/PVDF NFs composite membranes.



Fig. 4. Variation of the emission spectra of the CsPbI₃ QDs/PVDF NFs composites with storage time in (**a**) air and (**b**) water. (**c**) Time-dependent PL intensity of the composites. PL decay curves of the composites after different aging time in (**d**) air and (**e**) water. (**f**) Photographs of the (i) bare CsPbI₃ QDs and (ii) composite membrane upon excitation of 365 nm UV light after immersion in water for different time intervals.

days in water. The slight PL peak blue shift after aging may come from the partial decomposition of the QDs [37]. Time-resolved PL decay spectra after storage in air and water were shown in Fig. 4(d), (e). The decay lifetimes of the nanocomposites gradually reduced (Table S1), which may be caused by a rise in the defects in CsPbI₃ QDs which served as nonradiative recombination channels when stored at high humid conditions for a long time [38,39]. The composite membrane illustrated excellent water resistance and remained emissive after immersion in water for 7 days, as shown in Fig. 4(f). For comparison, the stability of the bare CsPbI₃ QDs in water was studied, and PL quenching was seen within 4 min. The water contact angle of the as-prepared PVDF NFs membrane was measured to be 119.84° (Fig. S6). The highly hydrophobic characteristic of the electrospun PVDF NFs matrix can effectively prevent the inner CsPbI₃ QDs from direct contact with water molecules, resulting in excellent water stability.

Temperature-dependent PL spectra were collected to examine the thermal stability (Fig. 5(a), (b)). The CsPbI₃ QDs/PVDF NFs composites maintained about 70% of the original PL intensity at a temperature of 340 K, and were still emissive up to 380 K (Fig. 5(c)). For comparison, bare CsPbI₃ QDs films were almost non-emissive when the temperature reached 340 K, indicating that the composite membranes owned significantly enhanced thermal stability than the bare CsPbI₃ QDs. The improved heat resistance of the composites was attributed to the excellent thermal insulation property of PVDF polymer, which weakened the heat-induced PL quenching of CsPbI₃ QDs embedded in PVDF NFs. The PL peaks of both the bare CsPbI₃ QDs and composites were slightly blue-shifted as the temperature increased (Fig. S7), which may be caused by the heat-induced lattice expansion. This abnormal blue shift was also reported in other halide perovskites [40,41].

The light stability was also investigated under 365 nm UV illumination. As can be seen in Fig. 5(d), after 30 h of exposure to UV light, the CsPbI₃ QDs/PVDF NFs composites still retained about 87% of the initial PL intensity, while the bare CsPbI₃ QDs films had only about 30% of the initial value after 10 h of irradiation. The greatly enhanced light stability was probably due to the



Fig. 5. Temperature-dependent PL spectra of (a) $CsPbI_3$ QDs and (b) $CsPbI_3$ QDs/PVDF NFs composites. The variation of PL intensity of the $CsPbI_3$ QDs films and $CsPbI_3$ QDs/PVDF NFs composites under (c) high temperature and (d) UV light illumination.



Fig. 6. Photographs of the CsPbI₃ QDs/PVDF NFs composites showing bright red luminescence under UV excitation: (a) patterned composites (SUEP) by cutting; a membrane in (b) its original state, and under versatile deformation including (c) bending, (d) rolling, (e) folding, and (f) twisting.

unique fibrous structure of PVDF NFs that can provide physical domain limiting space inhibiting the aggregation of perovskite QDs.

Furthermore, we have analyzed the mechanical features of the CsPbI₃ QDs/PVDF NFs composites. As shown in Fig. 6(a), luminous patterns (SUEP) under UV excitation can be easily produced by cutting the composite membranes directly. No corresponding curing molds were required to design patterns. In addition, no change in luminous color and intensity under UV excitation was observed after versatile deformation such as bending, rolling, folding, and twisting (Fig. 6(b)-(f)). The excellent mechanical properties such as high designability, flexibility, and easy preparation of the CsPbI₃ QDs/PVDF NFs composites show significant advantages over perovskite materials embedded in rigid polymers (for example, PMMA, PS, etc.).

4. Conclusion

In summary, we have prepared porous PVDF NFs by electrospinning. The CsPbI₃ QDs/PVDF NFs composites presented similar PL emission as the bare CsPbI₃ QDs. The composite membrane retained 80% of its initial PL intensity after immersion in water for 3 days, but PL quenching of the bare QDs occurred within 4 min. At an elevated temperature of 340 K, the composites retained about 70% of the original PL intensity, whereas the bare QDs films became almost non-emissive. In addition, the composites also showed high resistance to UV light aging, retaining about 87% of the initial PL intensity after 30 h. In contrast, the PL intensity of the bare QDs films was reduced to just about 30% of the initial value after 10 h of UV light exposure. The high hydrophobicity and thermal insulation of the PVDF NFs, as well as the uniform dispersion of CsPbI₃ QDs in PVDF NFs, contributed to the outstanding stability of the composite membranes against water, heat, and UV irradiation. Furthermore, the composites also exhibited exceptional mechanical characteristics, which can be easily patterned by cutting and is still highly emissive under versatile deformation such as bending, rolling, folding, and twisting. Highly emissive composites that are designable and stable may have a wide range of application prospects in flexible optical and energy-conversion devices.

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Data availability. Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

Supplemental document. See Supplement 1 for supporting content.

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