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Highly stable blue perovskite light-emitting electrochemical cell realized by synergistic optimization of polyethylene oxide and lithium salts

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Abstract

High-performance blue light-emitting devices are essential for solid-state lighting and full-color displays. Among them, metal halide perovskite semiconductors show great potential in solution-processed blue light-emitting devices. The mixed halide Cl/Br perovskite provides one of the easiest ways to achieve emission in the blue region. However, its inherent ion movement and separation lead to halide segregation under light and voltage driving. Here, polyelectrolytes and a salt additive are leveraged to suppress the halide segregation in the single-layer CsPbBr_{3-x}Cl_x perovskite light-emitting electrochemical cells (LECs). The ions from the salt addition are transported by the electrolyte, improving charge injection and maintaining the intrinsic perovskite lattice of luminescence, resulting in pure and sustained blue emission. These devices achieve stable blue electroluminescence at 490 nm with a maximum brightness of 242 cd/m². These results indicate polymer and salt additive inclusion increases photoluminescence efficiency, suppresses halide segregation, and induces thin-film smoothness and uniformity.

1. Introduction

Artificial lighting is one of the main driving forces of modern society. As we pay more and more attention to energy conservation and global warming, finding a new efficient light source and new lighting technology has become a hot spot. Organic light-emitting diode (OLED) has emerged in light-emitting devices due to its high efficiency, good toughness and thinness. However, the disadvantages of the complicated vacuum evaporation process and the use of unstable cathode metals result in significantly increased OLED production costs, limiting their further citations. In 1995, based on the characteristics of a polymer OLED device, Pei et al. fabricated a polymer light-emitting electrochemical cell (LEC) by blending polymer electrolyte in the polymer light-emitting layer[1]. Due to its high luminous efficiency, low operating voltage and the fact that it does not need to be prepared in an inert gas, LECs have been highly valued in scientific research and industrial applications[2, 3].

Blue electroluminescence is the basis for full-color display. However, achieving saturated, efficient and stable blue light emission has always been challenging for LEC optoelectronic devices[4, 5]. Perovskite has become a unique material for LEC devices due to its adjustable band gap, wide spectral range and high photoluminescence quantum yield (PLQY)[6-8]. The mixed halide Cl/Br perovskite provides one of the easiest ways to achieve emission in the blue region and gradually blue-shifted with the increase of Cl content. Due to the ionic nature of perovskite materials, under external stimuli (electric field, optical radiation), a shift in the emission spectrum is usually observed in mixed halide perovskites by halide segregation[9, 10]. Therefore, a reasonable structural design and a simple chemical method are needed to inhibit the movement of perovskite ions while maintaining efficient operation.

In this work, we demonstrate a simple and stable single-layer perovskite light-emitting electrochemical cell (PeLEC) device using a mixture of halide perovskite and ionic additives. The device is composed of ITO/PEDOT:PSS/CsPb(Br, Cl)₃:PEO:PVP:LiPF₆/Al. We increased the solubility of Cl in the solvent by special solvent engineering to avoid the short circuit problem of optoelectronic devices caused

by uneven film pinholes. Among them, LiPF_6 , as an ionic additive, produces a differential motion with the ionic motion of the perovskite itself, which inhibits the segregation of mixed halides. Therefore, our work is not only beneficial to improve the thin-film smoothness and uniformity, but also to optimize the spectral stability of perovskite films and devices. This novel chemical treatment may provide an alternative strategy to solve the spectral instability of blue-emitting perovskites, laying the foundation for future high-performance, cost-effective, solution-processable wide-gamut display applications.

2. Experimental Section

Materials: PEO (M.W. > 5000000), Lead (II) bromide (PbBr_2 ; 99.99% trace metal basis), Cesium chloride (CsCl ; 99.99% trace metal basis), lithium hexafluorophosphate (LiPF_6 ; 99.99%), methanol (MeOH ; >99.9%), and dimethyl sulfoxide (DMSO; anhydrous >99.9%) were purchased from Sigma Aldrich. poly(ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) were purchased from Xi'an Polymer Light Technology Corp. All chemicals are used as received without further purification.

Solution Preparation: PbBr_2 (320 mg) was dissolved in DMSO (2 mL) to obtain solution 1. CsCl (80 mg) was dissolved in MeOH (3 mL) to obtain solution 2. PEO (10 mg) was added to DMSO (1 mL), and the mixture was stirred on a heating plate at 50 °C (200 rpm) overnight to obtain solution 3. Solution 1 (167 μl) was added to 1 ml solution 3, and the mixed solution was heated and stirred at 100 °C for 10 minutes. Then solution 2 (587 μl) was added to the mixed solution to produce a precipitate, and the turbid solution was heated to transparency at 150 °C. PVP (10 mg mL^{-1}) and LiPF_6 (4 mg mL^{-1}) were dissolved in an anhydrous DMSO solvent. PVP (20 vol % $\text{CsPb}(\text{Br}, \text{Cl})_3$ solution) and LiPF_6 (0.5 wt % $\text{CsPb}(\text{Br}, \text{Cl})_3$) were added to the $\text{CsPb}(\text{Br}, \text{Cl})_3$: PEO solution to form the final emitting layer composite.

The ITO/glass substrates were cleaned in a range of non-ionic detergent washing, water bath ultrasonic treatment, and UV ozone treatment. The PEDOT:PSS aqueous solution was filtered through a 0.45 μm GHP filter (1.3-1.7 %, Clevios AI 4083) and spin-coated onto a clean ITO layer at 4,000 rpm for 45 seconds, followed by annealing

in ambient air at 150 °C for 10 minutes. The prepared active layer precursor solution was spin-coated onto PEDOT:PSS at a speed of 2500 rpm, followed by vacuum treatment for 10 minutes and thermal annealing at 200 °C for 35 seconds. The film was transferred to a vacuum chamber and 10Å LiF and 800Å Al were deposited using a shadow mask.

Characterizations: SEM images were obtained by scanning electron microscope (SU8010, HITACHI, Japan). Steady-state PL spectra were obtained by using an Edinburgh Instruments (FLS1000) spectrometer. Time-resolved PL spectra were measured by the time-correlated single-photon counting method using an Edinburgh Instruments FLS1000 spectrometer. The samples were excited by a 375 nm pulsed diode laser. EL spectra of the PeLEDs were obtained by PR670. The current density-voltage-luminance characteristics of the PeLEDs were measured by using a Keithley 2400 source meter and a multimeter with a calibrated silicon photodiode. The crystal structures of these samples were analyzed in the range of 10°-50° by an X-ray powder diffractometer (Bruker D8 advance) using Cu-K α ($\lambda = 1.5405$ Å) radiation.

3. Results and Discussion

Fig. 1a shows the device architecture of the blue PeLEC: ITO/PEDOT:PSS (40 nm)/perovskite (30 nm)/LiF (1 nm)/Al (80 nm), where ITO and PEDOT:PSS are indium tin oxide and poly (3,4-ethylenedioxythiophene) polystyrene sulfonate, respectively. The schematic diagram of the preparation of mixed halide perovskite precursor solution and the thin film is shown in Fig. 1b. PbBr₂, PEO and LiPF₆ were dissolved in DMSO, while CsCl was dissolved in methanol. Then all of them were mixed to form a perovskite precursor solution. After that, the thin film was deposited from the precursor solution by one-step spin coating. The deposited thin film was annealed at 200 °C for 30 seconds after the vacuum treatment. The CsPb (Br, Cl)₃, CsPb (Br, Cl)₃: PEO, CsPb (Br, Cl)₃: PEO: PVP: LiPF₆ samples were denoted as PeLEC-B, PeLEC-P, and PeLEC-PL, respectively. The XRD curve of PeLEC-PL shows the main diffraction peaks at 15.3 °, 21.8 °, and 31.1 °, corresponding to the diffraction planes of (110), (112) and (220), respectively, as shown in Fig. 1c, which is consistent with the orthorhombic

(Pnma) structure in the previous reports[11]. The diffraction peak at 12.7° corresponds to the (102) diffraction plane of 0D Cs_4PbBr_6 (416 phase). The $\text{Cs}_4\text{Pb}(\text{Br}, \text{Cl})_6$ phase is a derivative phase of the CsPbBr_3 perovskite and mixed halide system, which can effectively passivate the defects in $\text{CsPb}(\text{Br}, \text{Cl})_3$ [12, 13]. The crystal structure of $\text{CsPb}(\text{Br}, \text{Cl})_3$ is schematically illustrated in Fig. 1d. Compared to PeLEC-PL, the XRD curve of PeLCE-P only shows weak diffraction peaks at 21.8° and 31.1° . However, no prominent diffraction peaks were observed from the XRD curve of PeLEC-B. While the diffraction peaks of the perovskite crystals may be obscured by the hump peak caused by the glass substrate, our results suggest that the inclusion of PEO and LiPF_6 has a significant positive effect on the formation of the perovskite phase.

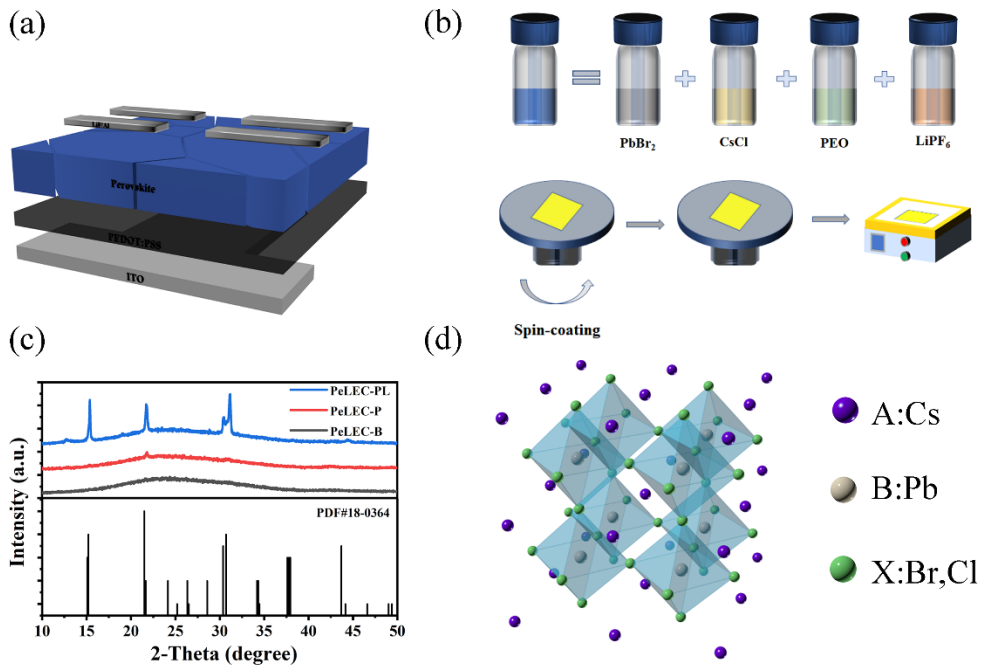


Fig. 1. (a) Device architecture of $\text{CsPb}(\text{Br}, \text{Cl})_3$: PEO: PVP: LiPF_6 Complex PeLEC. (b) Schematic diagram for the preparation of precursor solution and perovskite film. (c) X-ray diffraction (XRD) spectra of PeLEC-B, PeLEC-P and PeLEC-PL films. (d) Schematic of the Crystal structure of $\text{CsPb}(\text{Br}, \text{Cl})_3$.

Previous studies have shown that morphological defects in perovskite thin films can lead to high density trap states, hindering charge transfer characteristics and limiting blue luminescence[14-16]. In order to further verify the influence of polymers and ionic additives on the performance of perovskite thin films, the perovskite thin

films were characterized by scanning electron microscopy (SEM), as seen in Fig. 2. The SEM image of the PeLEC-B film has low surface coverage and high pinhole density, which is the main reason for the short circuit and instability of the device structure[17, 18]. Compared with PeLEC-B, the surface coverage of PeLEC-P film was improved, and the grains become more compact, but there are still pinholes[19]. Also, PeLEC-PL film has the least voids and holes among grains, showing uniform film morphology and good coverage. Therefore, the addition of polymers and salts helps to form smooth, pinhole-free films that contribute to the stability of the device.

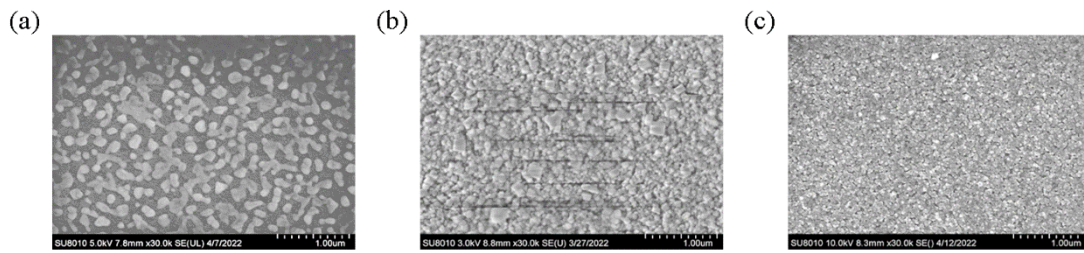


Fig. 2. SEM images of (a) PeLEC-B, (b) PeLEC-P, and (c) PeLEC-PL thin films.

As shown in Fig. 3a, the PeLEC-B has a weak PL intensity, while the PeLEC-PL exhibits a strong PL intensity. Fig. 3b shows the time-resolved PL (TRPL) decay curves of the three films, and the photoluminescence lifetime was obtained by fitting the photoluminescence decay curves with a double exponential function. Obviously, compared with the 5.2 ns of the PeLEC-B film, PeLEC-PL shows a much longer photoluminescence lifetime of 22.1 ns. This may suggest that the addition of lithium salts reduces the non-radiative recombination sites point, increasing the lifetime of the carriers in the perovskite film. In order to investigate the light-irradiation-caused halide segregation issue, all three samples were irradiated under a UV lamp with high intensity (210 mW/cm^2) for ten minutes. The emission peak of the PeLEC-B experienced a severe red shift from 479 to 512 nm, while the PeLEC-PL displayed stable blue emission, as shown in Figs. 3c and d, respectively. Compared to that of the PeLEC-B film, the PeLEC-PL film shows extremely suppressed halide migration, illustrating that the crystalline quality has got a large improvement. The improved crystalline quality can decrease the non-radiative recombination, and also lead to a potential passivation effect, which should give rise to improvement in the performance of PeLEC-PL[20, 21].

Moreover, it is interesting to find that the PL intensity of PeLEC-PL was enhanced after UV irradiation, as shown in Fig. 3d. This phenomenon has been also observed by other researchers, and this type of photo repair effect was attributed to the fact that illumination can promote the annihilation of defect states inside the film [22, 23].

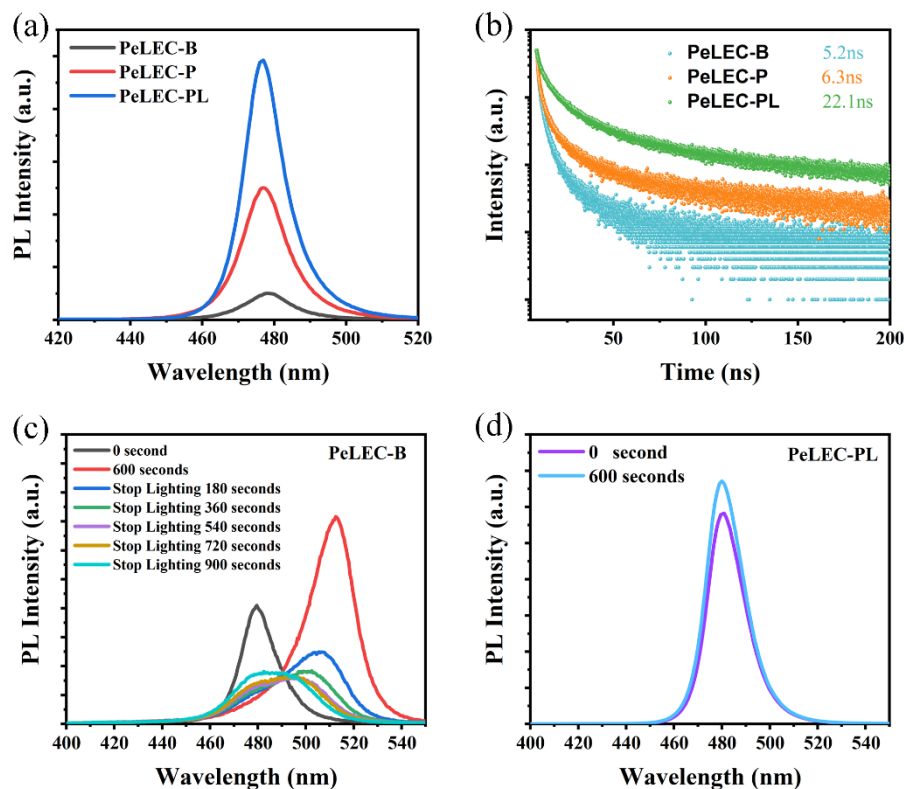


Fig. 3. (a) Photoluminescence spectra of the PeLEC-B, PeLEC-P, and PeLEC-PL. (b) Time resolved PL decay curves of the PeLEC-B, PeLEC-P, and PeLEC-PL. PL spectra of the PeLEC-B (c) and PeLEC-PL (d) under intense UV excitation with different time.

The current-brightness-voltage characteristics of PeLEC-P and PeLEC-PL devices were shown in Figs. 4a and b. PeLEC-P shows a minimum brightness of 105 cd m^{-2} , while PeLEC-PL displays a considerable increase in maximum brightness of 242 cd m^{-2} and current density. Considering salt additives can provide a significant amount of mobile ions in the films, these mobile ions would selectively replace the intrinsic perovskite ions to form an electric double-layer (EDL) at the electrode interface to facilitate electron and hole injection[24]. The EDL formation process is shown in Fig. 4c. The initial LEC state indicates that perovskite ions are ordered according to their crystal structure, and Li^+ and PF_6^- ions are uniformly distributed in the whole active

layer. When an appropriate voltage is applied to the two electrodes, PF_6^- anions drift to the anode and are oxidized to generate P-type doping, while Li^+ cations drift to the cathode and are reduced to generate N-type doping, thus forming double electrodes near the two electrodes[25,26]. Therefore, the addition of salt additives produces differential ion motion and inhibits the inherent ions motion of mixed halide perovskite. It is believed that this operation mode can inhibit the phase separation of halide peroxide and significantly improve the performance of PeLEC.

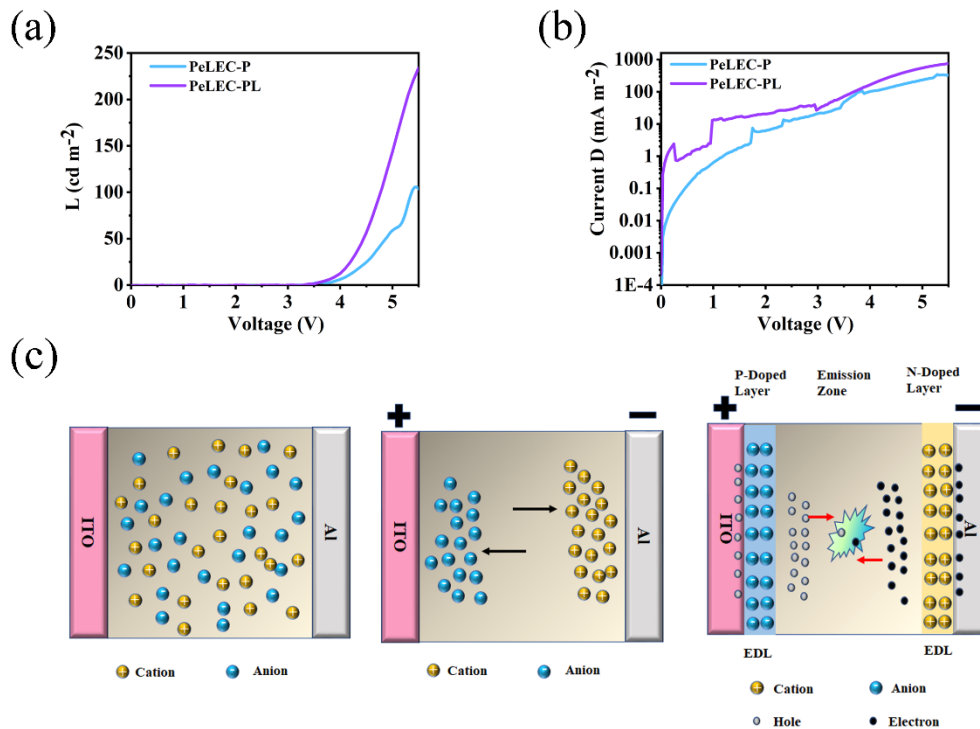


Fig. 4. (a) Luminance–voltage curves and (b) current density–voltage curves of PeLEC devices. (c) schematic diagram of EDL formation.

To investigate the effect of ionic additives on the stability of EL spectra, EL spectra of PeLEC-P and PeLEC-PL with different external biases were measured, as shown in Fig. 5a and 5b. With increasing voltage, PeLEC-P shows a significant instability of the EL spectrum with a shift of 18 nm. The EL spectral instability was attributed to halide segregation[27, 28]. This is because the increasing voltage causes the redistribution of perovskite ions to form voids and traps, limiting the luminescence. As shown in Fig. 5b, the EL peak shift of PeLEC-PL is negligible because the addition of LiPF_6 balances the necessary charge redistribution and limits the negative effect of the electric field on the perovskite structure due to the movement of the inherent ions.

The International Commission on Illumination (CIE) color coordinates under different voltages are shown in Figs. 5c and d. PeLEC-P shows green luminescence at 4v, and the color coordinate moves from (0.1081,0.4763) to (0.0864,0.7022) with the voltage increase. The PeLEC-PL shows blue luminescence at 4v, and the coordinate moves from (0.0771,0.2746) to (0.0639,0.3497). This result further indicates that the inclusion of lithium salt inhibits the segregation of mixed halide perovskite.

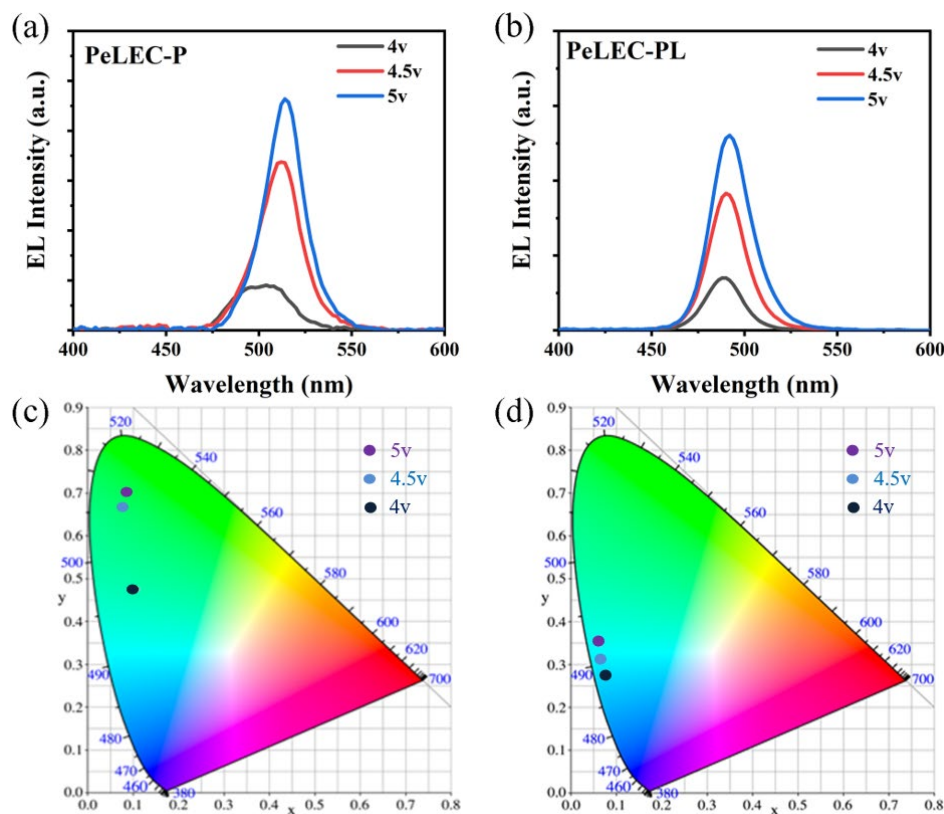


Fig. 5. EL spectra over time for (a) PeLEC-P and (b) PeLEC-PL operating at various voltages. International Commission on Illumination (CIE) coordinates corresponding to the EL spectra of (c) PeLEC-P and (d) PeLEC-PL at different voltages.

4. Conclusions

In summary, the smooth and uniform mixed halide perovskite films have been prepared by introducing polyelectrolytes and salt additives. An electroluminescent device based on CsPb (Br, Cl)₃: PEO: PVP: LiPF₆ film was designed. The emission center wavelength was 490 nm, and the maximum brightness was 242 cd/m². In addition, the addition of polyelectrolytes and salt additives also helps to improve spectral stability. In a wide bias range, the electroluminescent spectrum has only a slight

shift, which is significantly better than the electroluminescent devices based on traditional mixed halide perovskites. Our findings may provide an alternative strategy to address the spectral instability of blue-emitting perovskites LEC, laying the foundation for future high-performance, cost-effective, solution-processable wide-gamut display applications.

Declaration of interests

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.

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