Ultrasonication-Assisted Trace Amount Solvent Synthesis of Cs_4PbBr_6 Crystal with Ultra-Bright Green Light Emission

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Abstract

Cs_4PbBr_6 crystals have been synthesized by an ultrasonication-assisted trace amount of polar solvent method. The prepared green Cs_4PbBr_6 material was found to have high photoluminescence (PL) brightness and 10 ns PL lifetime. The prepared green light emitting device can achieve the maximum brightness of 3.8×10^9 cd/m^2, which is nearly 100 times higher than the wick. The white light emitting device based on Cs_4PbBr_6 crystals has better performance than commercial products, which can cover 32.3% of the colour gamut and achieves 107.3% coverage for International Commission on Illumination (CIE)1931 and 84.8% coverage for CIE1976.

Keywords: Cs_4PbBr_6; halide perovskite; light emitting; ultrasound; trace solvent; ultra high brightness

1. Introduction

Clean energy and efficient energy conversion are essential to achieve sustainable development. Halide perovskites are promising materials that can efficiently convert solar energy into electricity as well convert electricity into light energy. Significant progresses in halide perovskites ABX_3 ($A=CH_3NH_3^+$, Cs^+; $X=I^-$, Br^-, Cl^-, SCN^-) for solar cells make them to be the most promising materials for the next generation of solar cell. [1-5] Meanwhile, halide perovskites have attracted lots of research interest due to the high-performance optoelectronic properties, such as light-emitting,[6-13] photodetectors (PDs) [14, 15] and lasers.[16, 17] ABX_3 perovskites have a perfect three-dimensional (3D) structure consisting of corner-sharing $[BX_6]^{4+}$ octahedra, with A^+ ion filling the gap between adjacent $[BX_6]^{4+}$ octahedra, achieving super low trap state density of 10^{10} cm^{-3}.[18]

However, the joint angle of the three-dimensional perovskite structure prevents quantum confinement of charge carriers, resulting in low optical emissions. Furthermore, 3D perovskite ABX_3 has inherent vulnerability to moisture and heat, and long-term stability is still the biggest challenge. Therefore, it is an urgent goal to find other materials for solid halide perovskite illumination and to have high luminosity. In the commercialization of halide perovskites light emitting devices, the colour gamut and brightness can be increased by adjusting the structural size of halide perovskites. The A_4BX_6 structure materials are promising candidates. They are more stable due to the isolated $[BX_6]^{4+}$ octahedron is inserted in all dimensions through the A^+ ions in the crystal lattice and separated from each other. However, the synthesis method of Saidaminov et al. requires that $PbBr_2$ and CsBr to be dissolved in large amount of dimethyl sulfoxide (DMSO) and stirred for 1 hour.[19] The solution was filtered and heated to 120 °C for extra 3 hours. Then the sample was washed with DMSO and dried at 100 °C. Bastiani et al prepared the material with 1 ml of precursor solution (1 ml of DMSO, 0.25 mol of $PbBr_2$ and 0.25 mol of CsBr dissolved).[20]

The precursor was inserted into a closed container containing 4 mL of diethyl ether (DE). After as long as 48 hours, the immiscible interface between DE and DMSO formed crystals in the crystallization flask. Zhang's method for preparing Cs_4PbBr_6 nanocrystals requires a complex procedure in which Cs-oleic acid precursor, n-hexane and oleic acid (OA) were quickly injected into the flask with vigorous stirring with $PbBr_2$, HBr, OA and oleylamine.[21] These reported methods require either very long time, higher temperature or more complicated steps in the preparation of Cs_4PbBr_6 and in some cases there are certain restrictions for moisture and oxygen. They are disadvantages for the preparation of Cs_4PbBr_6 in large quantities. Meanwhile, a large amount of organic solvent has a huge impact on cost and environment.

Here, we discovered a one-step method for the rapid synthesis of Cs_4PbBr_6 high quality crystals at room temperature with trace amount of solvent, which is feasible for large-scale industrial preparation with low costs and less environmental concerns. It has also been found that the Cs_4PbBr_6 crystals have higher PL brightness and can be used for ultra-bright light-emitting device, achieving the maximum brightness of 3.8×10^9 cd/m^2.

2. Material and methods

2.1 Preparation of Cs_4PbBr_6: Green-DMSO

Preparation of Cs_4PbBr_6-Green: 0.1 mol of CsBr (Cesium bromide, 99.9%, Aladdin, China) and 0.025 mol of $PbBr_2$ (lead (II) bromide 99%, Aladdin, China) were placed in a 5 ml test tube. Then 10 μl of DMSO (dimethyl sulfoxide 99.8%, Aladdin, China) was added dropwise. The resulting mixture was then sealed and placed in an ultrasonic cleaner (Fisher Scientific FS110D, 100 W) for sonication (sample Green-DMSO). Duration of sonication can be found in Fig. 1c. The sample was dried at 140 °C.

2.2 Preparation of Cs_4PbBr_6: White-EtOH

0.1 mol of CsBr and 0.025 mmol of $PbBr_2$ were placed in a 5 ml test tube. Then 5 ml of ethanol was added dropwise. The resulting mixture was then sealed and placed in an ultrasonic generator for sonication (sample White-EtOH). Duration of sonication can be found in Fig. 1d. The sample was dried at 140 °C.

2.3 Preparation of light emitting device

$CaAlSiN_3:Eu^{2+}$ (Shenzhen Looking Long technology Co., China) and $BaMgAl_{10}O_{17}:Eu$ (Shenzhen Looking Long technology Co., China) were used as the blue light (450 nm) source and the red light (630 nm) source of the light emitting device, respectively. They were packaged by epoxy resin (Shenzhen Looking Long technology Co., China). White light emitting was excited by a 365 nm optical core (Shenzhen Looking Long technology Co., China). The Green-DMSO sample was prepared in a matrix powder and excited using a 365 nm optical core for green light emitting device.

2.4 Characterization

Cs_4PbBr_6 was imaged by a field emission scanning electron microscope (ZEISS Merlin Compact) at an accelerating voltage of 10-15 KV. The X-ray diffraction pattern was taken by an X-ray diffractometer (XRD-6000, Shimadzu, Japan) with Cu(Kα) radiation ($\lambda = 1.54$ Å). PL spectra were taken using Princeton Instruments SpectraPro HRS-750 (with superconducting nanowire single photon detector). The light emitting devices were powered and tested by Keithley meter (2400 and 6487) with photodetector (Digi-Key, USA).[8] Emission spectra (devices) were collected on F-380 fluorescence spectrometer (Tianjin Gangdong Sci. & Tech. Development Co., Ltd., China)

3. Results and discussion

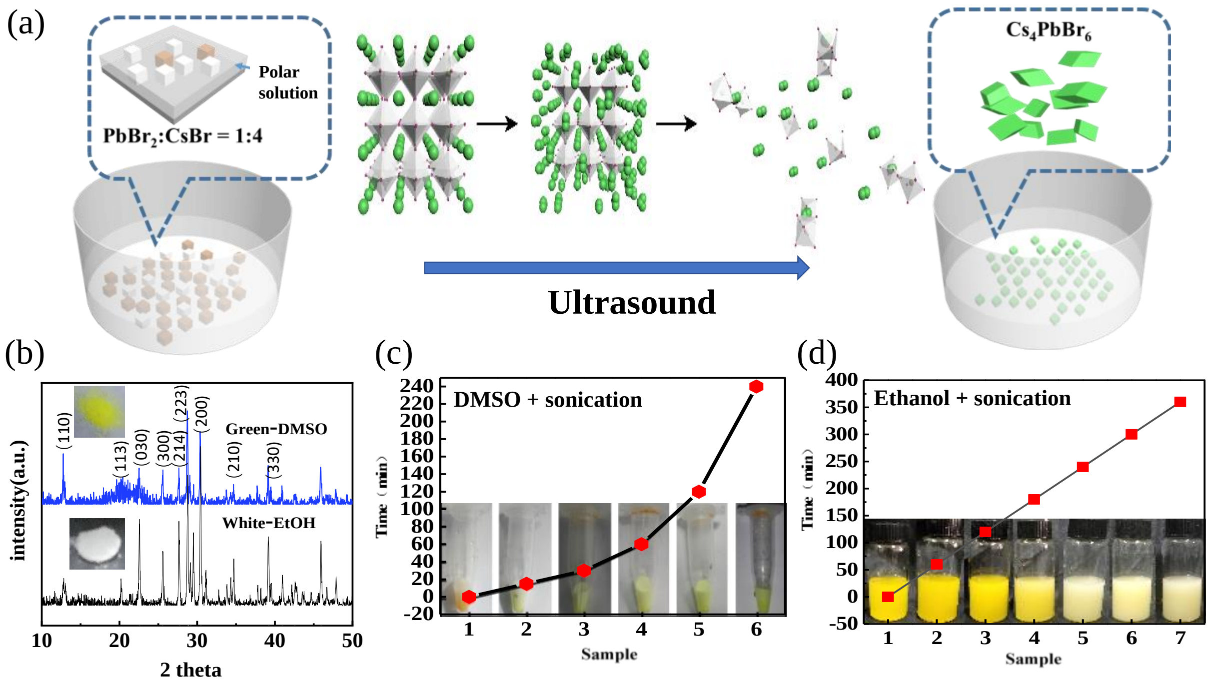

Fig. 1. (a) Synthesis of Cs_4PbBr_6 crystals; (b) XRD spectra of the powder for sample Green-DMSO (2th spot, 20 mins) and White-EtOH (7th spot, 350 mins); (c) ultrasonic synthesis of sample Green-DMSO with trace amount of DMSO; (d) ultrasonic synthesis of sample White-EtOH by ethanol.

In **Figure 1a**, 10 μl of DMSO was added in the mixture of 0.1 mol CsBr and 0.025 mol $PbBr_2$ (4: 1 ratio). The resulting mixture was then sealed and placed in an ultrasonic generator for sonication (sample Green-DMSO). It is worth to note that with the help of ultrasonication, Cs_4PbBr_6 can be formed faster with trace small amount of DMSO. As can be seen in **Figure 1c**, only 20 minutes (2nd dot in) is required to quickly prepare the Cs_4PbBr_6 crystals with the aid of ultrasonication. After drying in an oven, a yellow-green powder of Cs_4PbBr_6 was obtained as shown in the upper left corner in **Figure 1b**. If the trace amount solvent was changed from DMSO to ethanol, no Cs_4PbBr_6 crystals could be synthesized. As the ethanol was increase to 5 mL, a white bulk Cs_4PbBr_6 (sample White-EtOH) could be synthesized. As shown in **Figure 1d**, the colour changed obviously at the time of 250 mins (5th dot) and it cost approximately 300 mins (6th dot) to finish the reaction, which is 15 times longer than sample Green-DMSO. Moreover, energy-dispersive X-ray spectroscopy (EDS) results indicate that there are more impurities in sample White-EtOH (Table **S1**), while the atomic ratio for sample Green-DMSO is very close to theoretic ratio (Table **S2**). With this method, we could not synthesize Cs_4PbBr_6 crystals with other solvents, such as water or isopropanol. The polarity of DMSO is very high, which makes it a better solvent for this reaction. With the additional assistance of ultrasonication, this reaction can be done quickly. In the case of less polar ethanol, it has lower solubility and cause longer time to react. Even through the polarity of water is higher than DMSO. However, water decomposes the final product (Cs_4PbBr_6 crystals) immediately, which making water not a good solvent. Thus, the organic solvent DMSO with high polarity is the best choice for this reaction, which dissolve reactants quickly and barely dissolve product.

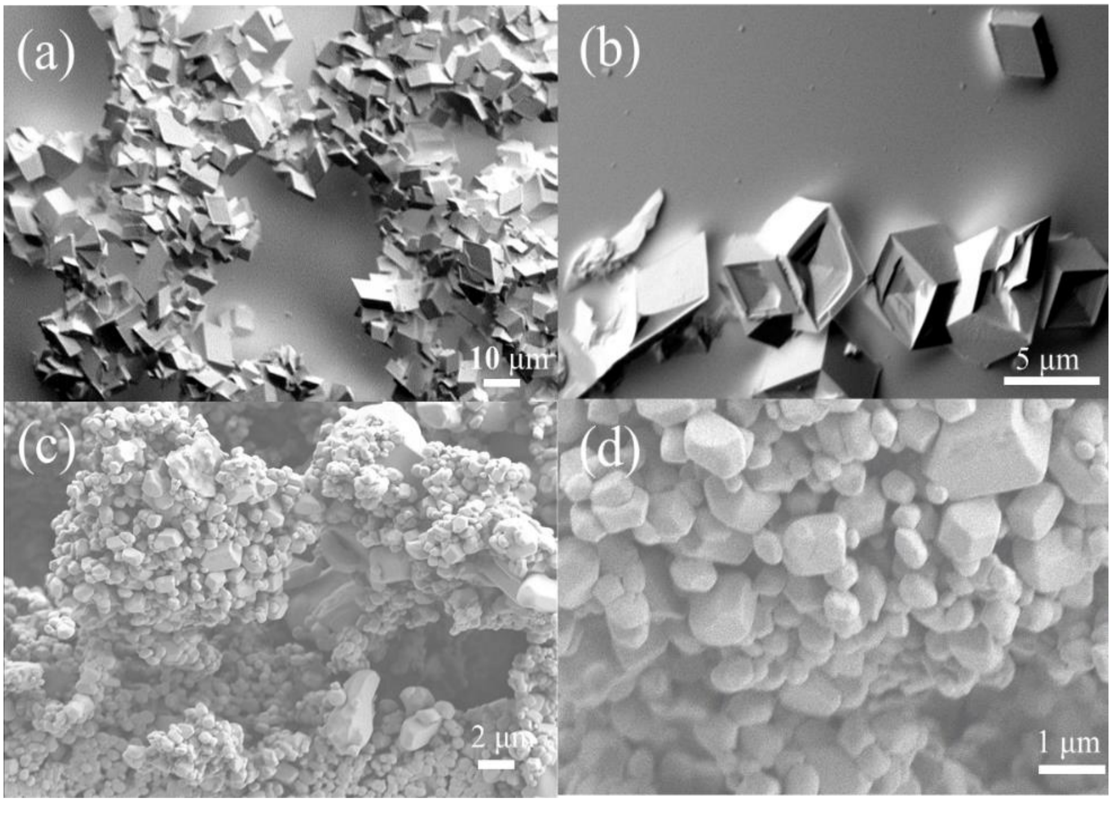

Fig. 2. SEM image of the sample Green-DMSO(a) and (b); sample White-EtOH (c) and (d).

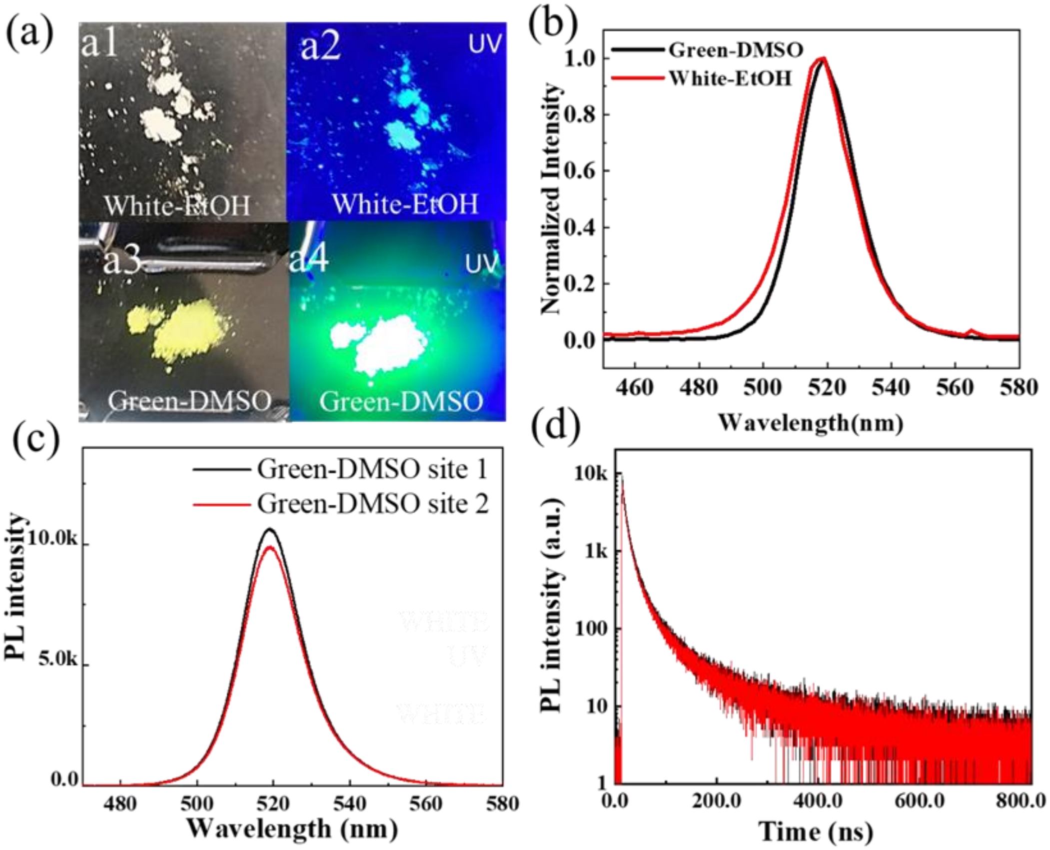

Fig. 3. (a) Optical images of sample Green-DMSO and sample White-EtOH under visible light (left) and ultraviolet light (right); (b) solid state PL spectra for sample Green-DMSO and sample White-EtOH; (c) PL for sample Green-DMSO at different spots; (d) solid state PL lifetime for sample Green-DMSO

Scanning electron microscopy (SEM) was used to image the morphology for the crystals of these two samples. As can been seen in **Figure 2a** and **2b**, sample Green-DMSO prepared with trace amount of DMSO was found to be well-shaped crystals of 2-8 μm. It has a typical Cs_4PbBr_6 crystal morphology.[22] As a comparison, the sample White-EtOH prepared with ethanol are shown in **Figure 2c** and **2d**. No good shape of Cs_4PbBr_6 crystals can be found even if at different synthetic conditions, such as different precursor ratios as shown in **Figure S1.** Cs_4PbBr_6 crystal is unstable under transmission electron microscopy (TEM) beam (supporting information).

Photoluminescence (PL) measurements were used to test the synthesized samples. In **Figure 3a, a1** and **a3** are optical images for sample Green-DMSO and White-EtOH under visible light, respectively; **a2** and **a4** are the corresponding optical images under the UV light. Sample Green-DMSO shows much stronger photoluminescence than sample White-EtOH. Solid state PL spectra for both samples were shown in **Figure 3b**. The emission peaks for both Green-DMSO and White-EtOH are at 519 nm, with the full width at half maximum (FWHM) at about 19 and 23 nm, respectively. The narrower FWHM for sample Green-DMSO indicates better colour purity than sample White-EtOH. Notably, the FWHM of 19 nm for sample Green-DMSO is narrower than previously reported Cs_4PbBr_6, including powder (~23 nm),[19] bulk crystals (24 nm)[20] and uniform size nanocrystals (23 nm).[23] Steady state PL spectra for sample Green-DMSO at different spots show almost the same peak profile indicating the uniformity of the synthesized Cs_4PbBr_6 crystal. In **Figure 3d**, the fluorescence lifetime was found to be about 10 ns for sample Green-DMSO, which is less than those in literature, such as 17.7 ns for Cs_4PbBr_6 in ref.[24] The PL measurement results indicates that the Cs_4PbBr_6 crystals prepared by ultrasonication-assisted trace amount solvent method have a better colour purity.

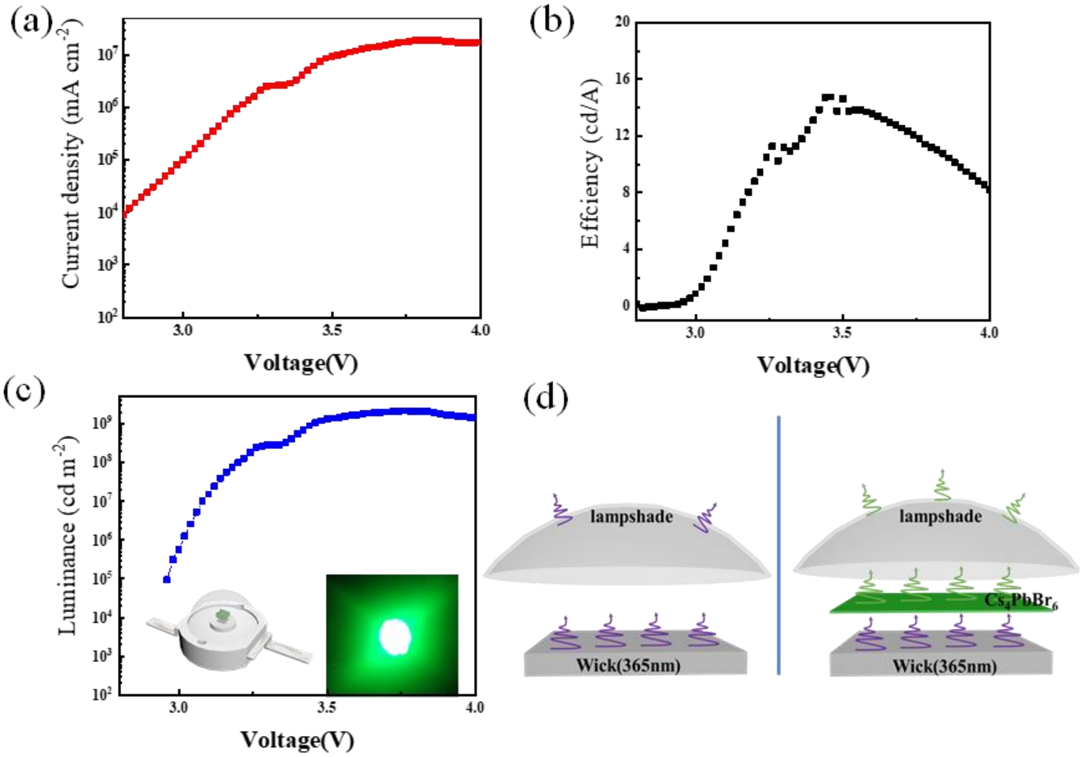

Fig. 4. Green light emitting device. (a) current density; (b) current efficiency and (c) luminance versus voltage characteristics of light emitting device; (d) schematic diagram of green light emitting device.

The green light emitting device was excited by a wick (**Figure S2-S4**). As shown in **Figure 4a**, the detector reached the maximum current density of 2.2×10^7 mA/cm^2 at 3.8 V. The current efficiency reached 14.8 cd/A at 3.5 V (**Figure 4b**). In the inset of **Figure 4c** shows the green light emitting device with ultra-high brightness of 3.8×10^9 cd/m^2, which is nearly 100 times higher than the wick (**Figure s2-s4**). A related video can be found in supporting information. **Figure 4d** are schematics displaying a better penetration of green light from Cs_4PbBr_6 micro crystal comparing with an ultraviolet light. The performance of light emitting device including PL is directly related to the quality of synthesized material. As can be seen from the SEM images, the material synthesized by ultrasonication-DMSO method used here shows very high quality Cs_4PbBr_6

crystals with smooth surfaces and sharp edges comparing with the material prepared with ethanol. These well-shaped micro crystals play the role of resonance cave during the light emitting with less dissipation.

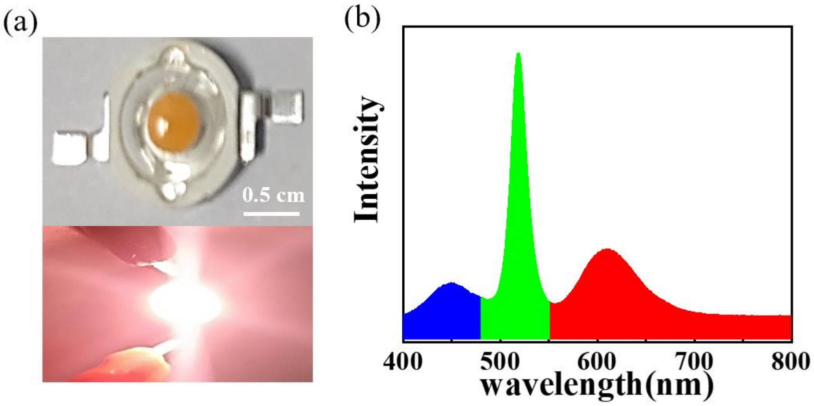

Fig. 5. (a) Optical images of a light emitting device prepared using a commercially available nitride ($CaAlSiN_3:Eu^{2+}$) and $BaMgAl_{10}O_{17}$: Eu mixed with Cs_4PbBr_6 crystal from sample Green-DMSO; (b) luminescence spectrum of the light emitting device.

In order to demonstrate the practicality of sample Green-DMSO, the ultrasonication-assisted trace amount solvent synthesized Cs_4PbBr_6 micro crystals were mixed with commercially available nitride ($CaAlSiN_3:Eu^{2+}$) with red fluorescent emission at 630 nm and $BaMgAl_{10}O_{17}$: Eu material with blue fluorescent emission at 450 nm to assemble a white light emitting device, which was excited by a 365 nm optical core. The pictures in **Figure 5a** demonstrate that the device with the ultrasonication synthesized Cs_4PbBr_6 crystals emit bright white light with a driven voltage of 3 V. A fluorescence detector was used to detect the emission spectrum. **Figure 5b** show that the light emitting device can achieve full-band emission (400 nm to 800 nm) in the visible range. The green light (wavelength around 520 nm) from Cs_4PbBr_6 micro crystals shows a strong and sharp peak, which is consistent with **Figure 3**. According to the emission spectrum, the colour coordinates are calculated by the software CIE1931xy (V.1.6.0.2), according to the formula I and II:

$$\text{colour gamut CIE1931} = (R_x \cdot G_y + R_y \cdot B_x + G_x \cdot B_y - R_x \cdot B_y - G_x \cdot R_y - B_x \cdot G_y) / 2 \quad \text{I}$$

$$\text{NTSC} = \text{colour gamut} / 0.1582 \quad \text{II}$$

Where R, G and B are red, green and blue, respectively.

The calculated results in **Figure 6** indicate that the prepared light emitting device based on Cs_4PbBr_6 crystal from sample Green-DMSO can cover 32.3% of the colour gamut and achieves 107.3% coverage for International Commission on Illumination (CIE) 1931 and 84.8% coverage for CIE 1976. The light emitting device with Cs_4PbBr_6 micro crystals as green light source can cover 105% of National Television system committee (NTSC) gamut. These values are higher than those of light emitting devices made with the commonly used green phosphors, such as β-SiAlON:Eu^{2+} and CdS nanocrystals with NTSC gamut 86% and 104%, respectively.[25-27] Comparing with these current commercially available phosphors, the starting materials for Cs_4PbBr_6 micro crystals are cheaper and the synthesis is easier. The resulting light emitting device can achieve wide colour gamut coverage. These results reflect the great potential of ultrasonication-assisted trace amount solvent method prepared Cs_4PbBr_6 micro crystals in the manufacture of wide colour gamut light emitting device, while allowing for their simplicity, environmental friendliness and scalable synthesis methods as potential materials for high device performance. This research expands the application of halide perovskite materials beyond solar cells.

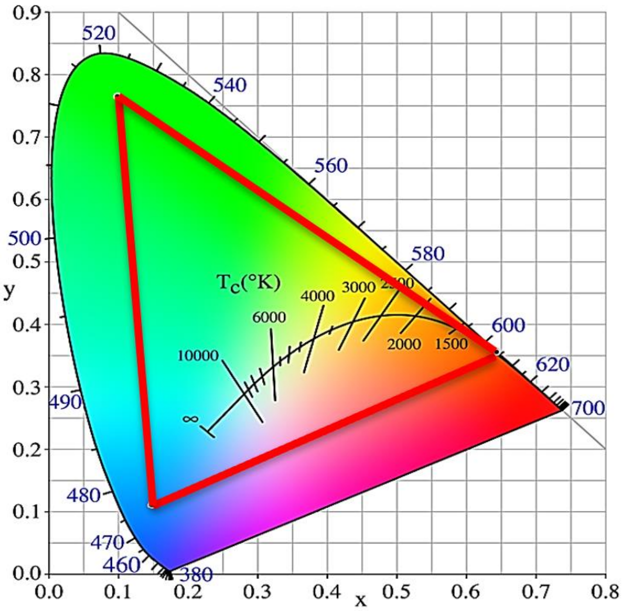

Fig. 6. CIE diagram of the light emitting device.

4. Conclusions

In summary, Cs_4PbBr_6 crystals have been rapidly synthesized by ultrasonication with trace amount of DMSO. It shows a strong fluorescence at 519 nm with lifetime of 10 ns. The corresponding green light emitting device achieve a maximum brightness of 3.8×10^9 cd/m^2. The white light emitting device can cover 32.3% of the color gamut, which covers 107.3% of CIE1931 and 84.8% of CIE1976. It is an excellent green light source material. This method offers a simply, fast and environmentally friendly way to prepare Cs_4PbBr_6 material.

Supplementary Material

See the supplementary material for a detailed description of the wick and video of the device.

Acknowledgments

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Data Statement

Data sharing is not applicable to this article as no new data were created or analysed in this study.

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Supporting information

Ultrasonication-Assisted Trace Amount Solvent Synthesis of Cs_4PbBr_6 Crystal with Ultra-Bright Green Light Emission

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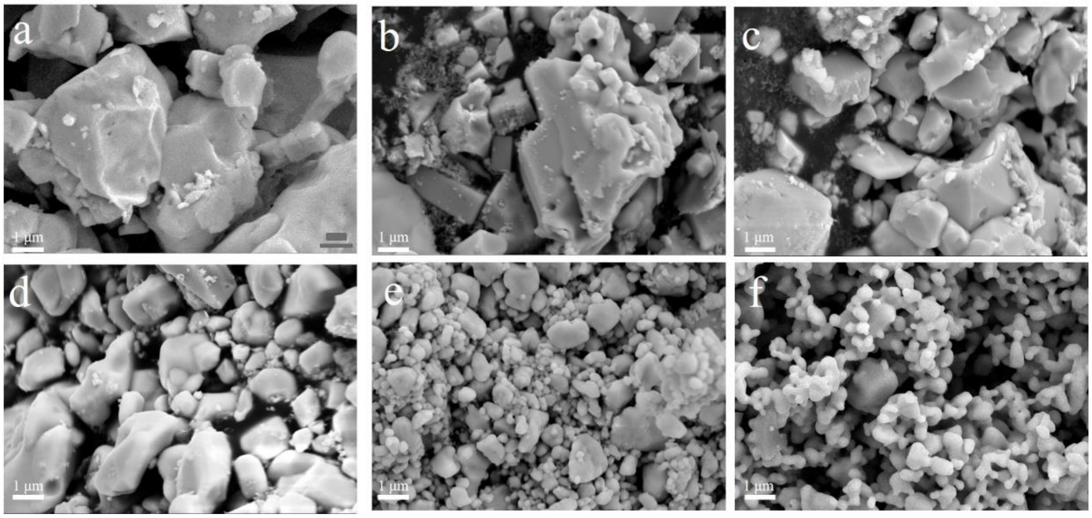


Figure S1. (a) Block $CsPbBr_3$ (b)-(f) ultrasonic preparation of white perovskite in proportion：$CsBr:PbBr_2$=1:1,2:1,3:1,5:1,6:1

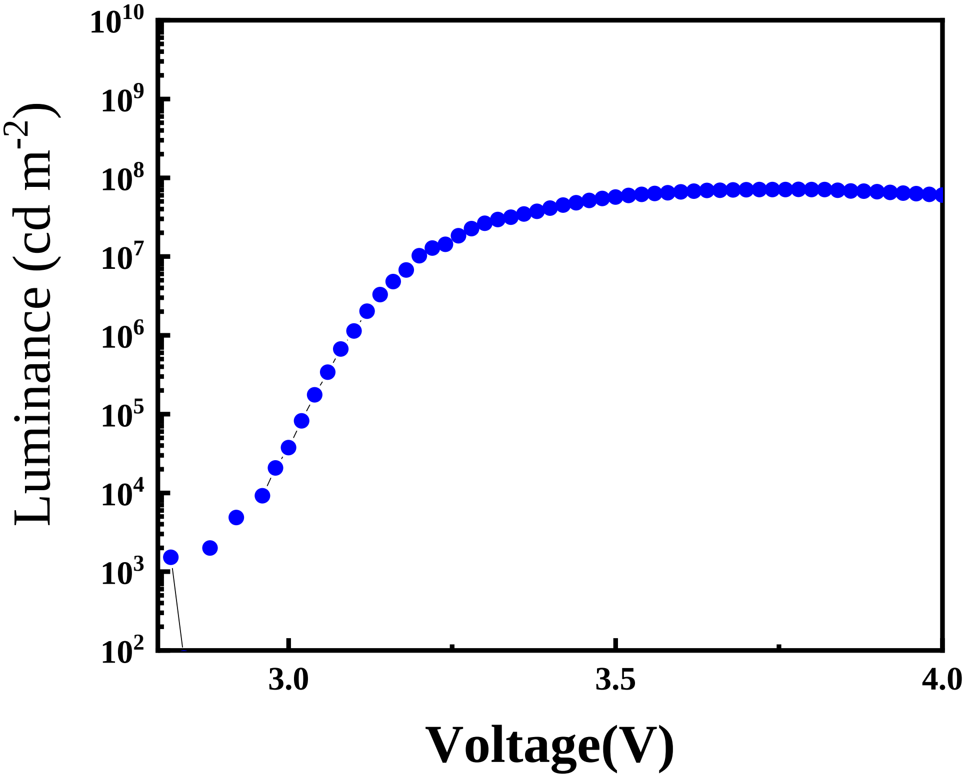


Figure S2. Luminance versus voltage characteristics of the wick.

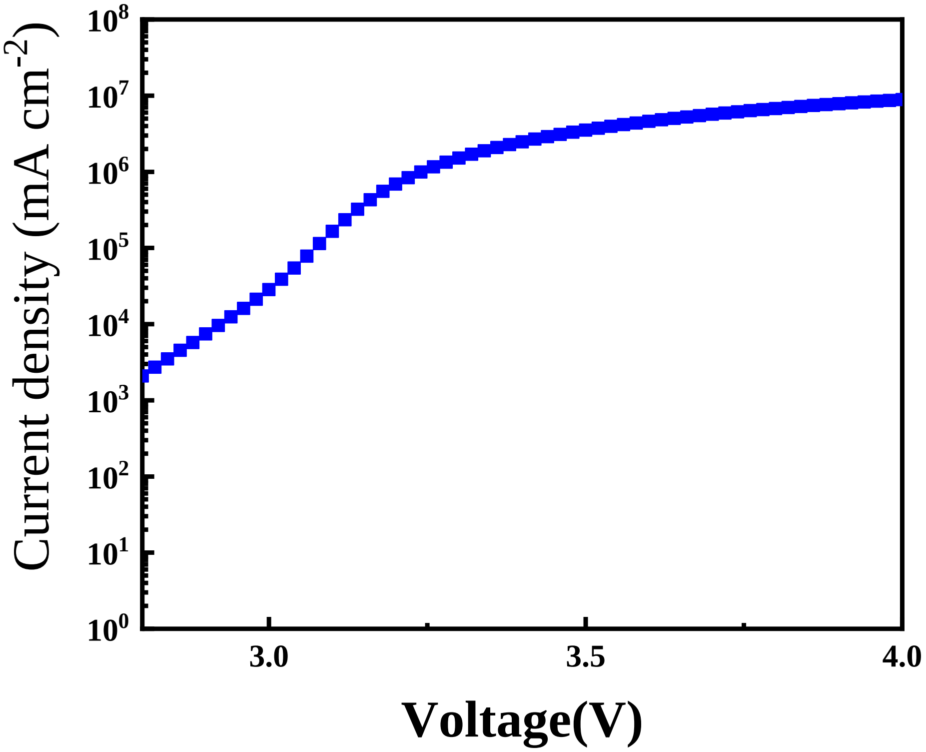


Figure S3. Current density voltage characteristics of the wick

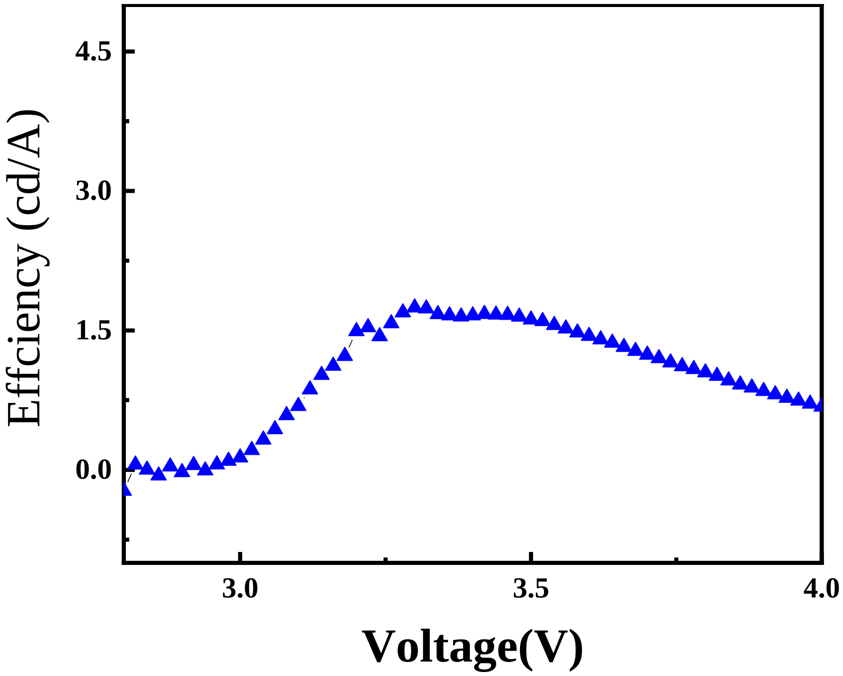


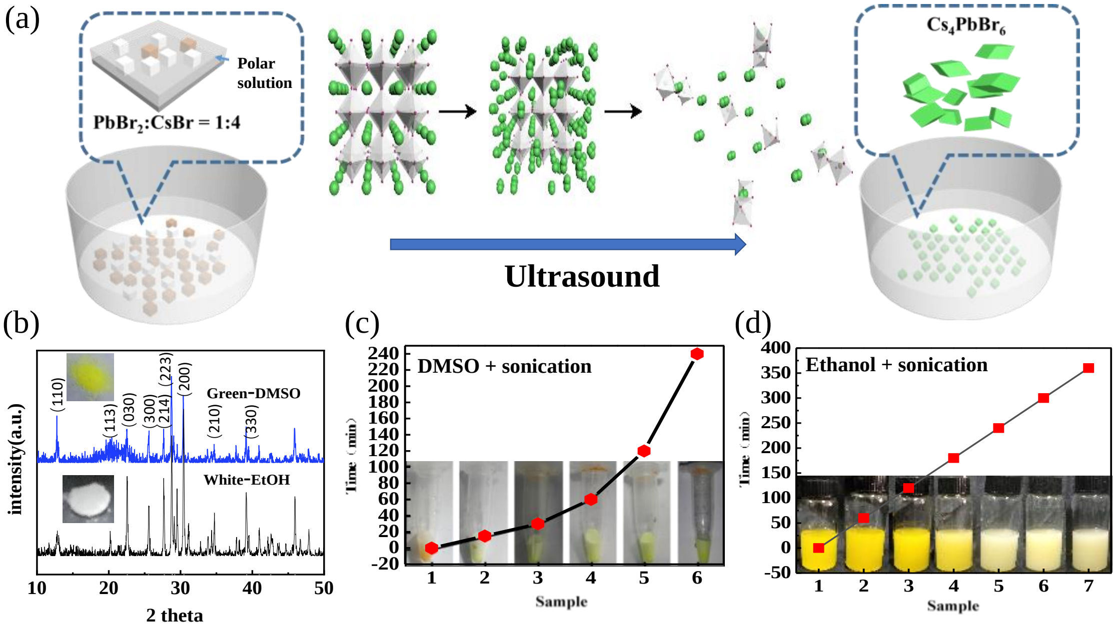
Figure S4. Current efficiency voltage characteristics of the wick.

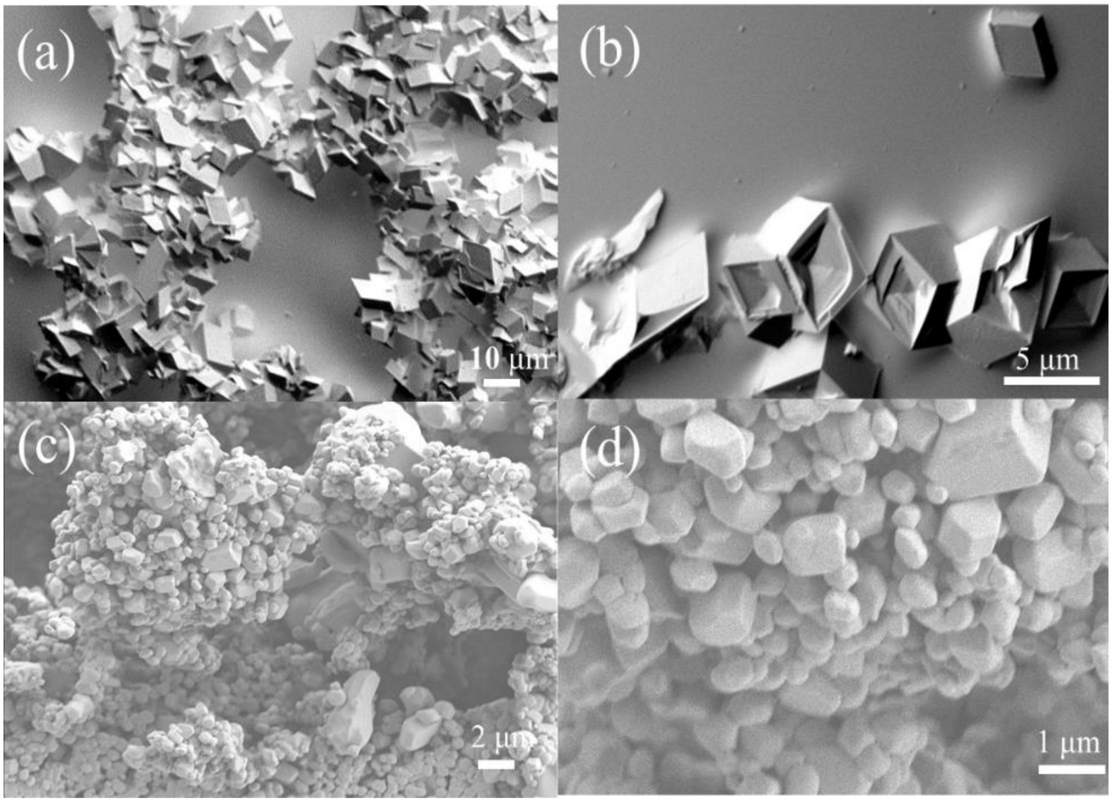
Table S1 Element ratio of supernatant of sample White-EtOH

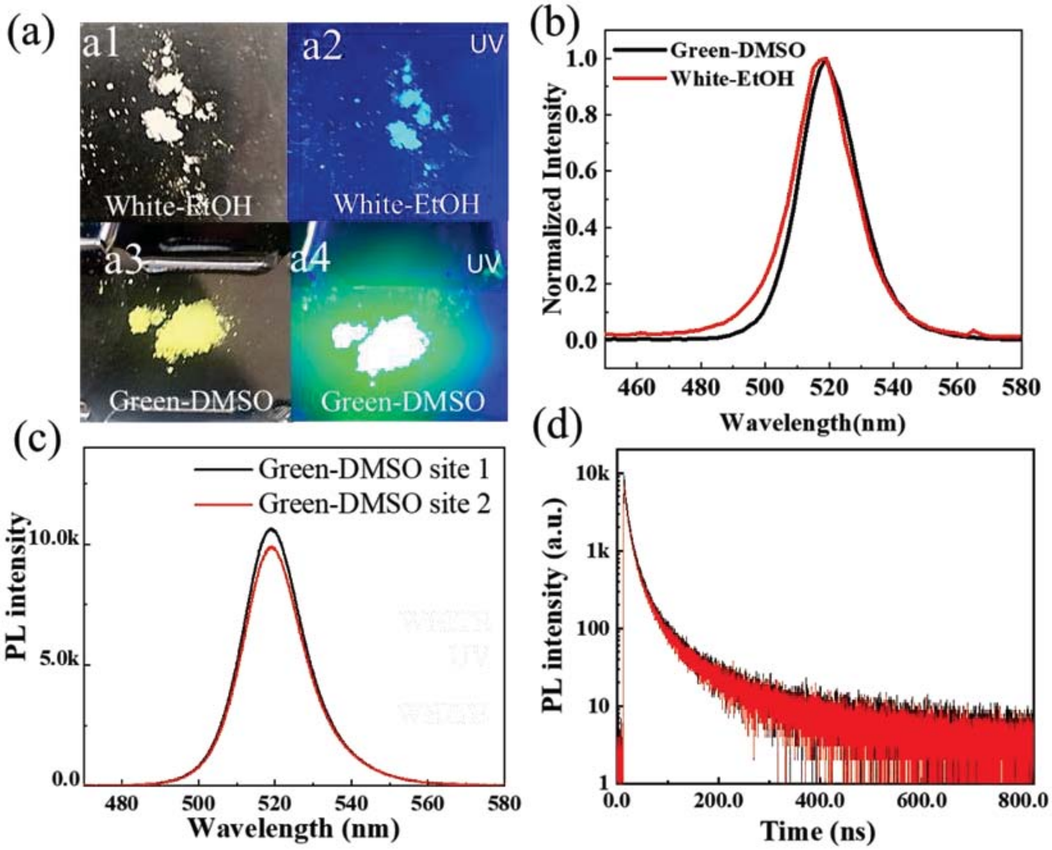
Element	Weight	Atomic percentage
Br	39.42	54.30
Cs	45.55	37.72
Pb	15.03	7.98

Table S2 Element ratio of supernatant of sample Green-EtOH

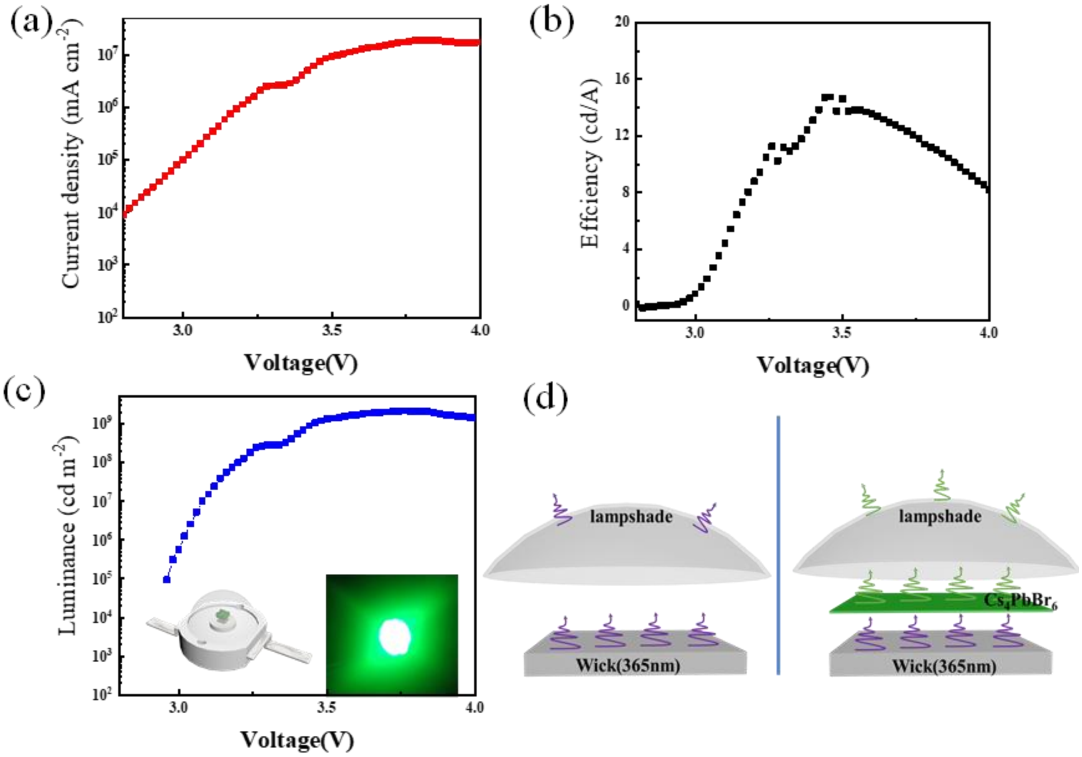
Element	Weight	Atomic percentage
Br	29.22	42.82
Cs	52.39	9.85
Pb	17.43	46.17

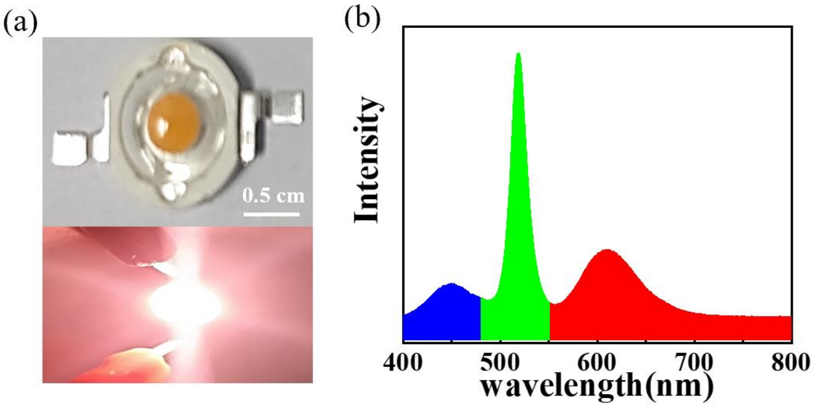

(a)
Polar solution
$PbBr_2$:CsBr = 1:4
Cs_4PbBr_6
Ultrasound
(b)
intensity(a.u.)
(110)
(113)
(030)
(300)
(214)
(223)
(200)
(210)
(330)
Green-DMSO
White-EtOH
10
20
30
40
50
2 theta
(c)
DMSO + sonication
Time（min）
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
1
2
3
4
5
6
Sample
(d)
Ethanol + sonication
Time（min）
400
350
300
250
200
150
100
50
0
-50
1
2
3
4
5
6
7
Sample

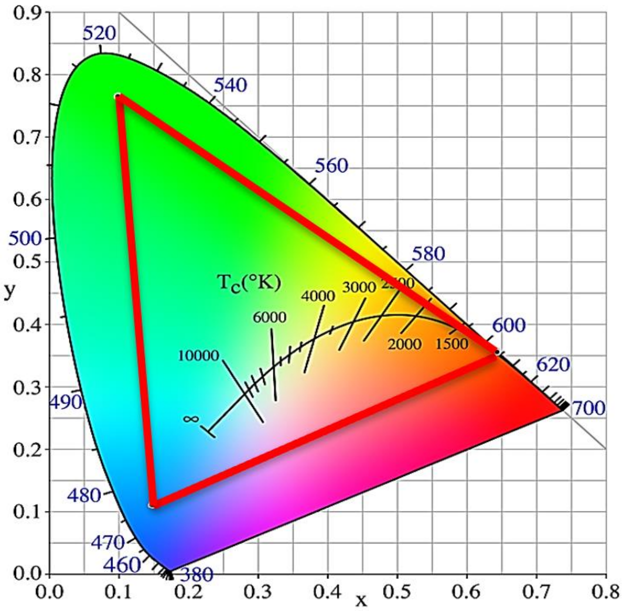

(a)
10 μm
(b)
5 μm
(c)
2 μm
(d)
1 μm



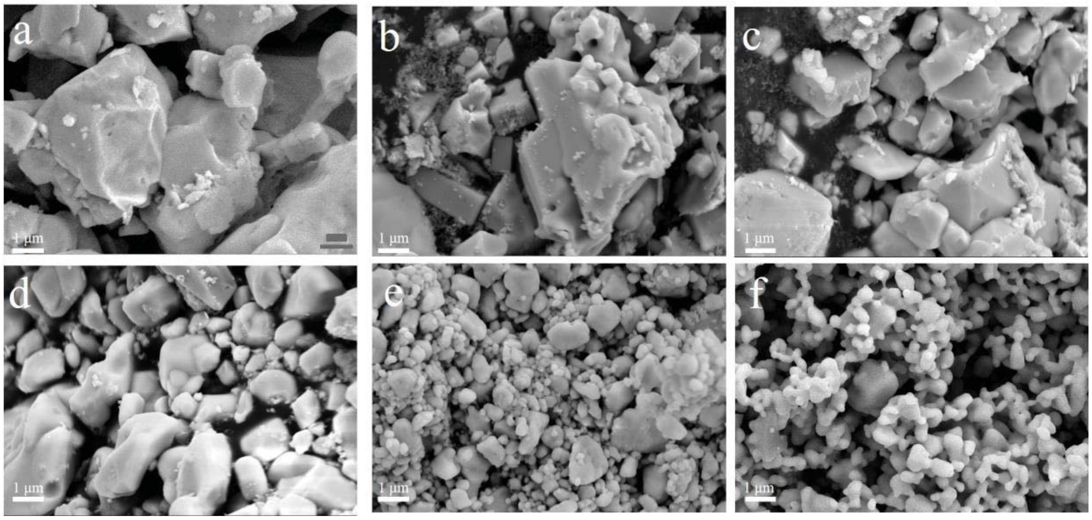
(a)
a1
White-EtOH
a2
UV
White-EtOH
a3
Green-DMSO
a4
UV
Green-DMSO
(b)
Green-DMSO
White-EtOH
Normalized Intensity
1.0
0.8
0.6
0.4
0.2
0.0
460
480
500
520
540
560
580
Wavelength(nm)
(c)
Green-DMSO site 1
Green-DMSO site 2
PL intensity
10.0k
5.0k
0.0
480
500
520
540
560
580
Wavelength (nm)
(d)
PL intensity (a.u.)
10k
1k
100
10
1
0.0
200.0
400.0
600.0
800.0
Time (ns)

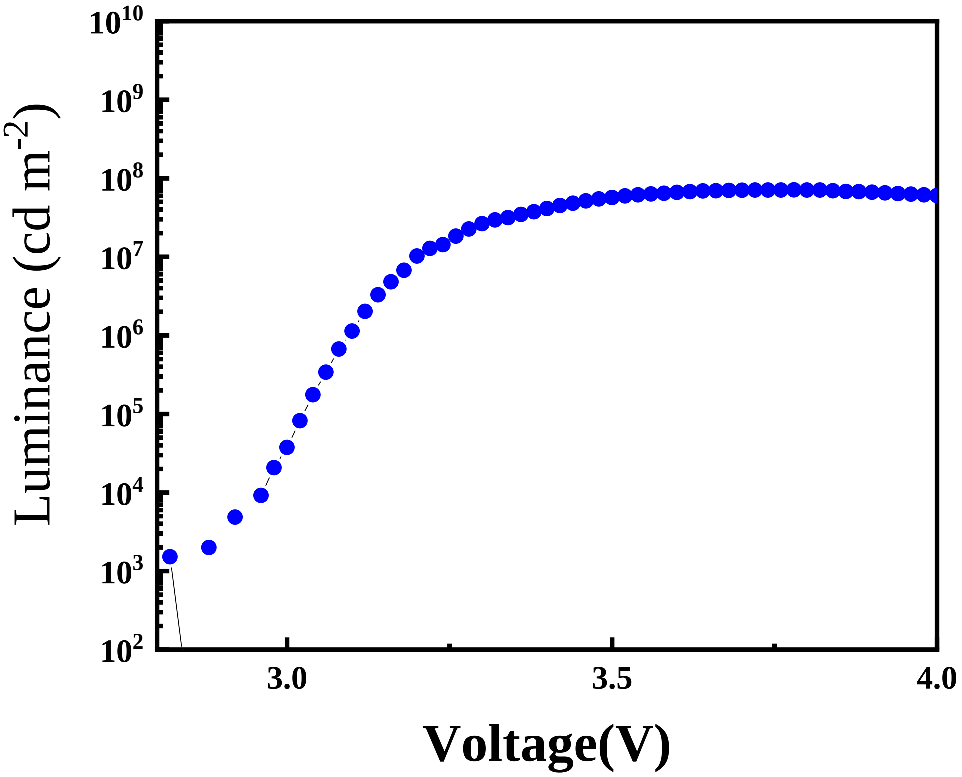

(a)
Current density (mA cm^{-2})
10^2
10^3
10^4
10^5
10^6
10^7
3.0
3.5
4.0
Voltage(V)
(b)
Effciency (cd/A)
0
4
8
12
16
20
3.0
3.5
4.0
Voltage(V)
(c)
Luminance (cd m^{-2})
10^2
10^3
10^4
10^5
10^6
10^7
10^8
10^9
3.0
3.5
4.0
Voltage(V)
(d)
lampshade
Wick(365nm)
lampshade
Cs_4PbBr_6
Wick(365nm)


(a)
0.5 cm
(b)
Intensity
400
500
600
700
800
wavelength(nm)

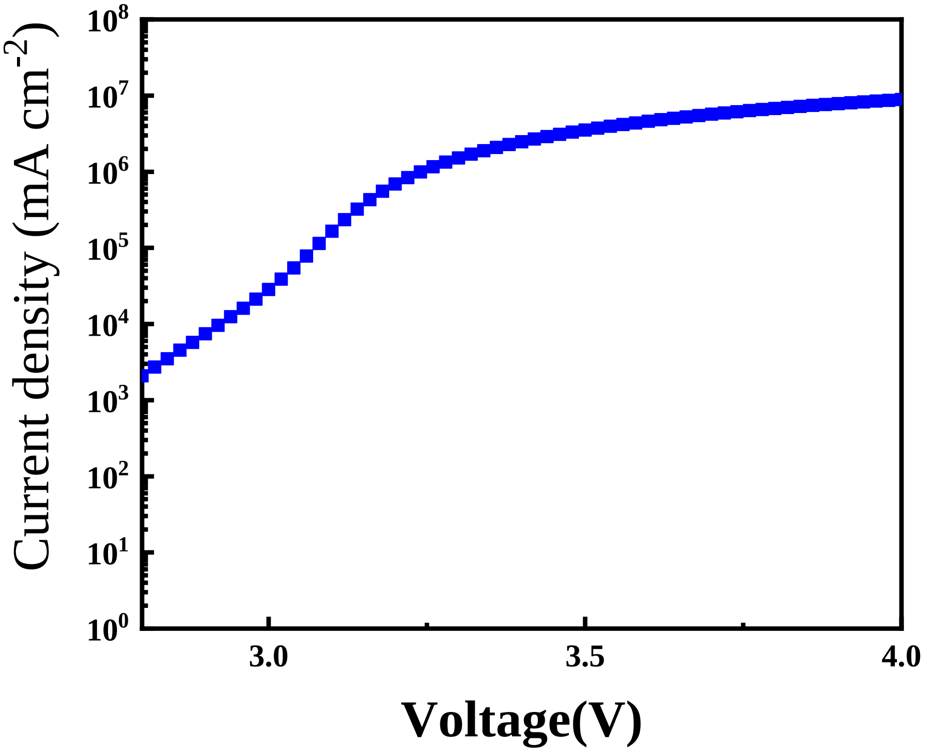


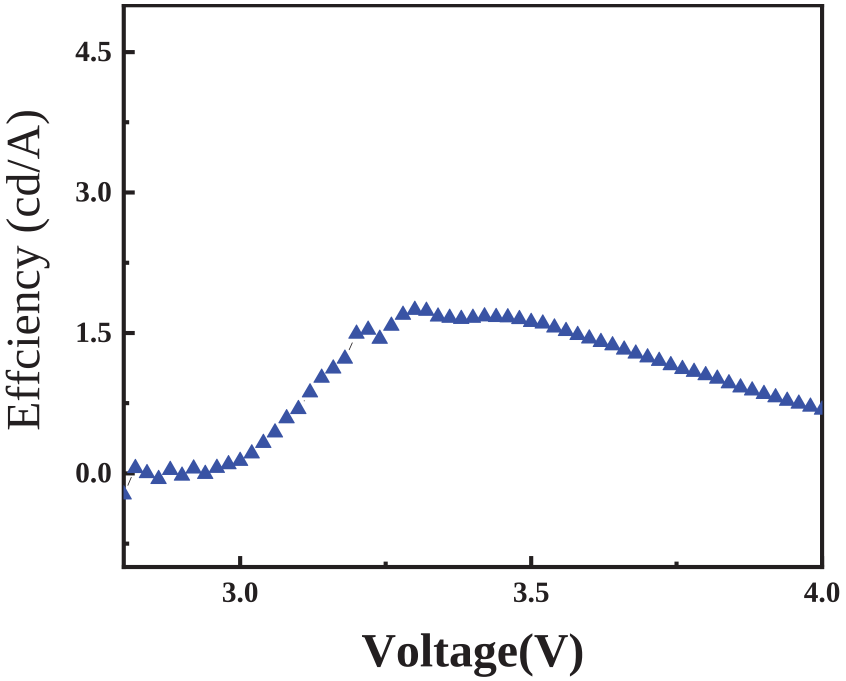
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
y
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
x
520
540
560
580
600
620
700
500
490
480
470
460
380
T_C(°K)
10000
6000
4000
3000
2500
2000
1500
∞


a
1 μm
b
1 μm
c
1 μm
d
1 μm
e
1 μm
f
1 μm



Luminance (cd m^{-2})
10^{10}
10^{9}
10^{8}
10^{7}
10^{6}
10^{5}
10^{4}
10^{3}
10^{2}
3.0
3.5
4.0
Voltage(V)


Current density (mA cm^{-2})
10^8
10^7
10^6
10^5
10^4
10^3
10^2
10^1
10^0
3.0
3.5
4.0
Voltage(V)


Effciency (cd/A)
4.5
3.0
1.5
0.0
3.0
3.5
4.0
Voltage(V)