It remains challenging to fabricate high-efficiency all-inorganic perovskite solar cells (PSCs) at low temperature. Herein, the low-temperature preparation of high-efficiency CsPbI₃ PSCs using hydroiodic acid (HI) and deionized water (H₂O) as additives at synthetic temperature as low as 100 °C is proposed, which is beneficial for constructing even more complicated tandem or flexible solar cells. The additive of HI facilitates CsPbI₃ to form a high-quality and smooth perovskite film at low temperature. The addition of an optimal amount of H₂O further induces perovskite growth with larger grains and a smoother surface by the dissolution and recrystallization process in the grain boundary, which can suppress defects-induced nonradiative recombination. In this scenario, the corresponding device with structure of ITO/PEDOT:PSS/CsPbI₃/PC₆₁BM/Ag exhibits a high power conversion efficiency of 11.41% and the fill factor is increased from 62% to about 69%, compared with device based on CsPbI₃ film without extra H₂O treatment. The appropriate amount of H₂O promotes the high-quality multigrain growth of the CsPbI₃ film and reduces the internal defects, contributing to a significant improved charge collection efficiency. Therefore, this research work has paved a significant way to improving the quality of the all-inorganic perovskite film for highly efficient solar cells.

1. Introduction

In the field of all-inorganic perovskite solar cells (PSCs) with good high temperature stability, CsPbI₃,[1–3] CsPbI₂Br,[4,5] CsPbBr₃,[6] and CsSnI₃[7] are the main representatives of light absorption layer materials. The CsPbBr₃ thin film with a bandgap of 2.3 eV is too large to be suitable for single-layer photovoltaic applications.[8–10] In general, cubic-phase CsPbI₃ with a suitable bandgap (1.73 eV) has become a promising candidate for efficient inorganic photovoltaic materials.[11–14] Ripollès et al.[15] thermally annealed CsPbI₃ films at 350 °C and fabricated solar cells based on the device structure of FTO/bl-TiO₂/mp-TiO₂/CsPbI₃/P₃HT/MoO₃/Au, achieving a maximum power conversion efficiency (PCE) of 4.68%. Cubic-phase CsPbI₃ could be stabilized at 300 °C by adding polymer polyvinylpyrrolidone (PVP) to its precursor solution, achieving 10.74% device efficiency and showing good humidity/thermal stability.[16] The PCE of PSCs based on CsPbI₃ had been greatly improved. Zhao’s group used dimethylammonium iodide (DMAI) additive to control the final crystal phase, crystallinity, and grain size of CsPbI₃. On this basis, phenyl-trimethylammonium chloride (PTACL) passivation was used to improve the device performance, wherein the device exhibited the highest efficiency of 19.03%.[17] However, it is necessary to conduct about 300 °C high-temperature annealing in the glovebox to obtain the black cubic-phase CsPbI₃, which can cause process complexity and high cost. Such a high annealing temperature of perovskite thin films is even incompatible for complicated tandem or flexible solar cells.[18] Therefore, at a relatively low temperature, such as 100 °C, the preparation of high-performance all-inorganic perovskite films could enable a simpler preparation process for PSCs. This also allows for richer application scenarios, such as silicon-perovskite tandem solar cells and flexible PSC.[19,20]

When CsPbX₃ perovskites are prepared at low temperatures (≤150 °C), they usually exhibit a yellow orthorhombic phase (β-phase) and poor crystallinity, which are not suitable for solar cell applications. It is still a challenge to fabricate the high-quality pure α-phase CsPbX₃ perovskite films at low temperatures due to the trade-off between processing temperature and device performance.[21] To circumvent these problems, some favorable strategies were proposed to stabilize cubic-phase CsPbI₃ at room temperature, including reduced dimensions, ionic incorporation, additive engineering, and surface passivation.
Ion doping such as Mn$^{2+}$ or Bi$^{3+}$ has been reported to stabilize α-CsPbI$_3$. Zhang and co-workers added Bi$^{3+}$ into CsPbI$_3$ to partly replace Pb$^{2+}$ to improve the phase stability at a low temperature and achieved a PCE of 13.21%. Hydroiodic acid (HI) as an additive provides new ideas for the low-temperature preparation of high-quality CsPbI$_3$ perovskite thin films. Interestingly, under the premise of adding an appropriate amount of HI, the black cubic-phase CsPbI$_3$ can be obtained at thermal annealing at a low temperature of 100°C, and a PCE of only 1.7% for the device structure of ITO/PEDOT:PSS/CsPbI$_3$/PCBM/Ca/Al was achieved. Hu et al. made use of HI to stabilize cubic-phase CsPbI$_3$ and solar cells based on the traditional structure (FTO/c-TiO$_2$/CsPbI$_3$/C$_{14}$-CsPbI$_3$) and achieved a PCE of 8.07%. Nevertheless, the inverted solar cells prepared using HI as the precursor additive still have large room for efficiency improvement, and the effect of the components of HI on PCE still has to be further clarified. As an additive, HI is dispersed in H$_2$O, and H$_2$O also has an important effect on the growth of CsPbI$_3$ perovskite crystals. Also, H$_2$O was known to be an important factor in PSC technology for controlled crystallinity. Therefore, it is critical to investigate the effects on all-inorganic CsPbI$_3$ crystal thin films and device performances.

In this article, high-quality CsPbI$_3$ thin films are prepared by the one-step spin-coating method with HI and H$_2$O as additive and N,N-dimethylformamide (DMF) as the solvent. To achieve a low-temperature process, we prepared devices with an inverted device structure of ITO/PEDOT:PSS/CsPbI$_3$/PC$_{60}$BM/Ag, avoiding the use of inorganic electron transport layers that require high-temperature processing. The amount of HI added and the effect of water on the film and device characteristics were further analyzed by various physical characterization methods. The results show that the optimal HI addition amount is 35 μL per mL of perovskite precursor solution. Furthermore, the perovskite films with micro-H$_2$O have larger grain size and a more uniform and smoother surface, which are likely attributed to the fact that water breaks down the structure of ionic perovskites into PbI$_2$ and CsI, which repairs the interface of grains by dissolution and recrystallization after H$_2$O removal to form a continuous smooth film. Moreover, the DMF + H$_2$O perovskite precursor solution has good adhesion on the hydrophilic PEDOT:PSS film and disperses evenly on the surface to improve the surface coverage of the perovskite film after spinning by matching the interfacial energy. The formation of high-quality pinhole-free films greatly inhibits the generation of defects and extends the lifetime of carriers. PSCs showed that the addition of H$_2$O effectively enhanced the fill factor (FF) and further increased the device efficiency from 10.34% to 11.42%. Therefore, the appropriate amount of HI and H$_2$O can have a synergetic effect on the performance improvement of the device.

2. Results and Discussion

The crystal particle size and uniformity of the perovskites are important references for evaluating their morphology. In this work, the morphology of CsPbI$_3$ thin films, deposited by the low-temperature solution process with different contents of HI and H$_2$O as additives, was conducted by a scanning electron microscope (SEM). Figure 1a-f shows the SEM images of CsPbI$_3$ thin films after thermal annealing at 100°C with different HI and H$_2$O contents. The perovskite CsPbI$_3$ film with 25 μL HI as additive (25 μL HI in 1 mL perovskite precursor solution) (Figure 1a) showed a more narrow grain size and it could be clearly observed that many needle-like pores were distributed on the surface of the film. As the amount of HI increases to 35 μL (Figure 1b), the perovskite crystal has a larger average grain size, fewer pinholes, and a more uniform surface, which means that the crystal growth process is further improved. Furthermore, with the addition of HI that increased to 45 μL (Figure 1c), fewer pinholes could be observed on the surface of film, while the crystal grains size was significantly reduced. As HI is dispersed in H$_2$O, then the extra H$_2$O was added into perovskite precursor to investigate its role in photovoltaic performance. Figure 1d-f

Figure 1. Top-view SEM images of the CsPbI$_3$ thin films with a) 25 μL HI, b) 35 μL HI, c) 45 μL HI, d) 35 μL HI + 5 μL H$_2$O, e) 35 μL HI + 10 μL H$_2$O, and f) 35 μL HI + 20 μL H$_2$O.
shows the effect of H₂O on the morphology of the perovskite thin film; it is clear that the thin film with 35 μL HI and 10 μL H₂O is uniform, and the grain size is significantly larger than that prepared under other conditions. When the amount of H₂O increased to 20 μL, the perovskite grain size decreased significantly.

It was reported that the roughness of the thin films has great influence on the performance of the PSCs; therefore atomic force microscopy (AFM) was conducted to explore the morphology and surface roughness of perovskite thin films with different additives. As shown in Figure 2, when the amount of HI added is 25 μL (Figure 2a), the surface roughness of the CsPbI₃ thin film is 6.89 nm. While the amount of HI is increased to 35 μL (Figure 2b), the roughness of CsPbI₃ thin film was reduced to 3.61 nm, and when it further increased to 45 μL, the roughness was 4.30 nm (Figure 2c). Interestingly, by adding 5 and 10 μL of H₂O into perovskites based on 35 μL of HI (Figure 2d,e), the roughness of the thin film become 4.17 and 3.19 nm, respectively. Further increasing the amount of H₂O leads into lower surface roughness, but the grain size of the film becomes smaller, which is consistent with the SEM results, as shown in Figure 1.

The devices with a structure of glass/ITO/PEDOT:PSS/CsPbI₃/PC₆_{11}BM/Ag (Figure 3a) were fabricated and characterized, and the energy-level alignment for thin films is shown in Figure 3b. The matched energy-level alignment assures that charge carriers are transported to the corresponding electrode. Figure 3c shows the J–V characteristics of PSCs with different contents of HI as additives under the reverse-scan direction. As the amount of HI increases from 25 to 35 μL, Jₑₑₑ increases from 13.76 to 16.97 mA cm⁻², Vₒₒₒ increases from 0.90 to 0.98 V, FF increases from 50.01% to 62.13%, and PCE increases from 6.11% to 10.34%. However, with more HI content, the PCE of device decreases from 10.34% to 9.09%. Specifically, PSCs with 45 μL exhibit reduced Jₑₑₑ, Vₒₒₒ, and FF of 15.21 mA cm⁻², 0.96 V, and 60.07%, respectively. To investigate this change, 35 μL HI + 10 μL H₂O as additive was added into the perovskite precursor for direct comparison with that of device with 45 μL HI. Surprisingly, in this case, the device exhibits the best PCE of 11.42% and a fill factor of 68.84%. Compared with the efficiency of the all-inorganic PSCs prepared by the low-temperature method recently published, the all-low-temperature-processed p-i-n-structured devices in this work achieved high efficiency performance (Table S1, Supporting Information). Furthermore, when we increase the amount of water to 35 μL HI + 20 μL H₂O as additives, the performance of the device is greatly affected and exhibits reduced Jₑₑₑ, Vₒₒₒ, FF, and PCE of 16.62 mA cm⁻², 0.95 V, 59.79%, and 9.41%, respectively (Figure S1, Supporting Information). Here, adding an appropriate amount of H₂O to the CsPbI₃ precursor solution can effectively improve the FF of PSCs.

Figure 3d shows the external quantum efficiency (EQE) of PSCs fabricated with 35 μL HI and 35 μL HI + 10 μL H₂O. It is observed that PSCs fabricated with 35 μL HI + 10 μL H₂O show an enhanced response in the wavelength ranging from 380 to 680 nm compared with that of PSCs fabricated with 35 μL HI, and the integrated current densities of devices fabricated with 35 μL HI and 35 μL HI + 10 μL H₂O as additives are 15.45 and 16.03 mA cm⁻², respectively. These values are in good agreement with the J–V results of the corresponding devices. The dark current density (J)–voltage (V) characteristic of the devices with different additives is shown in Figure 3e. By comparison, the device with excess 10 μL H₂O exhibits improved diode characteristics, which implies that the device will possess higher voltage. Figure 3f presents the steady photocurrents and PCEs of PSCs at the maximum power output. It is clear that, in the time range of 1000 s, both PSCs show stabilized photocurrents and PCEs, which agree with those measured from J–V scanning, confirming the accuracy of our

Figure 2. AFM height images of CsPbI₃ thin films with a) 25 μL HI, b) 35 μL HI, c) 45 μL HI, d) 35 μL HI + 5 μL H₂O, e) 35 μL HI + 10 μL H₂O, and f) 35 μL HI + 20 μL H₂O. The scanning range is 5 × 5 μm.
efficiency measurement. In addition to the enhanced PCE, the introduction of H₂O in the perovskite precursor also improves the device stability. The result is shown in Figure S1, Supporting Information, after being stored in the glovebox for 144 h, and the efficiency of the 35 HI + 10 μL H₂O PSCs is 82% of the initial PCE; in contrast, in the same period, the 35 HI PSCs exhibit a fast degradation with 68% of initial PCE. In Figure 4 and Table S1, Supporting Information, the J_{sc}, V_{oc}, FF, and PCE results are shown for 20 devices. Both PSCs exhibit a narrow distribution of PCEs, demonstrating the reliability and repeatability of our devices, especially as the addition of trace water improves the repeatability of the device. In addition, the passivation on the CsPbI₃ crystal grain boundary effect of H₂O makes the device show better storage stability. The device with extra H₂O maintains 85% of its original performance, which is much better than that of the PSCs with 35 μL HI (67%) (Figure S2, Supporting Information).

The X-ray diffraction (XRD) pattern of perovskite thin films obtained using HI as the additive is shown in Figure 5a. The result shows several obvious diffraction peaks at 2θ of 14.37°, 20.27°, 28.93°, 35.3°, and 41.09°, corresponding to the crystal planes of (100), (110), (200), (211), and (202) respectively, which are typical signals of the α-phase crystal structure. The black star in Figure 5a represents the yellow orthorhombic phase, which could be caused by the phase transition of the thin film in contact with the air during the test. Obviously, when the

Figure 3. a) Schematic diagram and b) energy-level diagram of CsPbI₃-based PSCs. c) J–V curves of the PSCs with 25 μL HI, 35 μL HI, 45 μL HI, and 35 μL HI + 10 μL H₂O as additives. d) EQE and integrated J_{sc} for the 35 μL HI with and without 10 μL H₂O. e) J–V curves of the PSCs in the dark. f) Steady-state output photocurrent at maximum power output point.
addition of HI increases from 25 to 35 and 45 μL, the intensity of the diffraction peaks first increases and then decreases. When the amount of HI is 35 μL HI + 10 μL H₂O, the diffraction peak of the perovskite has the maximum intensity value, which implies that the corresponding perovskite thin film has the best crystallinity and the largest grain size, as shown in Figure 1d and 2d. Furthermore, the results in Supporting Information, Figure 5, prove that H₂O treatment on the CsPbI₃ perovskite film could decompose it into PbI₂, decomposing the structure of the ionic perovskite into PbI₂ and CsI. Similarly, in the CsPbI₃ phase formation process, trace water will also cause the slight dissolution of the perovskite at the grain boundary to achieve passivation and recrystallization, and the corresponding perovskite thin film has the best crystallinity and the largest grain size. It can be observed that the addition of a small amount of water significantly improves the intensity of the diffraction peaks and the crystallinity of the thin film, which is beneficial for enhanced charge collection and better device performance.

To investigate the optical properties of CsPbI₃ thin films, UV–vis absorption (UV–vis) and photoluminescence (PL) spectra were conducted in Figure 5b. It was observed that all thin films showed the absorption band-edge positions of about 720 nm and the prepared film possesses optimal light absorption capacity when adding 35 μL HI + 10 μL H₂O. With the increase in HI content, the optical bandgaps of perovskite films decreased, which could be attributed to grain pulverization and quality degradation (Figure 2c), caused by excessive HI. PL can intuitively study the surface defect states of perovskite films.

Figure 4. Photovoltaic parameters of the devices containing 35 μL HI and 35 μL HI + 10 μL H₂O. Plot of the a) PCE, b) FF, c) J_sc, and d) V_OC.

Figure 5. a) XRD patterns, b) UV–vis absorption, and c) PL spectra of CsPbI₃ thin films with 25 μL HI, 35 μL HI, 45 μL HI, and 35 μL HI + 10 μL H₂O. d) TRPL spectra of the thin films deposited on glass substrate.
Huang and co-workers\textsuperscript{[41]} reported that passivation of defects will cause a blueshift of the PL peak. The PL spectra results in Figure 5c showed that the thermal-annealed films have obvious PL after adding appropriate amounts of HI and water. When adding 25, 35, 45, and 35 ± 10 μL H2O, the peak positions of the PL spectra are 706, 701, 701, and 697 nm, respectively. This means that different amounts of HI or water may cause the blueshift of the emission peak of the perovskite film, which also confirms the defect suppression process of small amounts of water.\textsuperscript{[42,43]}

To further understand the influence of the addition of trace water on the recombination and lifetime of charge carriers inside the thin films, time-resolved PL (TRPL) spectroscopy was operated for the films with 35 μL HI and 35 μL HI + 10 mL H2O as additives. Results shown in Figure 5d and Table 1 present the PL decay times (τ) and amplitudes (A). The average recombination lifetime (τ\textsubscript{ave}) was fitted and estimated by a biexponential decay function based on Table 2 and Equation (1)\textsuperscript{[44]} as follows.

\[
\tau_{\text{ave}} = \frac{\sum A_i \tau_i^2}{\sum A_i \tau_i}
\]  

(1)

The results show that the addition of trace water increases the carrier lifetime of the CsPbI\textsubscript{3} film from 4.9 to 6.9 ns. This implies that the nonradiative recombination inside the film is suppressed to a certain extent, crystal defects are reduced, and carriers will be better extracted, and therefore, better performance for devices based on this thin film is expected.\textsuperscript{[45,46]}

The influences of H2O addition on trap state density and charge mobility in CsPbI\textsubscript{3} perovskite films were further investigated. The dark current–voltage characteristics for the hole-only device with a structure of ITO/PEDOT:PSS/CsPbI\textsubscript{3}/MoO\textsubscript{3}/Ag was prepared and conducted to analyze the density of defect states. The results are shown in Supporting Information, Figure 3. The curves exhibit a linear ohmic response at low bias, a trap-filled limit regime, and a trap-free space–charge-limited current (SCLC) regime. Therefore, τ\textsubscript{c} is calculated by Equation (1). The hole trap density was decreased from 2.07 × 10\textsuperscript{16} to 1.37 × 10\textsuperscript{16} cm\textsuperscript{-3} with the addition of trace water, which is favorable for reducing charge carrier recombination. A smaller J–V hysteresis also could be achieved with extra 10 μL H2O as additive under forward- and reverse-scan direction (Figure S4, Supporting Information, ), indicating a reduction of defects in the CsPbI\textsubscript{3} perovskite thin film.

It is evident that HI and H2O can copromote the growth of perovskite thin films with large grain size and good properties. When appropriate HI as an additive, the black-phase CsPbI\textsubscript{3} perovskite crystal was obtained at a low temperature of 100 °C, and the corresponding device acquires PCE of 10.66%. Furthermore, the addition of H2O promotes the growth of grains and reduces the surface roughness, which suppressed the defect in CsPbI\textsubscript{3} polycrystalline films. In the process of CsPbI\textsubscript{3} perovskite crystallization, the addition of the optimal amount of H2O may lead to a dynamic process of dissolution and recrystallization at the grain boundaries. It provides a smooth film and an extended carrier lifetime. Reduced defects density and efficient charge carrier transport/extraction help device achieve higher fill factor and a PCE of 11.42%.

3. Conclusion

In summary, all-inorganic CsPbI\textsubscript{3} PSCs we fabricated showed high device performance under the combination of HI and trace water. The addition of HI enables a high-quality uniform CsPbI\textsubscript{3} film at thermal-annealed temperature as low as 100 °C. After optimizing the amount of HI, the device achieved better device performance when 35 μL HI was used as additive. In addition, the excess 10 μL H2O in perovskite precursor solution further increases the efficiency of the device to 11.41% and obviously suppresses the nonradiative recombination inside the film by a dissolution and recrystallization process that occurs at the grain boundaries. The fill factor of the device was increased from 62.13% to 68.84%, resulting from the improved CsPbI\textsubscript{3} crystallinity and grain size. This strategy of fabricating high-efficiency all-inorganic PSCs under low-temperature conditions is a significant exploration for promoting all-inorganic PSCs.

4. Experimental Section

Materials: PEDOT: PSS (Heraeus), 461 mg PbI\textsubscript{2}, and 260 mg CsI (alfa) were dissolved in 1 mL DMF at room temperature. Then, the quick mixer (KMSI) was used to rapidly mix the solution for 5 min and filtered with a 0.22 μm nylon filter to obtain a transparent yellow solution. Different amounts (25, 35, 45, and 55 μL) of HI were respectively added to the previously prepared 1 mL solutions and quickly mixed for 2 min to obtain the perovskite precursor solutions. 20 mg mL\textsuperscript{-1} PC\textsubscript{61}BM were prepared by dissolving in chlorobenzene and stirred overnight at 60 °C.

Preparation of Perovskite Films and Solar Cells: The ITO/glass (1.5 × 1.5 cm) underwent a preparation process of cleaning with ethanol, acetone, and deionized water and stored in a drying oven for use. PEDOT: PSS layer was spin coated at the speed of 3000 rpm for 30 s onto ITO sphere. Then, the as-prepared perovskite precursor solutions were spin dissolved in chlorobenzene and stirred overnight at 60 °C.

Table 1. Parameters of the TRPL spectra of perovskite films with different volumes of HI and H2O.

| Device    | τ\textsubscript{ave} [ns] | τ [ns] | A |
|-----------|--------------------------|-------|---|
| 35 μL     | 4.9                      | 4.9   | 1 |
| 35 + 10 μL| 6.9                      | 6.9   | 1 |

Table 2. Performance parameters of various volume doping with HI and H2O as additives.

| PSCs    | J\textsubscript{SC} [mA cm\textsuperscript{-2}] | V\textsubscript{OC} [V] | FF [%] | PCE [%] |
|---------|---------------------------------|----------------|--------|--------|
| 25 μL   | 13.76                           | 0.90            | 50.01  | 6.11   |
| 35 μL   | 16.97                           | 0.98            | 62.13  | 10.34  |
| 45 μL   | 15.21                           | 0.96            | 60.07  | 9.09   |
| 35 + 5 μL| 16.13                         | 0.98            | 68.83  | 10.58  |
| 35 + 10 μL | 16.81                        | 0.99            | 68.84  | 11.42  |
| 35 + 20 μL | 16.62                        | 0.95            | 59.79  | 9.41   |
were transferred into a vacuum chamber for the deposition of Ag (100 nm) electrode under a base pressure of 4.0 × 10⁻⁶ Pa.

Characterization of Perovskite Thin Films: SEM images were obtained by using a field-emission SEM (JEOL-7401). Diffraction of X-rays was measured by D8 Advance, Bruker, Germany, with a scan range of 10°–50° (10° min⁻¹). UV–vis absorption of thin films was recorded on HP 8453 spectrophotometer. The PL spectra of the perovskite films were tested by a fluorescence spectrophotometer with a standard light source (FLS920 Spectro fluorimeter, Edinburgh Instruments). AFM-based measurement was performed on Bruker Dimension Icon.

Characterization of PSCs: The current–voltage measurements of prepared devices were conducted in the dark and under white light and monochromatic light at 500 nm from a xenon lamp by a Keithley model 2400. The light intensity was AM 1.5 G white light, which was calibrated with a silicon reference cell. The spectral response was taken by an EQE measurement system, which was equipped with a monochromator, a lock-in amplifier, Xe lamp, and a current–voltage amplifier.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords
CsPbI₃ perovskite solar cells, hydroiodic acid, low-temperature preparation, water
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