Bication lead iodide 2D perovskite component to stabilize inorganic α-CsPbI₃ perovskite phase for high-efficiency solar cells

Taiyang Zhang,¹ M. Ibrahim Dar,² Ge Li,¹ Feng Xu,¹ Nanjie Guo,¹ Michael Grätzel,² Yixin Zhao¹*

Among various all-inorganic halide perovskites exhibiting better stability than organic-inorganic halide perovskites, α-CsPbI₃ with the most suitable band gap for tandem solar cell application faces an issue of phase instability under ambient conditions. We discovered that a small amount of two-dimensional (2D) EDAPbI₄ perovskite containing the ethylenediamine (EDA) cation stabilizes the α-CsPbI₃ to avoid the undesirable formation of the nonperovskite δ phase. Moreover, not only the 2D perovskite of EDAPbI₄ facilitate the formation of α-CsPbI₃ perovskite films exhibiting high phase stability at room temperature for months and at 100°C for >150 hours but also the α-CsPbI₃ perovskite solar cells (PSCs) display highly reproducible efficiency of 11.8%, a record for all-inorganic lead halide PSCs. Therefore, using the bication EDA presents a novel and promising strategy to design all-inorganic lead halide PSCs with high performance and reliability.

INTRODUCTION

In past years, the organic-inorganic hybrid lead halide perovskite solar cells (PSCs) have progressed in an impressive manner approaching commercialization (1–5). However, the instability of organic-inorganic hybrid perovskite, such as CH₃NH₃PbI₃ under thermal stress, might stem from the volatility of the organic cation and has become a challenge for long-term practical deployment. Although organic-inorganic mixed-cation PSCs demonstrating improved efficiency and stabilities have been documented (6–8), all-inorganic lead halide perovskite absorbers are much desired, specifically because the issues related to the release or decomposition of the organic component can be avoided. The most suitable all-inorganic structure could be based on CsPbX₃ perovskite because only the Cs is large enough to occupy the A sites with a resulting CsPbI₃ perovskite crystal units, rendering them less prone to unwanted phase transition to the δ structure. Stable α-CsPbI₃ films have been deposited by introducing a small amount of bication 2D perovskite of EDAPbI₄ into the CsPbI₃ precursor solution. This cross-links the α-CsPbI₃ films without impairing the charge-carrier transport. The resulting α-CsPbI₃ structures are highly robust at room temperature for months and can retain their phase even after annealing at 100°C for a week. In addition to the high stability, PSCs based on the α-CsPbI₃ films showed highly reproducible photoconversion efficiency (PCE) of 11.8%, a record for α-CsPbI₃ devices.

RESULTS

We used a typical one-step method to deposit CsPbI₃ films using a regular precursor containing stoichiometric CsI and PbI₂ dissolved in N,N’-dimethylformamide (DMF). A rough yellowish film was obtained after annealing the precursor film at 150°C. As shown in Fig. 1 (A and B), the absorbance peak located at 414 nm and a strong x-ray diffraction (XRD) peak at 9.78° suggest the formation of the unwanted nonperovskite δ-CsPbI₃ phase. However, a 2D segment of these structures may impair the electron transport across the device, lowering their efficiency (18). Therefore, it becomes imperative to judiciously design the bication 2D architecture.

Here, we report a new and facile one-step deposition method to obtain highly efficient and stable α-CsPbI₃ PSCs (2). We stabilize the α-CsPbI₃ perovskite films by introducing ethylenediamine cations (EDA²⁺) whose terminal NH₃⁺ groups are expected to cross-link the CsPbI₃ perovskite crystal units, rendering them less prone to unwanted phase transition to the δ structure. Stable α-CsPbI₃ films have been deposited by introducing a small amount of bication 2D perovskite of EDAPbI₄ into the CsPbI₃ precursor solution. This cross-links the α-CsPbI₃ films without impairing the charge-carrier transport. The resulting α-CsPbI₃ structures are highly robust at room temperature for months and can retain their phase even after annealing at 100°C for a week. In addition to the high stability, PSCs based on the α-CsPbI₃ films showed highly reproducible photoconversion efficiency (PCE) of 11.8%, a record for α-CsPbI₃ devices.

PEROVSKITES

A cation along with formamidinium in high-efficiency PSCs, but at concentrations above 20%, it leads to the formation of non-perovskite δ-CsPbI₃ (7–12). Previous reports have suggested that the phase stability of α-CsPbI₃ strongly depends on the crystallite size because stability improves by trimming the dimensions of the α-CsPbI₃ grains toward the nanoregime (13, 14). Size-dependent phase or thermal stability has been reported in nanomaterials, especially when surfactants were used as growth-controlling agents (15, 16). However, conventional deposition strategies, which do not involve surfactants, mostly yield large-sized CsPbI₃ crystallites. Recently, α-CsPbI₃ quantum dots with well-controlled size synthesized by hot injection have been fashioned into stable PSCs exhibiting an efficiency of up to 10% (17). In addition, a two-dimensional (2D) interface has been demonstrated as an effective strategy to stabilize the organic-inorganic hybrid perovskite or to increase the thermal stability of CH₃NH₃PbI₃ (18, 19). However, a 2D segment of these structures may impair the electron transport across the device, lowering their efficiency (18). Therefore, it becomes imperative to judiciously design the bication 2D architecture.

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indicating a band gap of 1.73 eV, consistent with the previous reports on α-CsPbI3 (13, 22). The XRD pattern (Fig. 1B) obtained from the brown CsPbI3 film could be indexed to a phase-pure cubic α-CsPbI3 perovskite structure (12, 13). However, the PSC based on this α-CsPbI3 film revealed a modest PCE of 5.59% (Fig. 1C), which is comparable to previous reports (10, 13, 23). Unfortunately, this phase-pure α-CsPbI3 film also suffers from phase instability issues. As shown in Fig. 1D, the brown α-CsPbI3 film transforms into a yellow δ-CsPbI3 film within 12 hours.

In organic-inorganic or inorganic lead halide perovskite, the cation usually occupies one A site in either 3D ABX3 or 2D A2BX4 perovskites. Here, the two NH3+ groups of EDA can occupy two A sites and cross-link these 2D layers. This bication 2D perovskite has been theoretically predicted, and the copper halide perovskite based on EDA has been experimentally confirmed (24–26). The EDAPbI4 perovskite film appears greenish, and its absorption spectrum and XRD pattern are listed in Fig. 2 (C and D). Atomic force microscopy (AFM) images reveal that the EDAPbI4 films are consisted of stacks of layered structure (fig. S2). Furthermore, an absorption peak around 420 nm observed in the UV-vis spectrum (Fig. 2B) suggested that the EDAPbI4 is a wider band-gap material (2). Around that energy, the PL lifetimes also increased with the content of EDAPbI4 (fig. S5B). Such a confinement effect was further manifested by the blueshift of absorption onset (Fig. 2D), which is quite evident for the CsPbI3·0.05EDAPbI4 sample. The photoluminescence (PL) spectra of CsPbI3·xEDAPbI4 (x = 0 to 0.05) perovskites.

Within the detection limit of XRD, no peak indexable to any impurity phase, such as δ-CsPbI3 or PbI2, is present. The absence of a PbI2 impurity peak further indicates that ethylenediamine dihydroiodide (EDAI2) is incorporated in the crystal structure. If we add an excess amount (x) of PbI2 without EDAI2 into the precursor, PbI2 crystallizes as a separate phase (fig. S5). The XRD data indicated that the addition of xEDAPbI4 in CsPbI3 does not form a mixture of 2D EDAPbI4 and 3D CsPbI3 perovskite. Furthermore, by increasing the content (x) of EDAPbI4 in CsPbI3·xEDAPbI4 samples, the intensity of the XRD peaks decreases, whereas their peak width broadens, indicating the decrease of crystallite size. Such a confinement effect was further manifested by the blueshift of absorption onset (Fig. 2D), which is quite evident for the CsPbI3·0.05EDAPbI4 sample. The photoluminescence (PL) spectra of CsPbI3·xEDAPbI4 (x = 0 to 0.05) perovskites.
Furthermore, the addition of a small amount of EDAPbI₄ also helps reduce the pinholes in the deposited CsPbI₃ perovskite films and passivates their surface, similar to a previously reported regular 2D/3D or cross-linked 2D/3D perovskite (18, 28). Figure S6B shows that the PL lifetime of the CsPbI₃·xEDAPbI₄ film increases with its EDAPbI₄ content, suggesting a suppression of radiation-less recombination. The scanning electron microscopy (SEM) and AFM images (Fig. 3, A and B) show that the grain size of CsPbI₃·xEDAPbI₄ decreases markedly from ~300 nm (x = 0) to ~35 nm (x = 0.025) with increasing EDAPbI₄ content. This is consistent with the XRD peak broadening observed in Fig. 2. Note that the pinholes become much less with the addition of EDAPbI₄, which is favorable for high-performance PSC fabrication.

All these results reveal that the addition of a small content of EDAPbI₄ has strongly affected the properties of CsPbI₃. To rationalize this observation, we first exclude the possibility of Cs⁺ substitution by EDA on a single A site of CsPbI₃ to form a EDA-Cs₁₋ₓPbI₃ mixed-cation perovskites on the basis of the large size of EDA (r = 0.31 nm). Moreover, replacing Cs⁺ by larger cations, such as EDA, should narrow the band gap and induce an XRD peak shift, although we observe a widening of the band gap and no shift in the XRD peak position.

We adopted a planar configuration to fabricate PSCs based on these CsPbI₃·xEDAPbI₄ perovskites. The photovoltaic parameters extracted from the J-V curves (Fig. 4A) are listed in Table 1. We found that CsPbI₃·xEDAPbI₄ (x = 0.0125 to 0.05) devices show a much better performance than those based on the pure CsPbI₃. The enhancement in all the photovoltaic parameters, that is, JSC, VOC, and FF, can be attributed to less pinholes, desired charge-carrier dynamics, and surface passivation by EDAPbI₄. For x ≤ 0.025, the transient photovoltage decay curves (Fig. S7) exhibit monotonic increase in the lifetime with the EDAPbI₄ incorporation, which is consistent with the PL decay dynamics (Fig. S6B). Furthermore, the 2D EDAPbI₄ in these CsPbI₃·xEDAPbI₄ (x = 0.0125 to 0.05) compositions seem to have less impact on the charge transfer because this can be significantly hindered in regular 2D/3D CsPbI₃·xEA₂PbI₄ perovskite. The transient photovoltage decay curves of CsPbI₃·xEA₂PbI₄ (x = 0.025) deteriorated as markedly as observed in the pure CsPbI₃. As shown in Fig. S11, the typical sample of brown CsPbI₃·0.025EA₂PbI₄ turns into a yellow phase within a day at room temperature. In contrast, the bication EDA with CH₂–CH₂ can effectively assemble the CsPbI₃ crystal units. Furthermore, all the CsPbI₃·xEA₂PbI₄ perovskite film-based solar cells exhibited poor photovoltaic performance, and the best J-V curve obtained from the PSCs based on CsPbI₃·0.025EA₂PbI₄ is listed in Fig. S12. The efficiency is only ~4.4%, which is way too low than that of CsPbI₃ PSCs. Such low stability could be due to either the weak

![Fig. 3. Effect of EDAPbI₄ on the evolution of morphology of CsPbI₃·xEDAPbI₄. (A) AFM and (B) SEM images of perovskite films obtained from the CsPbI₃·xEDAPbI₄ additive precursor (x = 0, 0.0125, 0.025, and 0.05).](http://advances.sciencemag.org/)
Like CsPbI3·0.025EDAPbI4, the CsPbI3·0.025BDAPbI4 samples are of the champion PSC fabricated from CsPbI3·0.025EDAPbI4 as a function of storage time in a dark dry box. (B) XRD pattern and images of the CsPbI3·0.025EDAPbI4 film heated at 100°C in a dry box for 1 week.

Fig. 5. Stability test of CsPbI3·0.025BDAPbI4-based devices and films. (A) PCE of the champion PSC fabricated from CsPbI3·0.025BDAPbI4 as a function of storage time in a dark dry box. (B) XRD pattern and images of the CsPbI3·0.025BDAPbI4 film heated at 100°C in a dry box for 1 week.

Steric effect of short alkyl chain containing the EA cation compared to EDA or the lack of bication effect. Not appropriately, we used a longer-chained alkylamine, such as butylamine (BA), with an even larger size to form the CsPbI3·0.025BA2PbI4 perovskite. The CsPbI3·0.025BA2PbI4 samples show some improved stability than those of CsPbI3·0.025EA2PbI4 but still far more unstable than the CsPbI3·0.025EDAPbI4 samples (fig. S13). This result suggested that both the steric effect and bication in the 2D perovskite improve the phase stability, although the latter might be more important.

To further understand the mechanism behind the high phase stability of CsPbI3·xEDAPbI4, we used other two bications, that is, 1,4-diaminobutane (BA) and (ethylenedioxy)bis(ethylammonium) dihydroiodide (EDBEI2), which are similar to EDA. The BDAI2 perovskite has been demonstrated to be a (100) layered bication 2D perovskite, whereas the EDBEPbI4 has been shown to be a (110) layered 2D perovskite (29–31, 33). Like CsPbI3·0.025EDAPbI4, the CsPbI3·0.025BDAPbI4 samples are composed of smaller perovskite crystallites (fig. S14), exhibiting significantly enhanced thermal stability than pristine CsPbI3. However, after holding at 100°C for 3 days, the CsPbI3·0.025EDAPbI4 film turns into the yellow δ phase, suggesting that its phase stability is lower than that of CsPbI3·0.025EDAPbI4 (fig. S15A). The main difference between EDAPbI4 and BDAI2 is that the former is a (110) layered 2D perovskite, whereas the latter is a (100) layered 2D perovskite. It is likely that the higher phase stability of CsPbI3·0.025EDAPbI4 could be ascribed to the unique (110) layered perovskite structure of EDAPbI4. The CsPbI3·0.025EDAPbI4 sample with the (110) layered EDBEPbI4 shows enhanced thermal stability than CsPbI3; however, as compared to CsPbI3·0.025BDAPbI4, its thermal stability is even poorer, as shown in fig. S15B. It is found that the CsPbI3·0.025BDA2PbI4 perovskite sample shows a larger crystal size than CsPbI3·0.025BDAPbI4 and CsPbI3·0.025EDAPbI4, suggesting that the lower thermal stability of CsPbI3·0.025BDA2PbI4 sample might be due to the presence of large crystallites. All these findings demonstrate that the confluence of reduced crystal size and the unique (110) layered bication 2D perovskite structure enhance the overall phase stability of α-CsPbI3. Specifically, we ascribe the enhanced phase stability of CsPbI3·xEDAPbI4 to the reduced crystallite size and the EDAPbI4 component’s unique (110) layered structure.

In summary, we report a phase-stable α-CsPbI3 film with an EDAPbI4 2D perovskite component prepared via a novel and facile single-step method under ambient conditions for high-efficiency all-inorganic PSCs. By introducing the 2D perovskite of EDAPbI4, the structurally robust α-CsPbI3 perovskite films were obtained even at temperatures several hundred°C below the phase transition point. The addition of a small amount of EDAPbI4 stabilizes the α-CsPbI3. Moreover, these perovskite films can retain the α-CsPbI3 phase even after annealing at 100°C for >150 hours and are also stable at room temperature for months. The EDAPbI4 (x = 0 to 0.05) not only enhances the phase stability of α-CsPbI3 crystals significantly but also connects them for effective electron transfer and passivates the surface defects. Finally, a champion α-CsPbI3 PSC based on CsPbI3·0.025EDAPbI4 perovskite films showing a PCE of 11.8%, a record for all-inorganic PSCs, was realized. Therefore, the concept of using bication presents a novel and promising strategy for designing all-inorganic lead halide PSCs yielding high performance and reliability. Such a bication 2D perovskite with different oriented layer structure concepts could also be extended to balance high performance and high stability in organic-inorganic hybrid lead halide perovskites with the incorporation of a 2D component for their use in optoelectronic applications.

### Table 1. Effect of EDAPbI4 on the metrics of planar CsPbI3 PSCs (12 to 32 cells for each type).

<table>
<thead>
<tr>
<th>Precursor type</th>
<th>JSC (mA/cm²)</th>
<th>VOC (V)</th>
<th>FF</th>
<th>η (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure CsPbI3</td>
<td>11.33 (11.63 ± 1.53)</td>
<td>1.04 (0.89 ± 0.09)</td>
<td>0.65 (0.53 ± 0.06)</td>
<td>7.66 (5.56 ± 1.16)</td>
</tr>
<tr>
<td>CsPbI3·0.0125EDAPbI4</td>
<td>13.59 (13.31 ± 0.36)</td>
<td>1.13 (1.09 ± 0.06)</td>
<td>0.65 (0.56 ± 0.06)</td>
<td>9.98 (8.22 ± 1.46)</td>
</tr>
<tr>
<td>CsPbI3·0.025EDAPbI4</td>
<td>14.53 (14.05 ± 0.57)</td>
<td>1.15 (1.13 ± 0.02)</td>
<td>0.71 (0.64 ± 0.08)</td>
<td>11.86 (10.42 ± 0.91)</td>
</tr>
<tr>
<td>CsPbI3·0.05EDAPbI4</td>
<td>13.97 (13.17 ± 0.88)</td>
<td>1.08 (1.06 ± 0.03)</td>
<td>0.65 (0.61 ± 0.02)</td>
<td>9.81 (8.58 ± 0.66)</td>
</tr>
</tbody>
</table>

#### MATERIALS AND METHODS

**Materials**

EDA12 was synthesized by reacting EDA and hydroiodic acid with a molar ratio of 1:2.2 in an ice bath for 2 hours. The precipitate was collected by rotary evaporation, washed three times with diethyl ether, and then vacuum-dried. 1,4-Diaminobutane dihydroiodide (BDAI2), 2,2′-(ethylenedioxy)bis(ethylammonium) (EDBEI2), and 1,4-Diaminobutane dihydroiodide (BDAI2), ethylenedioxybis(ethylammonium) dihydroiodide (EDBEI2), ethyleniodonium iodide (EAI), and butylammonium iodide (BAI) were synthesized following the similar procedure. The PbI2·xHI sample was synthesized as follows: 1 M PbI2 in DMF solution was reacted with a 1.5 molar ratio of hydroiodic acid for 1 hour, followed by rotary evaporation, washed three times with diethyl ether, and then vacuum-dried; the concentration of PbI2 in the final product should be within the range of 62 to 65 weight %. All the other materials were purchased from Sigma-Aldrich and used as received without any purification.

The CsPbI3·xEDAPbI4 precursor solution was prepared by dissolving 1 mmol of PbI2·xHI and 1 mmol of CsI (1 mM) in 2 ml of DMF to form a 0.5 M precursor solution mixed with different x ratios of...
0.5 M EDAPbI₄ solution. The 0.5 M EDAPbI₄ solution was obtained by dissolving 0.5 mmol of EDAl and 0.5 mmol of PbI₂ into 1 ml of DMF.

**Device fabrication**

A 20-nm-thick compact TiO₂ layer was first deposited on the patterned fluorine-doped tin oxide using 0.2 M Ti(IV) bis(ethyl acetoacetato) disopropoxide in 1-butanol solution at 450°C, followed by annealing at 450°C for 1 hour. The CsPbIₓ:EDApbI₄ precursor solutions were then spin-coated onto the prewarmed c-TiO₂-coated substrate (50°C) at 3500 rpm for 30 s, followed by annealing at 150°C for 2 min. After the films were cooled down to room temperature, a layer of hole transport material of 0.1 M spiro-MeOTAD, 0.035 M bis(trifluoromethane)sulfonii-nium lithium salt, and 0.12 M 4-tert-butylpyridine in chlorobenzene/ acetonitrile (10:1, v/v) solution was spin-coated at 4000 rpm for 20 s. Finally, a 100-nm-thick Ag contact layer was thermally evaporated as back contact.

**Characterization**

The crystal structures of the CsPbIₓ:EDAPbI₄ films were examined by a Shimadzu XRD-6100 diffractometer with Cu Kα radiation. The morphologies of the CsPbIₓ:EDAPbI₄ films were observed by a JSM-7800F Prime SEM and a Bruker Multimode Nanoscope IIIa AFM. The absorption spectra of the EDAPbI₄ and CsPbIₓ:EDApbI₄ perovskite films were taken on a Cary 60 UV–vis spectrophotometer. XPS spectra were acquired with a Kratos Axis Ultra DLD spectrometer (Kratos Analytical-A Shimadzu Group Company) using a monochromatic Al K source (1486.6 eV). Time-integrated PL and time-resolved PL experiments were performed by exciting the samples deposited onto a nonconducting glass using the second harmonic of a picosecond mode-locked Ti-sapphire laser (805 MHz) at 420 nm under ambient conditions. The average power was kept around 0.05 μJ/cm² per pulse. Using a 32-cm focal length monochromator equipped with a charge-coupled device, which has a spectral resolution of >1 meV, and a streak camera with a temporal resolution of >2 ps, the PL data were spectrally and temporally analyzed. The J-V curves of the PSCs were measured by a Keithley 2401 SourceMeter under simulated air mass 1.5-global illumination with a scan rate of 0.05 V/S (100 mW/cm²) (Enlitex SS-F5-3A Class AAA Solar Simulator; the light intensity was calibrated by a stand Si cell before test), equipped with a nonreflective metal mask with an aperture area of 0.12 cm²; the IPCE was measured in atmosphere with a relative humidity of 30 to 45%.

**Supplementary Materials**

For more information, please refer to the supplementary materials available online. This includes detailed experiments and additional results that support the findings presented in the main text. These materials are essential for reproducibility and further research in the field of perovskite solar cells.
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Li, G., Zhang, T., Guo, N., Xu, F., Qian, X., Zhao, Y., Ion-exchange-induced 2D–3D conversion of HMA1,3FAxPb1−xI3 perovskite into a high-quality MA1,3FAxPb1−xI3 perovskite. Angew. Chem. Int. Ed. 55, 13460–13464 (2016).


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