Formamide iodide: a new cation additive for inhibiting δ-phase formation of formamidinium lead iodide perovskite

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Perovskite solar cells (PSCs) employing organic–inorganic hybrid lead perovskite have attracted much attention as promising next generation solar cells because of their low fabrication cost and extremely high power conversion efficiency (PCE). Exploring new perovskite materials and additives is one of the effective strategies to improve the performance of PSCs. Here, we synthesized formamide iodide (FoAI) and applied it as both a cation material and additive. Although it was revealed that FoAI is not incorporated in the A-site of the perovskite structure, we found that the FoAI additive suppresses δ-FAPbI₃ formation and improved the performance of FAPbI₃ based PSCs. The PCE was improved from 12.29% to 14.49% by adding 5 mol% of FoAI in the precursor solution. Meanwhile, we found that FoAI additive can also improve the performance of triple-cation PSCs. We believe that FoAI is one of the promising additives to boost the PCE of PSCs without any influence on the composition of the perovskite materials.

Introduction

Perovskite solar cells (PSCs) employing organic–inorganic hybrid lead perovskite (APbX₃, A: organic cation, X: halogen) have recently attracted much attention as promising next generation solar cells because of their low fabrication cost and extremely high power conversion efficiency (PCE). ¹–³ A PCE of over 25% has been achieved because of excellent optoelectronic properties of the perovskite compounds such as strong absorption, low exciton binding energy and long carrier lifetime. ⁴–⁷ Flexible PSCs can also be achieved owing to low process temperature. ⁵,⁹ Moreover, PSCs can work effectively even under low illuminance conditions such as under indoor lighting; thus, various applications are expected. ¹⁰–¹³

Numerous strategies have been demonstrated to achieve high PCE PSCs such as solvent engineering, additive engineering, and interface modification. ¹⁴–¹⁸ One of the effective strategies is modifying the composition of perovskite compounds. In the early stage of investigations, methylammonium lead iodide (MAPbI₃) was mainly used as the light absorber. A few years later, many researchers shifted to investigating mixed-cation perovskites such as FAMAPbX₃, or CsFAMAPbX₃ (where FA is formamidinium). ¹⁹,²⁰ Mixed-cation perovskites have successfully shown better PCE and stability than conventional PSCs employing mono-cation perovskites. Currently, mono-cation FAPbI₃ has been the focus of halide perovskites again owing to its optimal bandgap for single junction solar cells. In the early investigations of FAPbI₃ PSCs, researchers suffered from poor efficiency and stability due to the formation of δ-FAPbI₃, which is a non-photoactive phase of FAPbI₃. ²¹ Nowadays, phase pure ⁴-FAPbI₃ films have been achieved by several strategies such as using pre-synthesized FAPbI₃ powder as a precursor ²² or adding additives in the precursor solution. ²³ To the best of our knowledge, the highest PCE of 25.17% was achieved with FAPbI₃ employing a little amount of methylenedianmonium dichloride and CsI as the additive. ³

Cation also affects the structure of perovskite compounds. Small cations such as MA, FA, Cs and these mixtures form 3D structured perovskite. Meanwhile, large cations such as phenethylammonium or butylammonium form 2D structured perovskite. ²⁴ 2D structured perovskite compounds show quite different characteristics such as stability and optical properties from 3D structured ones. ²⁴

As mentioned above, cation materials affect the characteristics of perovskite compounds. Therefore, exploring novel cation materials is an important work for further development of perovskite optoelectronics devices. Here, we have synthesized formamide...
iodide (FoAI) and investigated the potential of FoAI as a cation material for PSCs. It was revealed that FoAI itself has very limited inclusion in the 3D perovskite and does not change the perovskite structure from time-of-flight secondary ion mass spectrometry (ToF-SIMS), X-ray diffraction (XRD) patterns and Fourier transform infrared (FT-IR) spectroscopy. However, we found that FoAI suppresses the formation of δ-FAPbI3 when it is applied as an additive for FAPbI3 perovskite. Moreover, it was revealed that FoAI improves the photovoltaic performance of PSCs for both pure FAPbI3 and mixed-cation perovskite compounds. Based on experimental results, we believe that FoAI is one of the promising additives to boost the PCE of PSCs without influence on the composition of perovskite materials.

## Experimental

### Materials

Methylammonium bromide and FAI were purchased from Dyesol. PbI2, PbBr2, and CsI were purchased from TCI. SnO2 colloidal dispersion liquid (15% in H2O) was purchased from Alfa aesar. All other chemicals and solvents were received from Sigma Aldrich and used without purification.

### FoAI synthesis

Formamide (Sigma, ≥99%) was mixed with 2x molar excess of 57% w/w hydroiodic acid and stirred at 50 °C for 10 min. After drying at 100 °C overnight, a white-yellow powder was formed. The formed powder was washed with diethyl ether and recrystallized with ethanol at least five times and dried in an oven overnight.

### Device fabrication

Glass substrates (10 Ohm sq−1, Pilkington TEC10) coated with fluorine-doped tin oxide (FTO) were etched using zinc powder and HCl and then rinsed with pure water, ethanol, and acetone. To prepare the electron transport layer, titanium diisopropoxide bis(acetylacetonate) (75 wt% in isopropanol, Sigma-Aldrich) diluted with molar excess of 1 M PbI2 and HCl and then rinsed with pure water, ethanol, and acetone. To prepare the electron transport layer, titanium diisopropoxide bis(acetylacetonate) (75 wt% in isopropanol, Sigma-Aldrich) diluted with molar excess of 1 M PbI2 and HCl and then rinsed with pure water, ethanol, and acetone.

After cooling the substrate to room temperature, SnO2 colloidal solution diluted in pure water (1:2 weight ratio) was spin-coated on the FTO-coated substrates at 475 °C. Next, the substrates were annealed at 475 °C for 30 min. The active area of the solar cell (ToF-SIMS), X-ray diffraction (XRD) patterns and Fourier transform infrared (FT-IR) spectroscopy. However, we found that FoAI suppresses the formation of δ-FAPbI3 when it is applied as an additive for FAPbI3 perovskite. Moreover, it was revealed that FoAI improves the photovoltaic performance of PSCs for both pure FAPbI3 and mixed-cation perovskite compounds. Based on experimental results, we believe that FoAI is one of the promising additives to boost the PCE of PSCs without influence on the composition of perovskite materials.

### Results and discussion

The molecular structure of FoAI is shown in Fig. 1(a). FoAI has a similar structure to FAI, which is a well-known cation shown in Fig. 1(b). The powder XRD patterns of both compounds are shown in Fig. S1 (ESI†). Fig. 1(c) shows the FT-IR spectra of FoAI and FAI. A characteristic C—N stretching peak was observed in

![Molecular structure of (a) formamidine iodide (FoAI) and (b) formamidinium iodide (FAI). (c) FT-IR spectra of FoAI and FAI.](image-url)

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the spectrum of FAI at around 1700 cm\(^{-1}\). On the other hand, the C=O stretching bond from amide compounds, which usually appears around 1650–1515 cm\(^{-1}\) was not observed in the spectrum of FoAI. It is known that formamide has several resonance structures as shown in Fig. S2 (ESI†). Therefore, there is a possibility that the state which has C=O bonding is unstable and the signal from C=O became weak. We are assuming that the signal of FoAI at around 1400 cm\(^{-1}\) is C=N stretching or C=OH bending. The position of the FT-IR signals and related bonding are summarized in Tables S1 and S2.

Fig. 2(a) shows photographs of FA\(_1\)–FoA–PbI\(_3\) films prepared under various stoichiometries. The \(x=0\), FAPbI\(_3\) film showed black color and it was confirmed that the film mainly consists of PbI\(_2\). FT-IR spectroscopy was carried out to confirm whether FoAI powder includes iodide in the structure. Also, a diffraction peak that cannot be assigned to PbI\(_2\) or PbBr\(_2\) film was observed at around 12\(^\circ\) in the XRD patterns of FoAI–PbBr\(_2\) mixed film. It is reported that PbBr\(_2\) also has a diffraction peak at around 12\(^\circ\). Therefore, it is considered that the possible origin of the peak is from an intermediate mixed halide compound between PbI\(_2\) and PbBr\(_2\) such as PbI\(_1\)Br\(_2\). Actually, it is calculated that PbIBr shows a diffraction pattern around 12\(^\circ\). This compound is also considered as one of the possible origins of the diffraction peak.

Then, we investigated whether FoA could be inserted in the A-site of perovskite compounds with a system of FA\(_1\)–FoA–PbI\(_3\). Fig. 2(a) shows photographs of FA\(_1\)–FoA–PbI\(_3\) films prepared under various stoichiometries. The \(x=0\), FAPbI\(_3\) film showed black color and it was confirmed that the film mainly consists of \(\alpha\)-FAPbI\(_3\) with the presence of \(\delta\) phase from XRD patterns as shown in Fig. 2(b). By contrast, the \(x=1\), FoAPbI\(_3\) film showed bright yellow color which is similar to PbI\(_2\) films. The XRD patterns of FoAPbI\(_3\) films revealed that the film is mainly composed of PbI\(_2\). FT-IR spectroscopy was carried out to confirm whether FoA is present in the resulting films or not. Fig. 2(c) shows FT-IR spectra of each composition film. Films containing less FoA showed clear signals arising from N–H stretching and C=–N stretching in the spectra. By contrast, films containing a large amount of FoA did not show any signals in their spectra. Results from XRD and FT-IR measurements indicate that FoA does not form perovskite.

We found that a solution including FoAI and PbI\(_2\) does not form a precipitate even if chlorobenzene, which is an anti-solvent, is added in the solution. On the other hand, the pure PbI\(_2\) solution immediately forms a precipitate as shown in Fig. S4 (ESI†). This phenomenon indicates that there is a possibility that FoAI forms an adduct or complex with PbI\(_2\) and thus, improves the solubility of PbI\(_2\). Then, we applied FoAI as an additive for PSCs employing FAPbI\(_3\) and mixed-cation perovskite as the light absorber.

Fig. 3(a) shows \(J–V\) curves of PSCs employing FAPbI\(_3\) with and without FoAI as an additive in the precursor solution. The FoAI added sample showed higher PCE particularly with improved short-circuit current density (\(J_{SC}\)) compared to the control devices. Fig. 3(b) and (c) show external quantum efficiency (EQE) spectra of PSCs and absorbance spectra of perovskite films, respectively. The FoAI added device showed an entirely higher EQE value than the control device even though there is almost no change in the absorbance spectra of perovskite films. The band positions of FAPbI\(_3\) films with and without FoAI additive were evaluated with the Kelvin probe method and APS measurements. Fig. S5(a) (ESI†) shows the difference of the surface potential of both films against a reference tip. Fig. S5(b) (ESI†) shows photoemission spectra of both films obtained by APS measurement. There were no obvious changes in surface potential and photoemission spectra, indicating that both perovskite films have same work function and ionization potential. Also, both films showed the same absorption edge as shown in Fig. 3(c). From these results, we assume that the FoAI additive does not affect the band position of FAPbI\(_3\) films. Fig. 3(d) shows the XRD patterns of the FAPbI\(_3\) films with and without FoAI additive. The FAPbI\(_3\) film which does not include FoAI additive showed a diffraction peak originated from \(\delta\)-FAPbI\(_3\), which is not a
photoactive phase for solar cells. On the other hand, FoAI added films did not show diffraction peaks from 5-FAPbI₃. This result indicates that the FoAI additive suppresses the formation of 5-FAPbI₃.

Fig. S6 (ESI†) shows PL spectra of pure FAPbI₃ and FoAI added FAPbI₃ films. Both films show a PL peak at around 800 nm, which is consistent with the absorption edge of the films. Fig. S7 (ESI†) shows SEM images of pure FAPbI₃ and FoAI added FAPbI₃ films. There was almost no change in the morphology of FAPbI₃ films by adding FoAI.

Fig. S8 (ESI†) shows dark J–V curves of electron only devices employing pure and FoAI added FAPbI₃ films. The device structure is FTO/TiO₂/SnO₂/perovskite/PCBM/Ag. The current versus bias behavior can be divided into three different regions, ohmic region, trap filling region, and space charge limited current region. The voltage where the behavior changes from ohmic to trap filling is called the trap-filled limit voltage (V_{TFL}). The relationship between V_{TFL} and trap density (N_i) can be described as follows: N_i = 2e_0eV_{TFL}/eL^2, where e_0 is the vacuum permittivity, e is the relative dielectric constant of perovskite film, e is the electron charge, and L is the film thickness. Comparing the dark J–V curves, we found that the FoAI added sample showed slightly lower V_{TFL}, indicating that FoAI added films have less defects than pure FAPbI₃ films.

Hysteresis index, defined as (PCE_{reverse} – PCE_{forward})/PCE_{reverse}, of FAPbI₃ PSCs was calculated and summarized in Table S3 (ESI†). There was no significant difference in hysteresis index of FAPbI₃ PSCs with and without FoAI additive.

We have also investigated the effect of FoAI on the stability of FAPbI₃ PSCs. Fig. S9 (ESI†) shows the normalized PCE of FAPbI₃ PSCs kept at a maximum power point under continuous 1 sun irradiation without encapsulation. After 90 min irradiation, the PCE of pure FAPbI₃ PSCs decreased to 42.7% of the initial value. By contrast, FoAI added PSCs kept a slightly higher value of 49.3%. This result indicates that FoAI might also improve the stability of FAPbI₃ PSCs under working conditions.

The J–V characteristics of PSCs are summarized in Table 1. The PCE of our control sample (12.29%) seems to be low considering the record efficiency (>25%) of PSCs. However, it is comparable to the baseline of pure FAPbI₃ devices whose PCE is around 13–17%. We believe that our method offers an alternative strategy to stabilize and improve the performance of pure FAPbI₃ based devices.

Then, we applied FoAI additive in multi-cation PSCs, which are the baseline of high performing perovskite cells in general.

**Table 1**  J–V characteristics of FAPbI₃ perovskite solar cells. Values in brackets are from the best performing device under each condition

|          | J_{SC} (mA cm⁻²) | V_{OC} (V) | FF   | PCE (%) |
|----------|------------------|------------|------|---------|
| Control  | 16.86 ± 0.53     | 0.964 ± 0.023 | 0.649 ± 0.036 | 10.71 ± 1.10 |
| Forward  | 17.31            | 0.979      | 0.668 |         |
| Control  | 17.17 ± 0.48     | 0.977 ± 0.007 | 0.687 ± 0.016 | 11.47       |
| Reverse  | 17.55            | 0.983      | 0.704 |         |
| With FoAI| 18.98 ± 0.92     | 1.000 ± 0.005 | 0.685 ± 0.010 | 11.29       |
| Forward  | 19.63            | 1.007      | (0.696) | 13.16 ± 0.84 |
| With FoAI| 19.05 ± 0.75     | 1.005 ± 0.008 | 0.717 ± 0.010 | 13.90 ± 0.54 |
| Reverse  | 19.59            | (1.014)    | (0.721) |         |

Fig. 4(a) shows J–V curves of triple-cation PSCs with and without FoAI additives. EQE spectra of each sample are shown in Fig. S10 (ESI†). The V_{OC} was slightly improved by adding FoAI in the precursor solution. Fig. 4(b) shows PL spectra of triple-cation perovskite films with and without FoAI additives. Perovskite films containing 0.625 and 1.25 mol% of FoAI showed stronger PL intensity than the control films, indicating that FoAI helps the formation of less defect perovskite films and resulted in improved V_{OC}. There were no obvious changes in absorbance spectra, XRD patterns, and morphology as shown in Fig. S11 and S12 (ESI†). Dark J–V characteristics of the electron only device employing triple-cation perovskite films are shown in Fig. S13 (ESI†). In contrast to FAPbI₃ films, there was no significant difference in V_{TFL} of both triple-cation perovskite films. The J–V characteristics of triple-cation PSCs with and without FoAI additives are summarized in Table 2. The hysteresis index of triple-cation PSCs was calculated and summarized in Table S4 (ESI†). There was no significant difference in hysteresis index by FoAI concentration.

Fig. S14 (ESI†) shows FT-IR spectra of FoAI added triple-cation perovskite films. Although signals originated from N–H, C–N, and C==N bonding of MAI and FAI were observed, there was no clear signal at around 1400 cm⁻¹, where characteristic peaks were observed in the spectrum of FoAI (Fig. 1). This result also indicates that FoAI is not included as an A-site cation of a perovskite structure.

We further investigated whether FoAI is included in the final perovskite films with ToF-SIMS which has a higher detection limit than FT-IR measurements. Fig. S15 (ESI†) shows the ToF-SIMS profile (positive ion) of pure FAPbI₃ and FoAI added films.
It is difficult to conclude whether the FoA ion is included in the final films from positive ion mode because the FA ion and FoA ion have a similar m/z value (FA: m/z = 45, FoA: m/z = 46). Actually, the pure FAPbI3 film showed a signal of m/z = 46, which might result from isotopic atoms contained in FA ions. Then, we checked negative ions included in FoA added FAPbI3 films. Fig. S16 (ESI†) shows I and O ion distribution in FoA added films. O ions were not detected in the perovskite films, indicating that FoA is not included in the final films. We also could not detect the O ion in FoA added triple cation perovskite films as shown in Fig. S17 (ESI†).

From inhibited δ-FAPbI3 formation (Fig. 3(d)) and enhanced PL intensity of perovskite films (Fig. 4(b)), there is a possibility that FoA affects the crystal growth of perovskite films. It is also observed in the XRD patterns of the perovskite films (Fig. 3(d) and Fig. S11(b), ESI†) that the FoA added films showed stronger intensity than the pure perovskite films. This result also indicates that FoA affects the crystal growth of perovskite films. One of the possible mechanisms is that FoA forms adduct with PbI2 as mentioned above. It is known that adduct formation leads to highly efficient PSCs. Furthermore, it is reported that iodide defects in perovskite films affect the device performance of PSCs. There is a possibility that iodide from FoA reduces the defects and improves the efficiency of PSCs. Although further investigation is necessary to clarify the detailed role of FoA, we believe that FoA is one of the promising materials to boost the PCE of PSCs.

### Conclusions

In summary, we synthesized FoA and applied it as a material for PSCs. Through the fabrication and evaluation of FA1−xFoAxPbI3 films, it was confirmed that FoA is not incorporated in the perovskite structure. However, it was confirmed that FoA improves the solubility of PbI2 probably due to the formation of an adduct or complex. When FoA was applied as an additive for FAPbI3 based solar cells, it was confirmed that δ-FAPbI3 formation was suppressed and the photovoltaic performance of FAPbI3 PSCs was improved. The PCE of FAPbI3 PSCs was improved from 12.29% to 14.49% by adding 5 mol% of FoA in the precursor solution. We found that FoA additive also improves the photovoltaic performance of triple-cation (FA, MA, and Cs mixed-cation) PSCs. FoA added triple-cation perovskite films showed higher PL intensity than control films and FoA added PSCs showed slightly improved VOC. From the above results, we assume that FoA affects the crystal growth of perovskite films.

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We believe that FoA will be one of the promising materials to boost the performance of PSCs.
Communication

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