

**Understanding the Optoelectronic Properties of
Different Perovskite Semiconductors for
Development of Cost-efficient, Eco-friendly and
Recyclable Perovskite Technology**

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JANUARY, 2024**

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by

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Submitted

In fulfilment of the requirements of the degree of

Doctor of Philosophy

to the



INDIAN INSTITUTE OF TECHNOLOGY, DELHI

JANUARY, 2024

*Dedicated to
My Family....*

CERTIFICATE

This is to certify that the thesis entitled, “**Understanding the Optoelectronic Properties of Different Perovskite Semiconductors for Development of Cost-efficient, Eco-friendly and Recyclable Perovskite Technology**” submitted by **Ms. Priyanka Chhillar** to the Department of Energy Science and Engineering, Indian Institute of Technology Delhi, for the award of the degree of the **Doctor of Philosophy** is a record of bonafide research work carried out by her. She has worked under my supervision for the submission of this thesis, which to the best of my knowledge, has reached the requisite standards.

The research work presented in this thesis has not been submitted in parts or in full to any other University or Institute for the award of any degree or diploma.

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Acknowledgements

First and foremost, I want to offer my praise and gratitude to the God Almighty, for the blessings, peace, strength, and health, he bestowed upon me in order to finish my research. This thesis has become a reality with the kind support and help of many individuals. I would like to extend my sincere gratitude to all of them.

I would like to express my sincere gratitude to my dissertation advisor, Dr. Supravat Karak, who provided me with all the support and guidance in carrying out my research work and formulating the work into a publishable story. I profusely admire and appreciate his valuable suggestions and insights during my Ph.D. work. His prompt inspirations, timely suggestions with kindness, enthusiasm, and dynamism have enabled me to complete this thesis.

I would also like to express my sincere gratitude to my previous supervisor, Dr. Sandeep Kumar Pathak, under the guidance of whom I started learning the basics of my research work. I am deeply indebted to him for his continuous guidance, moral support, valuable suggestions, and enthusiastic encouragement during the entire research work. I learned a lot from him, how to carry out quality research and to express the work in the correct manner for publishing.

I would like to extend my sincere regards to Prof. Vamsi K. Komarala, Head of the Department of Energy Science and Engineering (DESE) for providing the essentials and Prof. Viresh Dutta, Prof. Josemon Jacob, Prof. Vamsi K. Komarala, my research committee members for their guidance, encouragement and support. I thank my department faculty members and supporting staff for their cooperation and valuable support.

My sincere regards to Nanoscale Research Facility and Central Research Facility faculty members and project staff for providing the characterization facilities to carry out my research. I extend my sincere thanks to the Indian Institute of Technology Delhi for providing me the financial support.

I convey my gratitude to my labmates Dr. Shailendra Kumar Gupta, Bhanu Pratap Dhamaniya, Amit Kumar, Kartiki Chandratre, Saurabh Pareek, Punit Sharma, Rakesh Suthar, and Dr. Abhijit T, along with all members of the PV group for providing their indispensable support and help throughout my research work.

Special thanks and gratitude to my parents and my younger brother for their unconditional love, care, support, guidance, prayers, and sacrifice for educating and preparing me for my future. It is their unconditional support, motivation, and the strength they enrooted in me throughout my life, that provided me strength for the completion of all my academic pursuits.

Before I end, I would like to extend my regards to everyone who has supported me in any aspect during my Ph.D., with my sincere apology, if I was unable to mention them individually.

Priyanka Chhillar

Abstract

The advent of organic-inorganic halide perovskites has been a major breakthrough in the field of photovoltaic research. The prominent interest of the photovoltaic community in this technology is accounted for its low cost and easy solution processable fabrication technique along with its excellent optoelectronic properties. However, its mass commercialization has not seen the light of day due to some inherent issues such as structural and ambient instability in operating environment conditions and the presence of toxic lead. In this thesis work, we have worked towards devising solutions for these hurdles in the path of the commercialization of perovskite solar cells. Initially, we worked on the MAPbI₃-based perovskite, which is known to be highly unstable under operating environmental conditions such as humidity, temperature, oxygen presence, and illumination. On exposure to the ambient, the MAPbI₃ perovskite is decomposed and left with a remnant lead iodide (PbI₂) layer as a by-product. Owing to the toxicity of lead which has health implications and the high expenses involved in the disposal of hazardous waste, the disposal of this lead waste is a major concern, especially in developing countries. Therefore, we realized that either we need to substitute the lead or we need to make sure to reuse it, therefore the idea of recycling perovskite solar cells comes as a great relief. In this thesis, we have shown that the final decomposed product of perovskite, the PbI₂ film, can successfully be recycled to perovskite extending the life span of the perovskite-based material to multiple folds and hence reducing the payback period even further. This work furnishes a comparative study among the three fabrication routes to prove the viability of efficient recycling, which paves the path for selecting a better precursor composition. We have carried out an extensive study on the recycling of degraded perovskite films which are fabricated via three different routes (i.e. Sequential deposition, 3CH₃NH₃I:Pb(Ac)₂ and 3CH₃NH₃I:PbCl₂) and encountered that not all the films can be recycled efficiently. The film deposited with precursor composition 3CH₃NH₃I:Pb(Ac)₂ shows efficient recycling pertaining to the optoelectronic properties comparable to the original perovskite but it is not the case with the other two methods. We have also stated the reasons behind ineffective recycling using the other two methods. We also underline the fact that if this highly crystalline degraded PbI₂ film can be utilized back into MAPbI₃ it may result in altogether supreme optoelectronic properties.

The presence of volatile MA^+ in $MAPbI_3$, which is highly unstable under thermal stress and humidity, prevents its mass commercialization. So, over the course of time, $FAPbI_3$ and $CsPbI_3$ perovskites have emerged as potential alternatives with better thermal stability and suitable band gap. However, both of these suffer from notable phase instability, rendering their application into photovoltaic devices quite challenging. In this thesis, we have devised a method to stabilize the γ - $CsPbI_3$ photoactive phase by incorporation of Mg^{2+} in the lattice. Successful replacement of around 15% toxic Pb with Mg has been achieved in the $CsPbI_3$ lattice. Two photoactive phases were obtained in Mg containing $CsPb_{1-x}Mg_xI_3$ films, one at a low temperature ranging from 80-140°C and the other above 320°C, whereas in the pristine $CsPbI_3$ film, the photoactive phase is obtained only above 320°C. Polymorph reversal mechanisms have been studied for the pristine $CsPbI_3$ films and $CsPb_{1-x}Mg_xI_3$ films. The work reported herein illustrates a comprehensive understanding of the structural and optoelectronic properties of the various phases obtained in fresh $CsPb_{1-x}Mg_xI_3$ films at various temperatures and after the recuperation of the photoactive phase post exposure to ambient. In addition, the role of Mg incorporation on the film morphology has been extensively studied. These findings suggest that the Mg incorporation not only drastically reduces the formation temperature for the photoactive phase but also enhances the phase stability and thermal stability of the photoactive phase of $CsPbI_3$. Interestingly, Mg incorporation has led to an enhancement in the optoelectronic properties of the $CsPbI_3$ film and resulted in better film morphology.

Although the incorporation of Mg led to enhanced phase stability and optoelectronic properties it still lacked behind in terms of long term ambient stability. Therefore in the subsequent study, we have devised a method for long term stabilization of the γ - $CsPbI_3$ photoactive phase by incorporation of Mg^{2+} and Tris(2-aminoethyl)amine (TAEA) in the lattice. Interestingly, our finding suggests that the presence of both Mg and TAEA is crucial for the long-term ambient stability of the photoactive phase. The $CsPb_{1-x}Mg_xI_3$ film containing TAEA crystallizes to the γ -phase at two temperatures, one at a low temperature ranging from 120-150°C and the other above 320°C, whereas the TAEA containing $CsPbI_3$ film crystallizes to a black phase only above 320°C. The $CsPb_{1-x}Mg_xI_3$ films containing TAEA formed at low temperatures are found to be the most stable among all the films. The optimized $CsPb_{0.85}Mg_{0.15}I_3$ containing TAEA is found to showcase drastically enhanced thermal stability of more than 7 months when continuously exposed to 85°C in an N_2 atmosphere and ambient phase stability of more than 20 days when stored in an ambient

environment with RH~60-70%. Along with the enhanced stability CsPb_{0.85}Mg_{0.15}I₃ containing TAEA has also shown superior optoelectronic properties than the CsPbI₃ and CsPb_{0.85}Mg_{0.15}I₃ films.

Owing to the notable phase instability of FA-based perovskites, FA_{1-x}Cs_x perovskite composition is a widely accepted alternative for better phase and thermal stability. In this thesis, we have presented a detailed comparative study of the TAEA containing films with the pristine FA_{0.83}Cs_{0.17}Pb(I_{0.90}Br_{0.10})₃ films. Interestingly, our findings demonstrate that the presence of TAEA leads to a drastic enhancement in optoelectronic properties of the FA_{0.83}Cs_{0.17}Pb(I_{0.90}Br_{0.10})₃ films also. This improvement in optoelectronic properties is accounted for by the improved structural and morphological properties and defect passivation in TAEA containing film. TAEA addition leads to a reduction in the metallic Pb⁰ content in the target films. The FA_{0.83}Cs_{0.17}Pb(I_{0.90}Br_{0.10})₃ films containing TAEA also exhibit drastically enhanced thermal stability of more than 7 days under continuous exposure to extreme conditions of high humidity, illumination, and thermal stress at 85°C. Additionally, these TAEA containing films also showcase extremely high ambient stability of more than 180 days in illumination and highly humid conditions with RH~60-70%.

In the subsequent study, we analyzed the effect of the incorporation of In³⁺ on the phase stability of γ -CsPbI₃ using the addition of InI₃ and InCl₃ in different quantities in the CsPbI₃ precursor. Based on the results, we found that the incorporation of both salts resulted in enhanced phase stability and absorption properties. It is noticed that the addition of InCl₃ to an optimum concentration leads to enhanced optoelectronic properties and improved surface morphologies. However, the InI₃ containing films show diminished photoluminescence properties.

All the above results pave the path toward the realization of the devised perovskite compositions for a cost-efficient, recyclable, comparatively less toxic, and long-term stable perovskite technology with enhanced optoelectronic properties.

सारांश

कार्बनिक-अकार्बनिक हैलाइड पेरोस्काइट्स का आगमन फोटोवोल्टिक अनुसंधान के क्षेत्र में एक बड़ी सफलता रही है। इस प्रौद्योगिकी में फोटोवोल्टिक समुदाय की प्रमुख रुचि इसकी कम लागत और आसान समाधान प्रक्रिया योग्य निर्माण तकनीक के साथ-साथ इसके उत्कृष्ट ऑप्टोइलेक्ट्रॉनिक गुणों के कारण है। हालाँकि, ऑपरेटिंग वातावरण की स्थितियों में संरचनात्मक और परिवेशीय अस्थिरता और विषाक्त सीसे की उपस्थिति जैसे कुछ अंतर्निहित मुद्दों के कारण इसके बड़े पैमाने पर व्यावसायीकरण को दिन का प्रकाश नहीं देखा गया है। इस थीसिस कार्य में, हमने पेरोस्काइट सौर कोशिकाओं के व्यावसायीकरण के मार्ग में इन बाधाओं के लिए समाधान तैयार करने की दिशा में काम किया है। प्रारंभ में, हमने MAPbI₃-आधारित पेरोस्काइट पर काम किया, जिसे आर्द्रता, तापमान, ऑक्सीजन की उपस्थिति और रोशनी जैसी परिचालन पर्यावरणीय परिस्थितियों में अत्यधिक अस्थिर माना जाता है। परिवेश के संपर्क में आने पर, MAPbI₃ पेरोस्काइट विघटित हो जाता है और उप-उत्पाद के रूप में अवशेष लेड आयोडाइड (PbI₂) परत के साथ छोड़ दिया जाता है। सीसे की विषाक्तता के कारण जिसका स्वास्थ्य पर प्रभाव पड़ता है और खतरनाक कचरे के निपटान में आने वाले उच्च खर्च के कारण, इस सीसे के कचरे का निपटान एक बड़ी चिंता का विषय है, खासकर विकासशील देशों में। इसलिए, हमने महसूस किया कि या तो हमें सीसे को प्रतिस्थापित करने की आवश्यकता है या हमें इसका पुनः उपयोग सुनिश्चित करने की आवश्यकता है, इसलिए पेरोस्काइट सौर कोशिकाओं को पुनर्चक्रित करने का विचार एक बड़ी राहत के रूप में आता है। इस थीसिस में, हमने दिखाया है कि पेरोस्काइट के अंतिम विघटित उत्पाद, PbI₂ फिल्म को सफलतापूर्वक पेरोस्काइट में पुनर्चक्रित किया जा सकता है, जिससे पेरोस्काइट-आधारित सामग्री का जीवन काल कई गुना बढ़ जाता है और इस प्रकार पेबैक अवधि और भी कम हो जाती है। यह कार्य कुशल पुनर्चक्रण की व्यवहार्यता साबित करने के लिए तीन निर्माण मार्गों के बीच एक तुलनात्मक अध्ययन प्रस्तुत करता है, जो एक बेहतर पूर्ववर्ती संरचना के चयन का मार्ग प्रशस्त करता है। हमने तीन अलग-अलग मार्गों (यानी अनुक्रमिक जमाव, 3CH₃NH₃I:Pb(Ac)₂ और 3CH₃NH₃I:PbCl₂) के माध्यम से निर्मित अपमानित पेरोस्काइट फिल्मों के पुनर्चक्रण पर एक व्यापक अध्ययन किया है और पाया है कि सभी फिल्मों को कुशलतापूर्वक पुनर्चक्रित नहीं किया जा सकता है। पूर्ववर्ती संरचना 3CH₃NH₃I:Pb(Ac)₂ के साथ जमा की गई फिल्म मूल पेरोस्काइट की तुलना में ऑप्टोइलेक्ट्रॉनिक गुणों से संबंधित कुशल रीसाइक्लिंग दिखाती है लेकिन अन्य दो तरीकों के मामले में ऐसा नहीं है। हमने अन्य दो तरीकों का उपयोग करके अप्रभावी रीसाइक्लिंग के पीछे के कारणों को भी बताया है। हम इस तथ्य को भी रेखांकित करते हैं कि यदि इस अत्यधिक क्रिस्टलीय अवक्रमित PbI₂ फिल्म को MAPbI₃ में वापस उपयोग किया जा सकता है तो इसके परिणामस्वरूप पूरी तरह से सर्वोच्च ऑप्टोइलेक्ट्रॉनिक गुण प्राप्त हो सकते हैं।

MAPbI₃ में अस्थिर MA⁺ की उपस्थिति, जो थर्मल तनाव और आर्द्रता के तहत अत्यधिक अस्थिर है, इसके बड़े पैमाने पर व्यावसायीकरण को रोकती है। इसलिए, समय के साथ, FAPbI₃ और CsPbI₃ पेरोव्स्काइट बेहतर थर्मल स्थिरता और उपयुक्त बैंड गैप के साथ संभावित विकल्प के रूप में उभरे हैं। हालाँकि, ये दोनों उल्लेखनीय चरण अस्थिरता से ग्रस्त हैं, जिससे फोटोवोल्टिक उपकरणों में उनका अनुप्रयोग काफी चुनौतीपूर्ण हो गया है। इस थीसिस में, हमने जाली में Mg²⁺ को शामिल करके γ -CsPbI₃ फोटोएक्टिव चरण को स्थिर करने की एक विधि तैयार की है। CsPbI₃ जाली में लगभग 15% विषैले Pb का Mg के साथ सफल प्रतिस्थापन प्राप्त किया गया है। CsPb_{1-x}Mg_xI₃ फिल्मों वाले Mg में दो फोटोएक्टिव चरण प्राप्त हुए, एक 80-140°C के कम तापमान पर और दूसरा 320°C से ऊपर, जबकि प्राचीन CsPbI₃ फिल्म में, फोटोएक्टिव चरण केवल 320°C से ऊपर प्राप्त होता है। प्राचीन CsPbI₃ फिल्मों और CsPb_{1-x}Mg_xI₃ फिल्मों के लिए पॉलीमॉर्फ रिवर्सल तंत्र का अध्ययन किया गया है। यहां बताया गया कार्य विभिन्न तापमानों पर ताजा CsPb_{1-x}Mg_xI₃ फिल्मों में प्राप्त विभिन्न चरणों के संरचनात्मक और ऑप्टोइलेक्ट्रॉनिक गुणों की व्यापक समझ को दर्शाता है और परिवेश के संपर्क में आने के बाद फोटोएक्टिव चरण की पुनरावृत्ति के बाद होता है। इसके अलावा, फिल्म आकृति विज्ञान पर एमजी निगमन की भूमिका का बड़े पैमाने पर अध्ययन किया गया है। इन निष्कर्षों से पता चलता है कि एमजी निगमन न केवल फोटोएक्टिव चरण के लिए गठन तापमान को काफी कम कर देता है बल्कि CsPbI₃ के फोटोएक्टिव चरण की चरण स्थिरता और थर्मल स्थिरता को भी बढ़ाता है। दिलचस्प बात यह है कि एमजी निगमन से CsPbI₃ फिल्म के ऑप्टोइलेक्ट्रॉनिक गुणों में वृद्धि हुई है और इसके परिणामस्वरूप बेहतर फिल्म आकारिकी प्राप्त हुई है।

हालाँकि एमजी के समावेश से चरण स्थिरता और ऑप्टोइलेक्ट्रॉनिक गुणों में वृद्धि हुई, लेकिन दीर्घकालिक परिवेश स्थिरता के मामले में यह अभी भी पीछे है। इसलिए बाद के अध्ययन में, हमने जाली में Mg²⁺ और ट्रिस (2-एमिनोइथाइल) अमाइन (TAEA) को शामिल करके γ -CsPbI₃ फोटोएक्टिव चरण के दीर्घकालिक स्थिरीकरण के लिए एक विधि तैयार की है। दिलचस्प बात यह है कि हमारी खोज से पता चलता है कि एमजी और टीईईए दोनों की उपस्थिति फोटोएक्टिव चरण की दीर्घकालिक परिवेश स्थिरता के लिए महत्वपूर्ण है। TAEA युक्त CsPb_{1-x}Mg_xI₃ फिल्म दो तापमानों पर γ -चरण में क्रिस्टलीकृत होती है, एक 120-150°C के कम तापमान पर और दूसरी 320°C से ऊपर, जबकि CsPbI₃ फिल्म युक्त TAEA केवल ऊपर एक काले चरण में क्रिस्टलीकृत होती है। 320°C कम तापमान पर बनी TAEA युक्त CsPb_{1-x}Mg_xI₃ फिल्में सभी फिल्मों में सबसे अधिक स्थिर पाई गईं। TAEA युक्त अनुकूलित CsPb_{0.85}Mg_{0.15}I₃ में N₂ वातावरण में लगातार 85°C के संपर्क में रहने पर 7 महीने से अधिक की थर्मल स्थिरता और RH के साथ परिवेशीय वातावरण में संग्रहीत होने पर 20 दिनों से अधिक की परिवेश चरण स्थिरता प्रदर्शित होती है। ~60-70%। बढ़ी हुई स्थिरता के साथ-साथ TAEA युक्त CsPb_{0.85}Mg_{0.15}I₃ ने CsPbI₃ और CsPb_{0.85}Mg_{0.15}I₃ फिल्मों की तुलना में बेहतर ऑप्टोइलेक्ट्रॉनिक गुण भी दिखाए हैं।

एफए-आधारित पेरोव्स्काइट की उल्लेखनीय चरण अस्थिरता के कारण, एफए1-एक्ससीएसएस पेरोव्स्काइट संरचना बेहतर चरण और थर्मल स्थिरता के लिए एक व्यापक रूप से स्वीकृत विकल्प है। इस थीसिस में, हमने प्राचीन $FA_{0.83}Cs_{0.17}Pb(I_{0.90}Br_{0.10})_3$ फिल्मों के साथ TAEA युक्त फिल्मों का एक विस्तृत तुलनात्मक अध्ययन प्रस्तुत किया है। दिलचस्प बात यह है कि हमारे निष्कर्ष दर्शाते हैं कि टीईई की उपस्थिति से $FA_{0.83}Cs_{0.17}Pb(I_{0.90}Br_{0.10})_3$ फिल्मों के ऑप्टोइलेक्ट्रॉनिक गुणों में भी भारी वृद्धि होती है। ऑप्टोइलेक्ट्रॉनिक गुणों में यह सुधार टीईई युक्त फिल्म में बेहतर संरचनात्मक और रूपात्मक गुणों और दोष निष्क्रियता के कारण है। टीईई के अतिरिक्त लक्ष्य फिल्मों में धात्विक Pb^0 सामग्री में कमी आती है। TAEA युक्त $FA_{0.83}Cs_{0.17}Pb(I_{0.90}Br_{0.10})_3$ फिल्में $85^\circ C$ पर उच्च आर्द्रता, रोशनी और थर्मल तनाव की चरम स्थितियों के निरंतर संपर्क में 7 दिनों से अधिक समय तक काफी बढ़ी हुई थर्मल स्थिरता प्रदर्शित करती हैं। इसके अतिरिक्त, ये टीईई युक्त फिल्में रोशनी में 180 दिनों से अधिक की अत्यधिक उच्च परिवेश स्थिरता और आरएच $\sim 60-70\%$ के साथ अत्यधिक आर्द्र स्थितियों को भी प्रदर्शित करती हैं।

बाद के अध्ययन में, हमने $CsPbI_3$ अग्रदूत में अलग-अलग मात्रा में InI_3 और $InCl_3$ को जोड़ने का उपयोग करके $\gamma-CsPbI_3$ की चरण स्थिरता पर In^{3+} के समावेश के प्रभाव का विश्लेषण किया। परिणामों के आधार पर, हमने पाया कि दोनों लवणों के समावेश से चरण स्थिरता और अवशोषण गुणों में वृद्धि हुई। यह देखा गया है कि इष्टतम सांद्रता में $InCl_3$ के जुड़ने से ऑप्टोइलेक्ट्रॉनिक गुणों में वृद्धि होती है और सतह आकारिकी में सुधार होता है। हालाँकि, InI_3 युक्त फिल्में कम फोटोल्यूमिनेसेंस गुण दिखाती हैं।

उपरोक्त सभी परिणाम लागत-कुशल, पुनर्चक्रण योग्य, तुलनात्मक रूप से कम विषैले और उन्नत ऑप्टोइलेक्ट्रॉनिक गुणों के साथ दीर्घकालिक स्थिर पेरोव्स्काइट तकनीक के लिए तैयार पेरोव्स्काइट रचनाओं की प्राप्ति का मार्ग प्रशस्त करते हैं।

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Abbreviations and symbols

IEA	International Energy Agency
TWh	Terra Watt hour
CdTe	Cadmium Telluride
CIGS	Copper Indium Gallium Selenide
a-Si	Amorphous Silicon
CdS	Cadmium Sulphide
NREL	National Renewable Energy Laboratory
Si	Silicon
Ag	Silver
Au	Gold
Al	Aluminium
Sn	Tin
Pb	Lead
Mg	Magnesium
In	Indium
PSC	Perovskite Solar Cell
PV	Photovoltaic
DSSC	Dye-Sensitized Solar Cells
OSC	Organic Solar cell
ITO	Tin doped Indium Oxide
TiO ₂	Titanium dioxide
FTO	Fluorine doped Tin Oxide
XRD	X-ray Diffraction
PL	Photoluminescence
SEM	Scanning Electron Microscopy
AFM	Atomic Force Microscopy
EDS	Energy Dispersive X-ray Spectroscopy
SLCM	Scanning laser Confocal Microscopy
FTIR	Fourier Transform Infrared
XPS	X-Ray Photoelectron Spectroscopy
HTL	Hole Transport Layer
ETL	Electron Transport Layer

DMF	Dimethyl formamide
DMSO	Dimethyl sulfoxide
IPA	Iso-propyl alcohol
DI	De-ionized
MAI	Methylammonium Iodide
FAI	Formamidinium Iodide
CsI	Cesium Iodide
PbI ₂	Lead Iodide
MgI ₂	Magnesium Iodide
InI ₃	Indium Iodide
InCl ₃	Indium Chloride
Pb(Ac) ₂ ·3H ₂ O	Lead Acetate Trihydrate
FAPbI ₃	Formamidinium Lead Iodide
RT	Room Temperature
RH	Relative Humidity
MAPbI ₃	Methylammonium Lead Iodide
CsPbI ₃	Caesium Lead Iodide
HI	Hydroiodic acid
PCE	Power Conversion Efficiency
GaAs	Gallium Arsenide
PbS	Lead Sulfide
P3HT	Poly(3-hexylthiophene)
PC ₇₁ BM	[6,6]-Phenyl-C71-butyric acid methyl ester
TAEA	Tris(2-aminoethyl) amine
Spiro-OMeTAD	2,2',7,7'-tetrakis[N,N-di(4-methoxyphenyl)amino]-9,9'-spirobifluorene
Li-TFSI	Lithium bis-(trifluoromethane)sulfonimide
LUMO	Lowest Unoccupied Molecular Orbital
HOMO	Highest Occupied Molecular Orbital
r.m.s.	Root-mean-square
GW	Gigawatt
k_B	Boltzman's constant
q	Elementary charge of electron

J_{sc}	Short-circuit current density
V_{oc}	Open-circuit voltage
FF	Fill Factor
μm	micrometer
μL	microliter
nm	nanometer
mL	milliliter
mg	milligram
eV	Electron Volt