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Solution-Processed Multifunctional Thin-Film Encapsulation of Perovskite Thin Films and Devices

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Herein, the effect of multicomponent composite encapsulation on the stability of perovskite thin films and perovskite solar cells, as well as lead leakage upon water immersion, is investigated. The encapsulation is simple and low cost since it is entirely deposited by solution processed techniques in the ambient atmosphere. It consists of a spray-coated composite layer sandwiched between two spin-coated layers. The composite layer contains hygroscopic nanomaterials, oxygen scavengers, and lead adsorbing nanomaterials, which enables reduced lead leakage and improved stability of encapsulated perovskite during storage in ambient, immersion in water, as well as illumination in dry air. The encapsulation layers show high transmittance and did not have a significant effect on the short-circuit current density and open-circuit voltage despite the deposition of encapsulation in ambient air. The encapsulated devices retain 80% of their initial performance after 4 h of immersion in water.

1. Introduction

Due to the need to improve the stability of perovskite solar cells (PSCs), the stability of metal halide perovskite materials and devices has been extensively studied.^[1–3] In addition to optimizing the perovskite material and PSC architecture, encapsulation plays a key role in improving operational stability of PSCs.^[2,4–7] The encapsulation can also serve an important function of minimizing lead leakage into the environment.^[5] Common encapsulation method, similar to other types of solar cells, involves the use of

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of thin-film encapsulation is of significant interest, in particular for applications in flexible solar cells and modules.^[5,7] Consequently, a number of different deposition materials and techniques have been reported for thin-film encapsulation of perovskite films and PSCs.^[6,8–35] Thin-film encapsulation can be used alone, which is commonly insufficient for stringent stability tests (outdoor, damp heat),^[5] or in combination with cover-glass/cover-sheet encapsulation^[5,30] since it can lead to improved stability compared to cover glass only encapsulation.^[30]

cover glass.^[4–7] However, the development

Thin-film encapsulation can be deposited by vapor deposition as well as solution processing.^[6] In addition, some less common methods involve mechanical transfer of layered materials.^[32,33] Among vapor

deposition methods, atomic layer deposition (ALD) is very common,^[6,8–16] since it allows deposition of conformal, continuous, dense, and pinhole-free films.^[6] Moreover, ALD can be used to deposit single layers^[8–12] or to deposit one or more layers in a multilayer structure.^[13–16] Multilayer films in particular provide excellent barrier properties.^[13–16] However, ALD is a time-consuming and costly deposition technique, which raises interest in the development of low-cost alternatives.^[6]

Therefore, solution-processed encapsulation is of significant interest due to its low cost^[4] and suitability for flexible/low weight

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device applications^[5,7] since it enables improved mechanical stability compared to inorganic thin films, which develop microfractures on bending which enables moisture and oxygen ingress.^[7] Polymer-based encapsulation layers can be spin-coated^[4] or spray-coated.^[17] A variety of different polymer materials have been explored to date, with different tests applied to assess the effectiveness of encapsulation. As humidity is well-known to negatively affect PSCs^[1] since exposure to liquid water is known to degrade perovskite within seconds,^[7] significant efforts in thin-film encapsulation have been devoted to the mitigation of the effects of moisture by designing hydrophobic encapsulation layers^[7,19,22,25,31] or encapsulation layers with low water vapor transmission ratios.^[14–16,20] However, to address the degradation of PSCs under illumination, it is necessary to consider not only moisture but also oxygen barrier properties, since oxygen has a significant contribution to the degradation under illumination.^[1,3,6] However, polymer-based encapsulation effectiveness as oxygen barrier is largely unexplored.^[7] Furthermore, the investigations of composite encapsulation materials have been scarce, and composite materials would typically include only one type of nanomaterial.^[28,29] Nevertheless, composite materials are of particular interest since they allow integration of materials with different functionalities, such as incorporation of hygroscopic nanoparticles^[28] to slow down the degradation upon exposure to moisture.

In this work, we investigate a multicomponent composite layer for thin-film encapsulation of PSCs. The composite is prepared by spray-coating, which is a promising deposition technique for composite films^[28] and it enables deposition of thick layers which would slow down permeation of oxygen and moisture into the devices.^[17] The composite films contain hygroscopic nanomaterials (MgO nanoparticles),^[36] mesoporous silica nanospheres (MSN) as oxygen scavengers,^[37,38] and reduced graphene oxide (rGO)^[39] as lead adsorbing material. MSN have been reported to have oxygen sorption capacity

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exceeding that of commercial scavengers (\approx 5–7 mmol g⁻¹, depending on synthesis conditions and relative humidity [RH]).^[37] The moisture adsorption capability of MgO is illustrated in Figure S1, Supporting Information, showing thermogravimetric analysis (TGA) curves of MgO samples with and without exposure to the ambient atmosphere (≈60-70% RH) for different times, where increased mass loss for MgO exposed to humidity clearly illustrates that it can function as an efficient desiccant. Similarly, Figure S2, Supporting Information, illustrates the lead adsorption capability of rGO. Thus, each nanomaterial component of the spray-coated layer serves a distinct function to either improve stability (by reducing moisture and oxygen penetration) or by reducing Pb leakage. The spraycoated layer is sandwiched between spin-on-glass^[40] and polyisobutylene (PIB) polymer layers which serve the function of protecting the PSC from damage during spray-coating in ambient and sealing any pinholes or cracks in the spray-coated film, respectively. Such multicomponent encapsulation has the advantage of high optical transmission and facile, low-cost solution-based processing. It is demonstrated to be effective protection of the devices subjected to harsh testing conditions (water immersion), and it also results in a significant reduction of lead leakage from the devices.

2. Results and Discussion

To test the function of the different components of our composite encapsulation, we first tested the encapsulation on perovskite films. Methylammonium (MA) lead iodide (MAPI) was used as a model perovskite material due to its poor stability under exposure to ambient and illumination. We have first investigated the effect of the inclusion of a spray-coated composite layer on the effectiveness of encapsulation by comparing spin-coated layers only (spin on glass (SOG)/PIB bilayer) to the full encapsulation



Figure 1. Schematic diagrams of thin-film encapsulation of a) perovskite films and b) perovskite solar cells; photos of perovskite films for different times of water immersion for c) SOG/PIB and d) SOG/Comp/PIB.

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stack (SOG/Comp/PIB). Water immersion test was used as a harsh environment test which causes quick degradation of perovskite films without encapsulation (Figure S3, Supporting Information), in agreement with expectations.^[7] Figure 1 shows the effect of encapsulation with and without a composite layer on the stability of MAPI film for different times of water immersion. We can observe that without the composite film, damage can already be observed after 1 h, while the inclusion of the composite film significantly improves the film stability. Past works on thin-film encapsulation often included short duration water immersion tests (tens of seconds or minutes),[25,28,29,35] while works reporting good stability for the order of hours or davs^[13,14,24,26] have been scarce. Thus, the investigated composite thin-film encapsulation is very promising. The encapsulation with SOG/Comp/PIB also provides effective protection of MAPI film during ambient storage (7 days, at 70% RH), as shown in Figure S4, Supporting Information.

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The thickness of different layers has been determined from cross-section scanning electron microscope (SEM) images (Figure S5, Supporting Information), while the morphology was additionally examined by top-view SEM imaging (Figure S6, Supporting Information). The average thickness values are $425\pm29\,\text{nm}$ for MAPI film, $215\pm33\,\text{nm}$ for SOG, $1.8\pm0.6\,\mu\text{m}$ for Comp, and $67 \pm 3.6 \,\mu\text{m}$ for PIB film. The encapsulation stack exhibits excellent transparency, with no additional absorption bands observable (Figure S7, Supporting Information), showing only \approx 3% reduction of transmission for quartz substrates and 4% reduction of transmission for PEN substrates. To investigate the function of different nanomaterials in the composite film, we have performed stability testing under illumination in different atmospheres, as shown in Figure 2. In all cases, encapsulation provides significant protection against degradation. We can observe that in dry air encapsulation containing MSN only results in the smallest degradation due to its oxygen scavenging properties. When the encapsulated films are exposed to humidity only, samples containing only MgO provide the best performance, verifying the function of the different components. It should be noted though that while higher quantity of the nanomaterial would allow higher adsorption of oxygen or moisture, too high a nanomaterial concentration results in worsening the spray-coated film morphology and consequently worsened stability. In addition to preventing the ingress of oxygen and moisture into the PSCs, encapsulation can serve an important function to

into the PSCs, encapsulation can serve an important function to reduce lead leakage from the devices.^[5,41–46] To test the function of rGO for lead adsorption, lead leaching from samples immersed in water was measured and the obtained results are shown in **Figure 3**. We can observe a significant reduction in lead leakage with the inclusion of rGO in the encapsulation. For encapsulation without rGO, we can observe that the concentration increases up to 6 h and then exhibits a small decrease. This can occur due to complexation of leaked Pb²⁺ and any solvent residue,^[41] or due to the fact that PbI₂ dissolution to Pb²⁺ and I⁻ is reversible, so that higher Pb²⁺ and I⁻ concentrations in the solution can slow down the dissolution and speed up the reverse reaction.^[46] It should be noted that both with and without rGO the lead leakage is lower than that from cover-glass-encapsulated devices with cover-glass damage when no lead



Figure 3. Pb concentration in water for different times of water immersion of MAPI films encapsulated with composite encapsulation with and without rGO.



Figure 2. XRD patterns of MAPI films with different encapsulation subjected to illumination for 24 h in a) dry air b) N_2 + moisture (40% RH).



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Figure 4. a) J-V curves and b) PCE distribution of control and encapsulated devices.

adsorber is used.^[43–45] In addition, the encapsulation technique is scalable, and similar protection of the perovskite film is obtained on substrates with 2.5×2.5 and 5×5 cm sizes, as shown in Figure S8, Supporting Information.

After verifying that our solution-processed encapsulation can effectively protect sensitive MAPI films, we prepared PSCs. In this case, we used a different perovskite composition $(FA_{0.9}Cs_{0.1}PbI_{2.9}Br_{0.1})$ to achieve higher efficiency, so that the effectiveness of encapsulation could be tested on devices with high performance. The prepared devices had an inverted architecture with a NiO_x-based hole-transport layer. The device performance of control and encapsulated devices is illustrated in **Figure 4** and summarized in **Table 1**. Figure S9, Supporting

Table 1. Photovoltaic parameters of PSCs with/without encapsulation (reverse scan). Average values are shown for 10 devices, while values corresponding to the best performing devices are shown in brackets.

	PCE [%]	$J_{\rm sc} [{\rm mA}{\rm cm}^{-2}]$	$V_{\rm oc}$ [V]	FF [%]
Control	21.44 ± 0.26	24.37 ± 0.43	1.07 ± 0.01	82.2 ± 0.7
Encapsulated	(21.82) 20.90 ± 0.12	(24.75) 24.33 ± 0.18	(1.03) 1.07 ± 0.01	(81.5) 80.3 ± 0.1
	(21.11)	(24.50)	(1.07)	(80.6)

Information, shows the corresponding external quantum efficiency (EQE) and stabilized power output curves. Both control and encapsulated devices exhibit good performance with a power conversion efficiency (PCE) of \approx 21%. We can observe that there is negligible difference in short-circuit current density, Isc, and open-circuit voltage, Voc. A small decrease in efficiency after encapsulation occurs almost entirely due to a reduction in fill factor (FF) due to increased series resistance. This likely occurs due to incomplete removal of some of the encapsulation layers (likely SOG) over the contact area or damage to the Ag metal coating caused by edge removal to ensure complete coverage of the device area by the thin-film encapsulation. To test whether the problem was caused by encapsulation, ethanol was spray-coated on the devices, and no significant difference in cell performance was obtained, as shown in Figure S10, Supporting Information. Ethanol was selected as a solvent since it did not damage perovskite films, and it was capable of dispersing all components of the spray-coated film (rGO, MgO, MSN) well, different from dichloromethane, toluene, or hexane, which were not capable of dispersing well one or more components. Then, we examined whether the edge removal process caused a decrease in FF. From Figure S11, Supporting Information, we can observe that removal of part of the top electrode results in a drop in FF (likely due to higher resistance of the underlying indium tin oxide [ITO]



Figure 5. a) Normalized PCE versus immersion time in water for devices with and without encapsulation. b) Normalized PCE versus time of maximum power point test in ambient air (RH = 70%).

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only compared to ITO/Ag), and that the FF value can be recovered by applying silver paint on the contact area. This shows the significant effect of contacts on the FF.

Device stability was tested as a function of time of water immersion, as shown in **Figure 5a**. Corresponding photos of devices for different times of water immersion are shown in Figure S12, Supporting Information. We can observe that the performance of control devices degrades rapidly, while the encapsulated devices retain 80% of the initial performance after 4 h of water immersion (\approx 70% of initial PCE after 6 h of water immersion). Significant enhancement in stability of encapsulated devices is observed under maximum power point tracking in ambient air (RH 70%), as shown in Figure 5b. The thin-film encapsulation enabled an increase in T₈₀ by \approx 4.5 times compared to control devices.

3. Conclusions

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We investigated solution-processed multicomponent thin-film encapsulation for perovskite thin films and devices. We demonstrated that composite encapsulation films could be used to target different functions, with hygroscopic material protecting the perovskite from moisture degradation, oxygen scavenger from photooxidation, and rGO providing a lead adsorption function. The multicomponent encapsulation provided excellent protection for water immersion, with devices retaining 80% of initial efficiency after 4 h of immersion in water.

4. Experimental Section

Materials: Cetyltrimethylammonium bromide (CTAB, purity 99%) and cesium iodide (purity 99.9%) were purchased from Thermo Scientific. Lead (II) bromide (purity 98%), lead (II) iodide (purity 99.99%), (3-aminopropyl)triethoxysilane (APTES, purity 98%), and (4-(3,6-dimethyl-9Hcarbazol-9-yl)butyl)phosphonic acid (Me-4PACz, purity 99%) were purchased from TCI. Methylammonium iodide (MAI), formamidinium iodide (FAI), and phenethylammonium bromide (PEABr) were purchased from Great Cell Solar. Tetraethyl orthosilicate (TEOS, purity 99%) was purchased from Dieckmann. Ethanol (purity 99.5%), chlorobenzene (purity 99.8%), and isopropyl alcohol (IPA, purity 99.5%) were purchased from Sigma Aldrich. Ammonium hydroxide (ACS, 28-30%), 3,3',5,5'-tetramethylbenzidine (TMB, purity 98%), dimethylformamide (DMF, 99.9%), and dimethyl sulfoxide (DMSO) were purchased from Alfa Aesar. The [6,6]-phenyl-C61-butyric acid methyl ester (PCBM, purity 99.5%) and bathocuproine (BCP, purity 99.5%) were purchased from Lumtec. Hexane (purity 99%) was purchased from RCI Labscan. PIB (M.W. 600 000) was purchased from J&K Scientific. IC1-200 was purchased from Futurrex. Commercial magnesium oxide nanoparticles (200 nm, purity 99%) were purchased from NanoAmor. rGO solution (purity 99%) was purchased from Timesnano. All materials were used as received without any further purification.

Synthesis of Mesoporous Silica Nanospheres: MSN were synthesized as described previously.^[37] Briefly, 0.6 g of CTAB was added to a 20 mL mixture of 16 mL deionized water and 4 mL of ethanol. An amount of 0.3 mL of ammonium hydroxide was then added to the mixture, followed by 15 min of ultrasonication. An amount of 0.9 mL of TMB and 2 mL of TEOS were added to the reaction mixture, followed by 2 h of stirring on a magnetic hot plate at 800 rpm. The gel product underwent centrifugation washing at 4000 rpm for 30 min, followed by 2 rounds of washing using ethanol at 4000 rpm for 10 min each round. The gel was then dried in a vacuum oven at 70 °C overnight before being ground using a mortar and pestle to give a fine MSN powder.

Fabrication of MAPbI₃ Films: Briefly, 160 mg of MAI and 461 mg of lead (II) iodide were dissolved in a 715 μ L mixture of 640 μ L of DMF and 75 μ L of DMSO. Stirred at 60 °C for 2 h and filtered before use. The precursor solution was then spin-coated onto the substrate at 4000 rpm for 30 s. At 7 s after the start, 300 μ L of chlorobenzene anti-solvent was dropped on the substrate. The film was then annealed at 100 °C for 15 min.

Fabrication of $FA_{0.9}Cs_{0.1}PbI_{2.9}Br_{0.1}$ Films: Briefly, 155 mg of FAI, 26 mg of cesium iodide, 433 mg of lead (II) iodide, and 22 mg of lead (II) bromide were dissolved in a 714 µL mixture of 571 µL of DMF and 143 µL of DMSO. The mixture was stirred overnight at 60 °C and filtered before use. The perovskite film was prepared by an anti-solvent method with a two steps program: 2000 rpm for 10 s and 4000 rpm for 30 s. During the second step, 250 µL of chlorobenzene was dropped on the substrate 10 s before the end of the program. The films were then annealed at 100 °C for 30 min.

Fabrication of Perovskite Solar Cells: The device structure of the PSC was ITO/NiO_x/Me-4PACz/FA_{0.9}Cs_{0.1}Pbl_{2.9}Br_{0.1}/PEABr/PCBM/BCP/Ag. To prepare the devices, a NiO_x nanoparticle dispersion^[47] was prepared and spin-coated onto a clean ITO/glass substrate at 3000 rpm for 40 s, followed by annealing at 110 °C for 10 min to form the hole-transporting layer. Then, a 0.5 mg mL⁻¹ solution of Me-4PACz in ethanol was spin-coated at 4000 rpm for 30 s, followed by annealing at 100 °C for 10 min. The perovskite was fabricated as described earlier. For the 2D perovskite layer, a 0.8 mg mL⁻¹ solution of PEABr in IPA was spin-coated dynamically at 5000 rpm for 30 s, followed by annealing at 100 °C for 3 min. For the electron-transport layer, a 20 mg mL⁻¹ solution of PCBM in chlorobenzene was spin-coated at 1200 rpm for 30 s, followed by annealing at 100 °C for 10 min. The BCP interlayer was then spin-coated at 4000 rpm. Finally, silver electrodes (100 nm thick) were deposited using a thermal evaporator.

Encapsulation: The encapsulation consisted of three parts. First, for the spin-on glass layer: 200 μ L of IC1-200 solution was deposited at 4000 rpm for 30 s via spin-coating, followed by annealing at 110 °C for 15 min in an ambient environment. The substrate was allowed to cool to room temperature naturally. Second, for the spray-coated layer, 20 mg of MgO nanoparticles, 200 mg rGO, and 20 mg MSNs were added to a vial of 10 mL ethanol. The solution was then sonicated for 1 h before $50\,\mu\text{L}$ of APTES was added before use. An amount of 1 mL of the as-prepared solution was spray-coated uniformly onto the substrate at 55 °C. It was then annealed for 2 min at 55 °C before being cooled down to room temperature. Third, for the PIB layer: the substrate surface was covered with 12 mg mL PIB solution (dissolved overnight in hexane). The solvent was allowed to evaporate at room temperature for 2–3 min. An amount of 200 μL of the PIB solution was then dynamically spin-coated onto the top at 1000 rpm for 30 s. The casting and spin-coating steps were then repeated once more to give a thicker layer with better coverage.

Water Immersion Testing: MAPbI₃ films were prepared with different encapsulation strategies. They were then immersed in 30 mL of deionized water in a petri dish for a specified time. To study the effectiveness of the encapsulation strategy on device performance, PSCs were prepared and encapsulated as previously mentioned. They were immersed in 30 mL of deionized water in a petri dish, periodically taken out and dried with nitrogen gas to record any change in efficiency overtime.

Ambient Storage: To test the effectiveness of the encapsulation strategy in ambient conditions, control and encapsulated MAPbI₃ films were stored in darkness (petri dish wrapped with aluminum foil) at 22 °C and 70% RH for 7 days.

Oxygen and Moisture-Only Environment Illumination Experiments: Control and encapsulated MAPbI₃ films were illuminated under 1 sun simulated solar illumination (AM1.5 G) using a G2V Sunbrick LED solar simulator for 24 h. Films were in either oxygen-only or controlled-moisture conditions following a previously reported protocol.^[48] Perovskite stability was determined by measuring changes in their X-Ray diffraction (XRD) patterns before and after illumination.

Characterization: Transmittance spectra was measured using the Agilent Cary 60 UV–vis spectrometer. XRD measurements were carried out using a Rigaku MiniFlex600 with a Cu K α X-Ray source. For the XRD measurements of encapsulated samples, the encapsulation layer was removed using Kapton tape before measurement. Inductively coupled plasma optical emission spectroscopy measurements were carried out



using an Agilent 5110VDV to determine the lead (Pb) content leaked from the MAPbl₃ films during water immersion and the lead adsorption capability of rGO. For determining the moisture adsorption capacity of MgO, TGA was carried out using the TA Instruments Q50 Thermogravimetric Analyzer. For determining the PCE of devices, current density–voltage (J–V) scans were measured using a Keithley 2400 Source Measure Unit with a scan step size of 0.03 V and delay time of 10 ms. The active area of 0.04 cm² was defined by a shadow mask. The devices were illuminated with 100 mW cm⁻² AM 1.5 G illumination from an ABET Sun 2000 solar simulator calibrated with a silicon reference cell. SEM images were taken using a Hitachi S-4800 field-emission SEM to determine the cross section of the multilayer encapsulation.

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.

Author Contributions

Abdul Khaleed: Investigation (equal); Visualization (equal). Hongbo Mo: Investigation (equal); Visualization (equal). Ali Asghar Syed: Conceptualization (supporting); Investigation (supporting). Atta Ur Rehman: Investigation (supporting); Methodology (supporting). Yin Li: Investigation (supporting). Jingbo Wang: Investigation (supporting). Yixuan Wang: Investigation (supporting). Tao Zhu: Investigation (supporting). Yanting Shen: Investigation (supporting). Gang Li: Funding acquisition (supporting); Supervision (supporting). Kaimin Shih: Supervision (supporting). Aleksandra B. Djurišić: Funding acquisition (lead); Supervision (lead); Writing—original draft (lead). Abdul Khaleed and Hongbo Mo have contributed equally to this work.

Data Availability Statement

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

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encapsulations, lead halide perovskites, solar cells

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