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Stability of perovskite solar cells on flexible substrates

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ABSTRACT

Perovskite solar cells are emerging photovoltaic technology with potential for low cost, high efficiency devices. Currently, flexible devices efficiencies over 15% have been achieved. Flexible devices are of significant interest for achieving very low production cost via roll-to-roll processing. However, the stability of perovskite devices remains a significant challenge. Unlike glass substrate which has negligible water vapor transmission rate (WVTR), polymeric flexible film substrates suffer from high moisture permeability. As PET and PEN flexible substrates exhibit higher water permeability then glass, transparent flexible backside encapsulation should be used to maximize light harvesting in perovskite layer while WVTR should be low enough. Wide band gap materials are transparent in the visible spectral range low temperature processable and can be a moisture barrier. For flexible substrates, approaches like atomic layer deposition (ALD) and low temperature solution processing could be used for metal oxide deposition. In this work, ALD SnO_2, TiO_2, Al_2O_3 and solution processed spin-on-glass was used as the barrier layer on the polymeric side of indium tin oxide (ITO) coated PEN substrates. The UV-Vis transmission spectra of the prepared substrates were investigated. Perovskite solar cells will be fabricated and stability of the devices were encapsulated with copolymer films on the top side and tested under standard ISOS-L-1 protocol and then compared to the commercial unmodified ITO/PET or ITO/PEN substrates. In addition, devices with copolymer films laminated on both sides successfully surviving more than 300 hours upon continuous AM1.5G illumination were demonstrated.

Keywords: Perovskite, stability testing, encapsulation, flexible solar cells

1. INTRODUCTION

Among all challenges faced by our current society, energy collection is still one of the largest obstacles. Fossil fuel is still the major resource for our daily energy consumption but renewable energy is the way out for energy crisis.[1] Sun solely provide around 120 petawatt of power to the earth while only a small fraction (17 terawatt) is needed globarlly.[2] Majority of sun power will be lost to hear unless using photovoltaic (PV) devices to harvest the energy which can transfer photon from sun light to electrical energy. Nowadays, surging of power generation through PV devices was observed from less than 1% in 2000 to more than 9% in 2014 globally thanks to the development of PV technologies.[3] Over 90% of the current PV market was dominated by silicon solar cells, particularly crystalline silicon (c-Si). Although price for c-Si was dropping significantly from \$70 to 0.7\$ per W_p from 1970s to 2014, researchers are aiming for lower cost with less than \$0.5 per W_p.[4] Latest generation of PV like dye-sensitized solar cells and organic solar cells aims for low-cost large area depositions but efficiency (12-14%) cannot catch up with c-Si (over 25%). Recently, hybrid organic-inorganic perovskite solar cells may have potential to compete with c-Si technology.

Hybrid organic-inorganic perovskite refers to materials with crystal structure with formula ABX_3, where A is monovalent cation, B is divalent metal cation and X is typically halide anion (Cl^-, Br^-, I^-). BX_6 octahedra will be formed by having 6-fold halide anion surrounding B metal cation. For PV applications, methyl ammonium lead iodide ($MAPbI_3$) was widely studied due to its low density of defects, over 150 μm charge carriers (both electrons and holes) diffusion lengths, good absorption coefficient with 103 cm^{-1}, low exciton binding energy and also with the excellent bandgap of 1.55eV.[5,6,7] Therefore, it is possible to use perovskite for planar heterojunction configurations. Perovskite solar cells are typically fabricated into either conventional (n-i-p) or inverted (p-i-n) structure. Both structures having light absorber

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perovskite sandwiched between electron transport layer (ETL) and hole transport layer (HTL) that can effectively extract electrons or holes generated from perovskite layer, where perovskite is deposited on ETL and HTL respectively for conventional and inverted structure.[6] Metal electrodes and transparent electrodes are used for guiding carriers out of solar devices.

Development of perovskite solar cells is rapidly in recent. Originally, perovskite solar cell was first reported by Kojima and co-workers in 2009 with efficiency of 3.8% through replacing organic sensitizer with perovskite in dye sensitized solar cells.[8] Degradation observed due to solubility of perovskite in electrolyte is high. Later in 2012, Grätzel and his coworkers fabricated cell achieved 9.7% with mesoporous TiO_2 layer replacing liquid electrolyte while Snaith and his coworkers fabricated planar TiO_2 based perovskite solar cells with efficiency of 12.3%.[9,10] Throughout these few years, perovskite solar cells is a popular research field attracting researchers, rapid advancement of power covered efficiency (PCE) was achieved with over 22% closing the gap with c-Si PV technology.[11,12] Other than PCE advancement, perovskite solar cells are also low cost owning to the fact that deposition of perovskite layer is low temperature below 150°C so manufacturing cost can be reduced compared to c-Si. However, high PCE perovskite solar cells typically fabricated on rigid glass substrates that also include using high temperature (over 450°C) processed TiO_2 layer as ETL. In order to further lower the cost, low temperature processing ETL was reported for perovskite solar cells fabrications.[13,14] Lower thermal budget also allows the possibility of using flexible plastic films as substrates such as polyethylene terephthalate (PET) and polyethylene naphthalate (PEN), with cost roughly 30% lower than using glass substrate.[15] Moreover, this enables the use of roll-to-roll printing system which can do massive production while cutting down manufacturing costs.

Although PCE of perovskite based solar cells on glass substrates achieved over 22%, fabricating devices on flexible substrates poses challenges like low temperature limitation and distortions from flexible substrates affecting the deposition quality of perovskite. In general, efficiencies of devices on glass substrates are higher than flexible devices counterparts. Flexible inverted perovskite solar cells with efficiency more than 17% can be achieved while on rigid glass efficiency can be over 20%.[16] Nevertheless, these performance is still higher than those achieved by organic PV and dye sensitized solar cells (less than 15%).[17,18] Other than closing the efficiency gap between glass and plastic substrate, stability issue of perovskite is still the major challenge hindering the commercialization of this new technology.[19] Concerns about the long term stability of perovskite are raised because perovskite is sensitive to humidity and UV exposure.[20] Degradation of $MAPbI_3$ when exposed to moisture starts with formation of reversible hydrate form. Although without illumination this process is reversible, the hydrate form will further degrade into PbI_2 upon continuous illumination. Once moisture infiltrates into perovskite, transport of water to whole perovskite is in a scale of seconds. This degradation occurs resulting in $MAPbI_3$ degradation into PbI_2 and MAI, and MAI is then furthered degraded into CH_3NH_3 and HI. UV illumination will facilitate HI to react with oxygen and produce H_2 and I_2.[21] Therefore, in order to improve the lifetime of perovskite solar cells, less moisture sensitive perovskite derivatives with better intrinsic stability or devices plus better encapsulation isolating the active area of solar cells from moisture upon exposure under ambient environment are needed. As most of researchers focus on pushing PCE of perovskite solar cells to its limit, there is still room of improvement for extending lifetime of these devices, particularly for flexible perovskite solar cells. For practical applications, high efficiency with long lifetime is the target, and that is the reason why this work focuses on extending stability.

For flexible perovskite solar cells, encapsulation will be a greater challenge as it is more difficult to encapsulate effectively. Plastic films used instead of glass substrate are less resistive to moisture and oxygen infiltration. For non-oriented PET and PEN flexible substrates, permeability of oxygen transport is 0.462 and 0.167 cc(STP) cm m^{-2} day^{-1} atm^{-1} respectively.[22] Plastic films with low water vapor transmission rate (WVTR) should be considered. As a result, encapsulation is needed for both the active area side and the polymeric side of devices to minimize the rate of infiltration of moisture and oxygen into devices. On the active area side of devices, plastic film with low WVTR can be used to cover the surface such as barrier layer developed for OPV or OLEDs.[23] Adhesion can be done using double sided adhesive tape.[24] Material which has high transmission to visible spectrum and low WVTR should be used for polymeric side. Atomic layer deposition of metal oxides can act as protective layers on the polymeric side of films as the temperature is low enough, transparent to visible spectrum and resistive to infiltration of water. It has been demonstrated

that perovskite solar cells with metal oxide ALD deposition can survive after direct contact with water for few seconds.[25] On the other hand, PEN may work better to protect devices from UV illumination than PET as PEN can block UV light. Otherwise, extra UV filters should be incorporated on the backside of solar cells.

In this present work, commercially available plastic barrier tape with incorporation of highly transparent cyclic olefin copolymer (COC) films as topside encapsulation for active area. Extension of electrodes from perovskite solar cells were done using tin coated copper tape and sealed up using UV-curable epoxy on the edges. Both indium tin oxide (ITO) coated PET and PEN substrates were used for solar cells fabrications. After determining which polymer substrates are better for stability performance, different wide band gap metal oxides with good transmission of visible light were deposited using low temperature processing methods on the polymeric side of flexible substrates, including atomic layer deposition and solution processed spin-coating. Primary aim of this work is to investigate the best encapsulation strategy to improve the stability of flexible perovskite based solar cells. Perovskite solar cells were fabricated using conventional structure with SnO_2 as ETL. ISOS-L-1 with laboratory constant continuous light source (AM1.5G) from solar simulator was strictly followed for stability testing.[28]

2. EXPERIMENTAL DETAILS

2.1 Materials

Solvents used including anhydrous N,N-Dimethylformamide (DMF, ≥99.9%), anhydrous dimethyl sulfoxide (DMSO, ≥99.9%) and anhydrous isopropanol (IPA, ≥99.5%) were purchased from Alfa Aesar. Tin(IV) oxide, 15% in H_2O colloidal dispersion (SnO_2) was purchased from Alfa Aesar. Lead iodide (PbI_2, 99%), bis-(trifluoromethane) sulfonamide lithium salt (LiTFSI, 99.95%), 4-tert-butylpyridine (96%), chlorobenzene (CB, ≥99.5%), and anhydrous acetonitrile (ACN, 99.8%) were purchased from Sigma Aldrich. Methylammonium iodide (MAI) was purchased from Lumtech. 2,2',7,7'-tetrakis[N,N-di(4-methoxyphenyl)amino]-9,9'-spirobifluorene (spiro-OMeTAD) was obtained from Shenzhen Feiming Co., Aluminum pellets (Al) and molybdenum oxide (MoO_3) pellets were purchased from Kurt J. Lesker Company. ITO/PEN (15 ohm/sq) was purchased from Peccell with thickness of 0.125 mm while ITO/PET (15 ohm/sq) was purchased from WuHan Jinge-solar Energy Technology Co., Ltd with thickness of 0.175 mm.

2.2 Preparation of precursors

For the electron transport layer material, SnO_2 colloidal dispersion solution was diluted to 3 wt% in H_2O with deionized water, followed by 30 minutes of sonication. Methylammonium lead iodide precursor was prepared for one step spin coating. The as-prepared perovskite precursor solution was mixed with PbI_2 and CH_3NH_3I in a molar ratio of 1:1 giving the concentration of 1.1M. DMF and DMSO in a volume ratio of 2:1 were then added. Magnetic stirring was also applied overnight to ensure chemicals were completely dissolved. For hole transport layer material, Spiro-OMeTAD was prepared in a concentration of 72.3 mg per 1 ml of chlorobenzene. 28.8 µL of 4-tert-butylpyridine and 17.5 µL of Li-TFSI solution (in a concentration of 520 mg per 1 mL of acetonitrile) were also added at the same time. Spiro-OMeTAD precursor was stirred overnight.

2.3 Fabrications of flexible perovskite solar cells

Conventional tructure of perovskite solar cells was used, from bottom to top is plastic films (PET or PEN)/ ITO/ SnO_2/ perovskite / spiro-OMeTAD/ MoO_3/ Al, where perovskite is $CH_3NH_2PbI_3$ acting as a light absorber , planar SnO_2 nanoparticle dispersion acting as an electron transport layer and spiro-OMeTAD is (2,2',7,7'-tetrakis-(N,N-di-p-methoxyphenylamine)-9,9'-spirobifluorene acting as a hole transport layer. Patterned ITO/PEN or ITO/PET substrates were sequentially cleaned through sonication in acetone, ethanol and deionized water at last for 10 minutes each. Substrates were then dried with nitrogen spray gun and underwent UV ozone treatment for 20 minutes. SnO_2 colloidal precursor was span coated in ambient environment with spin speed of 3000 rpm for 30 seconds in room temperature, followed by 10 minutes of annealing at 120°C on a hotplate. Spin coating of perovskite and spiro-OMeTAD was done

inside glove box to minimize the exposure of moisture. Precursor of perovskite was filtered with 0.45 µm PTFE syringe filter and then coated on ITO/SnO_2 flexible substrates with spin speed of 1000 rpm for 10 seconds then 5000 rpm for 25 seconds. Chlorobenzene was drop-cast using a pipette when last 15 seconds of spin coat process remained. Edges of substrates were cleaned with DMF and dried immediately using spin coater. After that, 50°C of annealing was followed by for 5 minutes and 100°C for 10 minutes. Spiro-OMeTAD was span coated on perovskite for 4000 rpm for 30 seconds. 15 nm of MoO_3 and 100 nm of Al were thermally evaporated through a 0.1 cm^2 mask on hole transport layer to complete devices fabrication.

2.4 Encapsulation of solar cells

Device encapsulation was done inside glove box filled with argon. Barrier tape was provided from Tesa with the thickness of 25 µm, WVTR of 35 g m^{-2} day^{-1} with transmission > 99% from 400 nm to 800 nm. Cyclic olefin copolymer (COC) films was provided by TOPAS with thickness of 70 µm. Norland optical adhesive 61 was used as edge sealant. Devices were encapsulated in two different conditions: (1) device with active area side were encapsulated with COC film with barrier tape as adhesion using laminator at 80°C. (2) device (2 cm x 2 cm) was sandwiched with two larger pieces of COC films (3 cm x 3 cm) using barrier tapes as adhesive, then laminated at 80°C. For both conditions, edge sealant was then applied and then cured under UV exposure (365nm). Tin-coated copper tape was used to guide the electrodes outside of encapsulation for the ease of measurement. For polymeric side encapsulation, substrates were coated with SnO_2, TiO_2, and Al_2O_3 for 20 nm respectively by ALD before fabrication of cells. In addition, spin-on-glass (SOG) was also spin-coated on backside of substrates with 3000 rpm for 30s and then annealed at 120°C for 60 minutes.

2.5 Characterization of solar cells

Current density against voltage sweep was recorded using a Keithley 2400 sourcemeter under the solar simulator with AM1.5G illumination at 100 mW m^{-2}. Voltage sweep was done from 1.2V to -0.2V with voltage step and delay time of 0.01 V and 0.01 s respectively. Stability testing was done following the testing protocol ISOS-L-1. For laboratory weathering testing condition, devices were kept under constant AM1.5G illumination from ABET Sun 2000 solar simulator with temperature 25°C and relative humidity of 85% monitored. I-V characterization was done at specified interval of time during stability testing.

3. RESULTS AND DISCUSSION

Devices with the conventional structure (PET or PEN)/ ITO/ SnO_2/ perovskite / spiro-OMeTAD/ MoO_3/ Al were fabricated as shown on Figure 1. SnO_2 colloidal precursor was used as electron transport layer because it can be processed at low temperature (<150°C) and in ambient environment. PCE of SnO_2 based perovskite solar cells on glass substrate from us was 15.2% (J_{sc} is 22.4 mA/cm^2, V_{oc} is 1.04V and fill factor FF is 0.66). Encapsulated flexible devices exhibited PCE ranging from 1.7% to 6.8% with average PCE of 3.7. We have observed lower initial efficiencies for PET substrates that may due to inferior quality of ITO on PET. We carried out stability testing for all encapsulated devices and trend of PCE through time under continuous illumination was observed.

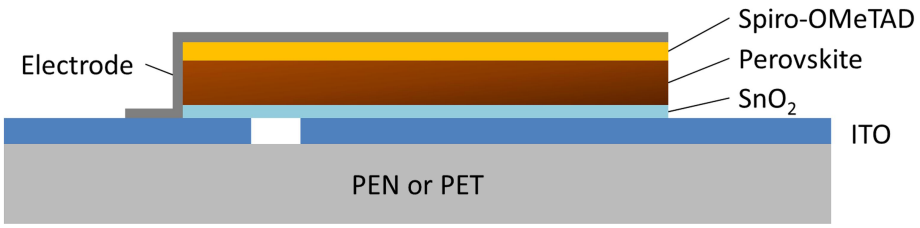

Figure 1. Schematic diagram of conventional structure of flexible perovskite solar cells fabricated.

Schematic diagram of detailed encapsulation methods was shown on Figure 2. The whole active area on top of polymer substrates including the electrodes are completely covered and encapsulated in both cases, i.e. 2 cm x 2cm COC film covered on 2 cm x 2 cm substrates. COC films was chosen as the barrier film because of its high transmission (>91% transmission through 2mm from 300 nm to 800 nm) and low water vapour permeability (0.023 g mm m^{-2} day^{-1}). Contact extensions from electrodes on devices were done by using tin-coated copper tapes cut into specific shape that guide electrodes outside of encapsulated devices. In addition, copper pin contacts are used to minimize the damage done on extended copper tapes for each measurement as electrode clips will damage the electrodes in long term stability testing. Silver paint should not be used as degradation of silver contact can be observed over time of stability testing in our previous designs.[26] During the whole stability test, no significant contact degradation was observed. Referring to Figure 2b, another encapsulation method was used as well with COC film laminated on the polymeric side of solar cells. Double sided barrier tapes were used as adhesive plus low WVTR films for protection surrounding whole device.

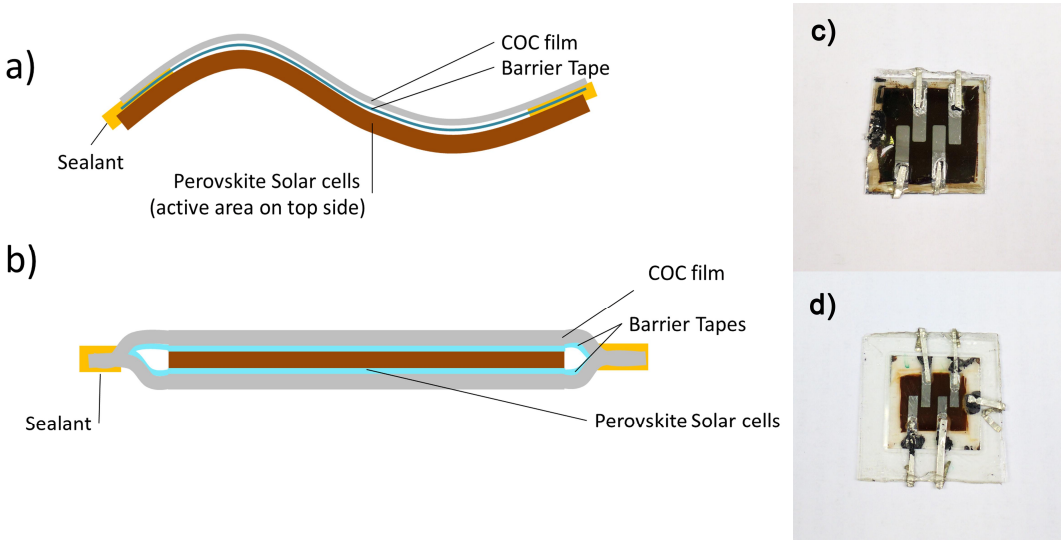

Figure 2. Schematic diagram of encapsulation for flexible solar cells. (a) COC film laminated on the active area side of devices with barrier tape. (b) devices sandwiched in between two COC films by using two barrier tapes. In both conditions, sealant was applied on edges of devices to complete the encapsulation. Photos of encapsulated devices based on method (a) and (b) are shown with (c) and (d) respectively.

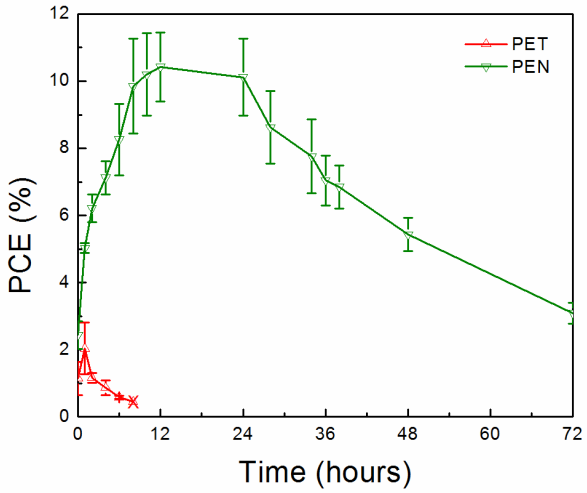

Figure 3. Power conversion efficiency of SnO_2 based devices on PET or PEN under continuous illumination at 25°C and 85% RH as a function of time. Devices on PEN survived 72 hours under continuous AM1.5G illumination.

Uunencapsulated devices with maximum 2 hours survival lifetime can be observed due to significant oxidation of aluminum electrode. For stability performance comparing SnO_2 based devices on PET or PEN, increase in efficiency can be observed in the beginning of test. Light soaking was reported to improve to the initial PCE performance of devices as illumination improve the crystallinity of perovskite layer.[27] Even though initial increase was observed, PCE will degrade gradually from peak to lower efficiency and then reached 0% efficiency immediately once the encapsulation failed completely for PET case. Devices on PEN survived 72 hours continuous AM1.5G illumination under ISOS-L-1 protocol. As shown on transmission graph on Figure 4(a), ITO/PEN is able to shield the solar cells from ultraviolet illumination of 370 nm and below, while ITO/PET allows transmission of light with 315 nm and above. As mentioned, UV illumination will accelerate degradation of perovskite, particularly under continuous illumination. On the other hand, PET has almost 3 times higher water vapour permeability than PEN, with 0.462 and 0.167 cc(STP) cm m^{-2} day^{-1} atm^{-1} respectively for PET and PEN.[22] Even though PET substrates used is 50 μm thicker than PEN, faster moisture infiltration and lack of UV blocking are expected to result in a shorter lifetime. Therefore, devices with PEN exhibited better stability and PEN was chosen for further deposition of different metal oxides on polymeric side.

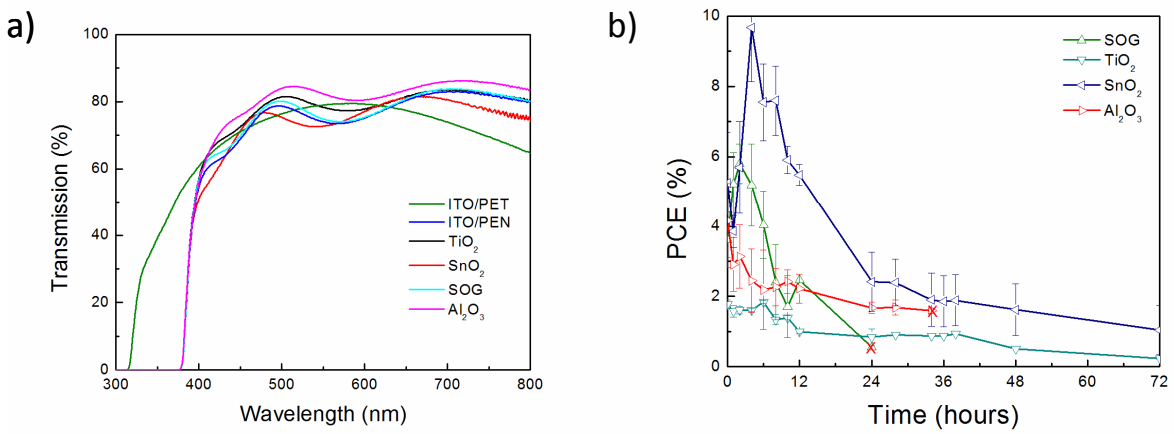

Figure 4. (a) UV-Vis transmission spectroscopy of ITO/PET, ITO/PEN and as deposited ALD TiO_2, SnO_2, Al_2O_3 and spin coated SOG on polymeric side of ITO/PEN. (b) Power conversion efficiency of SnO_2 based devices on different metal oxide coated PEN under continuous illumination at 25°C and 85% RH as a function of time. Devices on SnO_2 and TiO_2 coated PEN survived 72 hours continuous AM1.5G illumination.

After identifying better polymer substrate, we tried to identify better metal oxide for back side encapsulation. Different metal oxides were deposited on the polymeric side of ITO/PEN substrates before fabrication of devices. Transmission of substrates is kept almost identical to ITO/PEN for all conditions except for SnO_2 which have a slightly lower transmission on 400 nm. Under the same ISOS-L-1 protocol, longest time survived for Al_2O_3 and SOG is 34 and 24 hours respectively while Al_2O_3 and SnO_2 both survived 72 hours. Even though devices with Al_2O_3 coated survived, the efficiencies are much lower compared with SnO_2 coated counterparts. This may due to the fact that the ALD deposition of TiO_2 is not as optimized as SnO_2. Although there is significant increase in PCE in the first 6 hours, faster degradation was observed in the following 12 hours. Eventually significant slower degradation can be observed in prolonged stability testing.

As the devices with metal oxide deposition on polymeric sides showed no significant improvement on the stability performance of ITO/PEN, encapsulation incorporating two COC barrier films sandwiching solar cells was used. In figure 5, encapsulated device successfully survived for over 300 hours upon continuous AM1.5G illumination retaining 25% initial PCE. Similar PCE trend can be observed but with longer lifetime. The increase in PCE is mainly due to the increase of fill factor and V_{oc} at the beginning while PCE drop mainly depends on the slow decrease in J_{sc} over time. Even though PCE decreased after 300 hours of continuous illumination due to infiltration of moisture, the lifetime of SnO_2 based perovskite solar cells is extended with stable under high humidity condition with proper encapsulation.

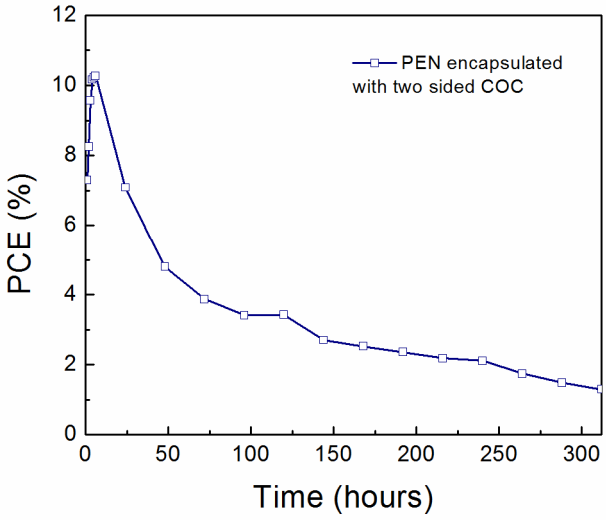

Figure 5. Power conversion efficiency of SnO_2 based devices PEN using second encapsulation sandwiched between two COC films under continuous illumination at 25°C and 85% RH as a function of time.

4. CONCLUSION

Different encapsulation methods were examined for flexible perovskite solar cells. All stability testing followed ISOS-L-1 with continuous AM1.5G illumination. Devices on PEN exhibited better stability compared to those on PET. Even though devices with ALD TiO_2 and SnO_2 coated on the polymeric side of devices survived 72 hours, no significant improvement of stability was observed. COC films laminated on both sides of solar cells survived more than 300 hours retaining 25% of initial PCE, while devices without encapsulation cannot survive more than 2 hours due to obvious Al electrode degradation. Lifetime of flexible devices with proper encapsulation was extended with stable despite perovskite sensitivity to ambient humidity under continuous illumination.

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