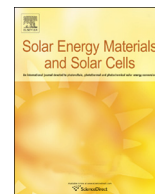




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## Reduction of moisture sensitivity of PbS quantum dot solar cells by incorporation of reduced graphene oxide

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## ABSTRACT

PbS nanocrystals are an important narrow-gap material for solar cells and photodetectors. Nevertheless, their application may be limited because device performance can be affected by atmospheric conditions. Indeed, the presence of oxygen and/or water can degrade the active layers, possibly leading to device failure. Strategies to address this issue are therefore actively explored. Here we report a solution-processed PbS quantum dot solar cell, consisting of a PbS-silane functionalized reduced graphene oxide (PbS-rGO) layer on top of the PbS absorber film, which enhances device stability, especially when the solar cells are exposed to moisture. Power conversion efficiency (PCE) measurements demonstrate a slower degradation under continuous illumination for solar cells with PbS-rGO. When storing the samples under saturated water vapor, differences are even more remarkable: with PbS-rGO the solar cells essentially maintain their initial PCE, while the PCE of the PbS reference devices is reduced by 50% after 5 days. Scanning electron microscopy, energy dispersive X-ray and X-ray photoelectron spectroscopy reveal the damage to the PbS films and the formation of PbSO<sub>4</sub> crystals in the PbS reference devices. Such crystals are not observed in the PbS-rGO devices, further supporting the importance of the PbS-rGO barrier layer.

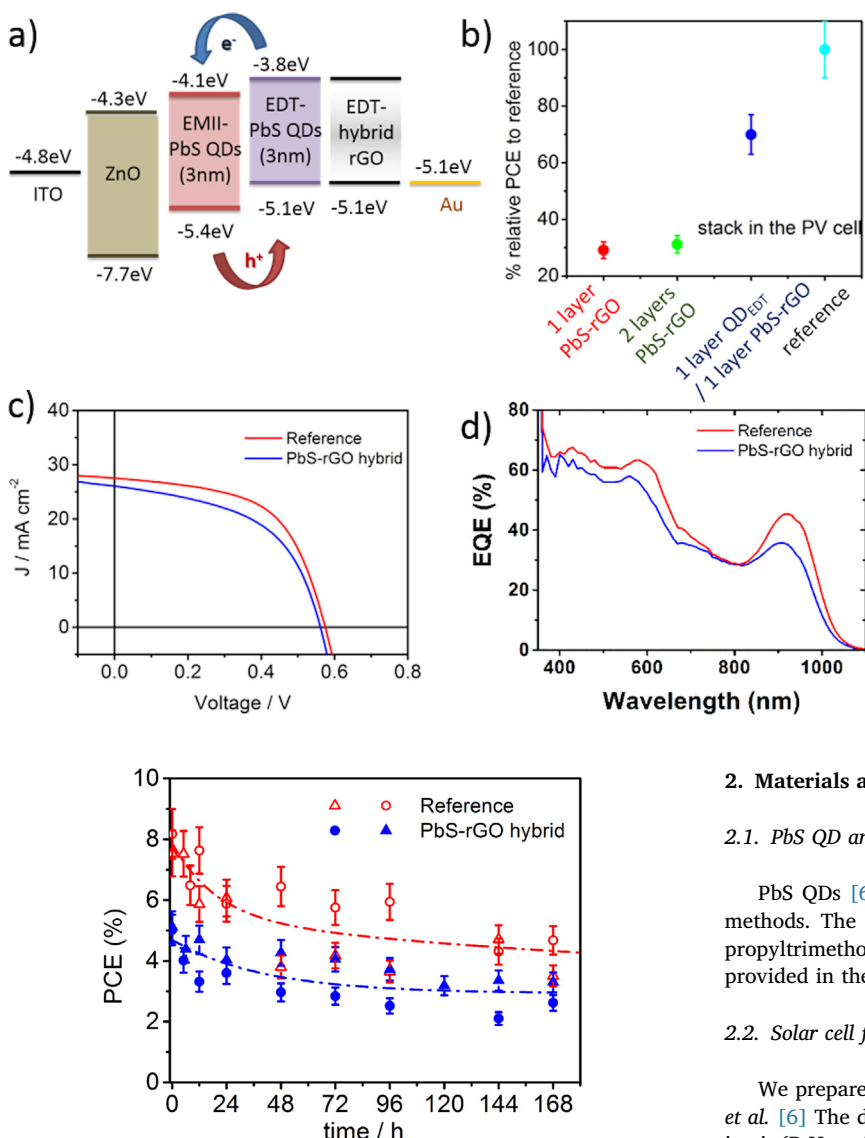
## 1. Introduction

Lead sulfide quantum dots (QDs) have demonstrated to be highly suitable materials for solution-processed solar cells, currently reaching power conversion efficiencies (PCE) up to 11.6% [1]. Fast, sensitive and broad-band near-infrared photodetectors based on PbS QDs have also been implemented in a variety of architectures [2]. However, the degradation of such devices under ambient conditions remains an important challenge for practical applications. In presence of oxygen, byproducts such as PbO, PbSO<sub>3</sub> or PbSO<sub>4</sub> can form and reduce the device performance [3,4]. Focusing on solar cells, several strategies have been proposed to address this issue, involving QD surface passivation with ligands [5,6], optimization of the device layer architecture [7,8], or device encapsulation [9]. Studies were mostly designed to improve the shelf life [7,10], or the stability under continuous illumination [3,6,11]. However, another important parameter, the relative humidity (R.H.) in ambient atmosphere, has not received similar attention so far. In several areas (excluding deserts or high altitudes), the long-term average R.H. can exceed 70% [12], hence values clearly

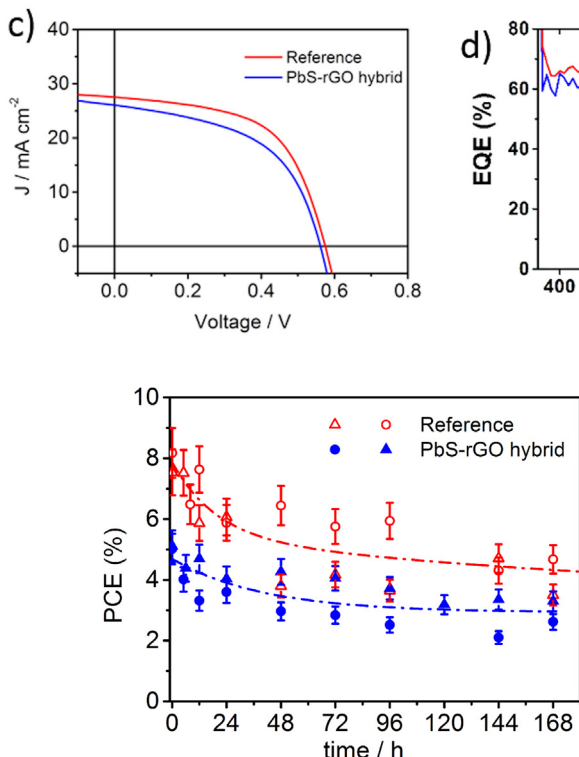
surpass typical laboratory conditions of 30–50%. Humidity has already shown to degrade solar cells based on perovskites [13,14], or organic materials [15,16], and therefore stability under humid conditions is an important issue that needs to be addressed.

Here, we increased the stability of PbS QD solar cells under both dry and humid atmosphere by adding a spin-coated top layer consisting of a PbS-reduced graphene oxide (PbS-rGO) hybrid material [17]. Previously, inclusion of graphene or graphene oxide has been shown to yield PbS solar cells with good power conversion efficiency (PCE) [18,19], although device stability was not investigated. However, organic (OPV) and perovskite-based photovoltaics have already experienced the advantages of rGO integration, with prolonged stability under ambient conditions. In OPV, even if the initial performance was not improved by rGO inclusion [20,21], the lifetime of the device can be increased by 50% under 100% R.H. conditions [21]. In the case of perovskites [22–24], studies were carried out in air with only ≤ 50% R.H. Already under these conditions, the endurance tests showed an efficient suppression of the material degradation. More specifically, when using spiro-rGO [22] the solar cells almost retained the initial

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**Fig. 1.** (a) Band alignment determined from UPS for the materials comprising the PbS/PbS-rGO solar cells. ITO, ZnO and Au values are taken from Brown et al. [28,29] (b) Relative PCE for the different layouts on top of EMII-exchanged PbS QD layers [see SM, Table S1]. The reference (cyan) has a 20 nm PbS<sub>EDT</sub> layer, while the other samples combine either: 1 layer (44 nm) of PbS-rGO (red), 2 layers of PbS-rGO (100 nm, green) and 1 layer PbS<sub>EDT</sub> (10 nm) + 1 layer PbS-rGO (44 nm, blue). (c) *J-V* curves obtained for the best reference (PCE 9.0%,  $J_{sc}$  27.51 mA cm<sup>-2</sup>,  $V_{oc}$  0.57 V and *FF* 0.57) and PbS-rGO (PCE 7.6%,  $J_{sc}$  26.02 mA cm<sup>-2</sup>,  $V_{oc}$  0.56 V and *FF* 0.52) devices after 30 days ageing. (d) The corresponding EQE curves for the same devices (freshly prepared): reference (PCE 7.7%,  $J_{sc}$  21.42 mA cm<sup>-2</sup>,  $V_{oc}$  0.56 V and *FF* 0.64) and PbS-rGO (PCE 6.7%,  $J_{sc}$  21.17 mA cm<sup>-2</sup>,  $V_{oc}$  0.56 V and *FF* 0.56).



**Fig. 2.** PCE evolution for different solar cells under continuous illumination (AM1.5) and ambient conditions. We prepared in total two substrates with PbS reference devices (red) and two with PbS-rGO (blue). On each substrate, we measured 3 devices. The dashed lines are exponential fits [ $PCE = C_0 + A_0 e^{-t/\tau}$ ] that yield the average behavior of both types of solar cells, with following decay times  $\tau$ : 38.2 h for the PbS reference and 40.8 h for PbS-rGO cells.

PCE after 120 h, while with PCBM-rGO [24] the PCE is reduced from 13.5% to 7% (52% retention) after 120 h. This in contrast with the reference cells, that passed from a PCE of 12% to 1% already after 50 h.

In this work we show that rGO inclusion in PbS QD solar cells yields a significant improvement in stability compared to corresponding PbS-only devices [6], even under 100% R.H. conditions. Our devices retained 96% of the initial PCE after 120 h (5 days) of continuous exposure to a saturated water vapor atmosphere, in stark contrast to the PbS-only reference cells, whose PCE nearly halved after the same treatment. Characterization of the PbS film morphology after exposure showed that the rGO reduces structural damage caused by humidity. Hence, our results hold great promise to stabilize the performance of PbS and other solution-processed solar cells, which can suffer similar degradation processes in presence of moisture.

## 2. Materials and methods

### 2.1. PbS QD and rGO synthesis

PbS QDs [6] and rGO [17] are prepared according to established methods. The PbS are coupled to the rGO sheets using 3-mercaptopropyltrimethoxysilane, as outlined in Ref. [17]. Further details are provided in the Supplementary material (SM).

### 2.2. Solar cell fabrication

We prepared the ITO/ZnO/QD<sub>EMII</sub>/QD<sub>EDT</sub> solar cells following Cao et al. [6] The device was constructed using layer-by-layer spin coating in air (R.H.  $\leq$  50%). For the ligand exchange on the PbS QD thin films, EMII (7 mg mL<sup>-1</sup> in MeOH) and EDT (0.02 vol% in ACN) solutions were used, respectively. The reference cell consisted of 12 PbS<sub>EMII</sub> layers and 2 PbS<sub>EDT</sub> layers. In the case of the PbS-rGO solar cell, we replaced the second PbS<sub>EDT</sub> layer (10 nm thick) with a PbS-rGO<sub>EDT</sub> layer (44 nm thick, determined by cross section SEM measurements, [SM, Fig. S1e and f]). To deposit this layer, the PbS QD-rGO dispersion was spin coated at 700 rpm for 120 s. Then, the film was covered by an EDT solution in ACN for 30 s before spin coating at 2500 rpm for 40 s. The substrate was rinsed three times by placing several drops of ACN, followed by spinning at 2500 rpm for 40 s to dry the film. All solar cells were stored in a N<sub>2</sub>-filled glove box overnight before thermal deposition of a 100 nm Au layer (deposition rate of 0.5 Å s<sup>-1</sup>) in a Kurt J. Lesker Nano 36 system (base pressure < 1.0 10<sup>-6</sup> mbar). The final devices, with an Au contact area of 0.03 cm<sup>2</sup>, were taken out from the evaporator and transferred into the glove box for annealing at 80 °C for 5 min.

### 2.3. Photovoltaic characterization of the devices

*J-V* curves were recorded in air using a Keithley 2400 source meter under 100 mW cm<sup>-2</sup> (AM1.5G) illumination, provided by a Newport sol3A (class AAA) solar simulator (freshly prepared devices) or a LOT-Oriel LSH601/LSZ163 solar simulator (stability tests). The light intensity was calibrated with a Newport 91150V reference cell in both cases. The external quantum efficiency (EQE) was measured with a

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