



Cite this: *Chem. Commun.*, 2024, 60, 1004

Received 28th November 2023,
Accepted 21st December 2023

DOI: 10.1039/d3cc05781j

rsc.li/chemcomm

Enhanced photocatalytic hydrogen evolution through MoS_2 quantum dots modification of bismuth-based perovskites†

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Efficient and cost-effective photocatalysts are pivotal for advancing large-scale solar hydrogen generation. Herein, we report a composite photocatalyst by incorporating MoS_2 quantum dots (MoS_2 QDs) as a cocatalyst into $\text{Cs}_3\text{Bi}_2\text{I}_9$, resulting in a high enhancement in photocatalytic performance. Remarkably, the optimum MoS_2 QDs/ $\text{Cs}_3\text{Bi}_2\text{I}_9$ composite achieves an impressive hydrogen evolution rate ($6.09 \text{ mmol h}^{-1} \text{ g}^{-1}$) in an ethanol and $\text{HI}/\text{H}_3\text{PO}_2$ mixed solution. This rate is 8.8 times higher than pristine $\text{Cs}_3\text{Bi}_2\text{I}_9$ ($0.69 \text{ mmol h}^{-1} \text{ g}^{-1}$) and notably surpasses $\text{Pt}/\text{Cs}_3\text{Bi}_2\text{I}_9$ ($2.47 \text{ mmol h}^{-1} \text{ g}^{-1}$). Moreover, the composite displays exceptional stability during an 18-hour reaction, showcasing its potential for sustainable photocatalytic hydrogen evolution.

The urgent need for sustainable energy due to extensive fossil fuel use has sparked interest in harnessing solar energy as a renewable solution.¹ Photocatalytic hydrogen evolution, a promising pathway for generating clean hydrogen from sunlight, faces challenges like efficiency, stability, and cost, underscoring the crucial importance of developing high-performance and cost-effective photocatalysts to drive innovation in this technology.^{2–5} Lead halide perovskites (LHPs) have gained significant attention as efficient photocatalysts in the past five years, exhibiting favorable visible light absorption, exceptionally-long charge-carrier diffusion length when employed for photocatalytic fuel production and chemical synthesis.^{6–11} However, the presence of

toxic lead (Pb) in LHPs severely limits their potential for practical application. Therefore, researchers are exploring alternatives like Sn and Bi metals to replace Pb within halide perovskite structures. Despite this, the introduction of Sn^{2+} often leads to instability, while Bi^{3+} shows promise due to its similar isoelectric state to Pb^{2+} and success in stabilizing bismuth-based halide perovskites.^{12–16} However, the severe charge recombination due to the high exciton binding energy of Bi-based perovskite photocatalysts, results in their poor photocatalytic activity. For example, Zhao *et al.* reported the $\text{MA}_3\text{Bi}_2\text{I}_9$ photocatalyst for photocatalytic hydrogen evolution reaction (HER) in the saturated HI solution, which initially exhibited low hydrogen generation activity ($12.19 \mu\text{mol h}^{-1} \text{ g}^{-1}$) and was subsequently improved ($169.21 \mu\text{mol h}^{-1} \text{ g}^{-1}$) with Pt as a cocatalyst.¹⁷ Nonetheless, its activity remains notably lower than that reported for LHP photocatalysts ($1\text{--}20 \text{ mmol h}^{-1} \text{ g}^{-1}$).

Recently, several non-noble metal co-catalysts, including rGO,¹² CoP,¹⁸ BP,⁹ Ni_3C ,¹⁰ and MoS_2 nanosheet,¹⁹ have been introduced to the LHP photocatalysts and the resulting composite demonstrated an impressive photocatalytic performance improvement. It is widely acknowledged that their beneficial impact stems from the creation of various heterojunctions between lead perovskites and the co-catalyst, which effectively enhances the electron–hole separation efficiency and augments photocatalyst carrier transport rates.^{20–22} Notably, although MoS_2 quantum dots (QDs) have proven to be a high-performance, sustainable, and cost-effective alternative to Pt for electrocatalytic and photocatalytic HER applications,^{23–25} their potential as a cocatalyst to enhance the photocatalytic HER activity of Bi-based perovskite photocatalysts remains unexplored.

Herein, we present a composite of $\text{Cs}_3\text{Bi}_2\text{I}_9$ perovskite microcrystals modified with MoS_2 QDs for efficient photocatalytic hydrogen generation *via* HI splitting in an HI/ethanol solution. The photocatalytic performance of the MoS_2 QDs/ $\text{Cs}_3\text{Bi}_2\text{I}_9$ composite demonstrated an impressive rate of $6.09 \text{ mmol h}^{-1} \text{ g}^{-1}$ for hydrogen evolution, surpassing pristine $\text{Cs}_3\text{Bi}_2\text{I}_9$ ($0.69 \text{ mmol h}^{-1} \text{ g}^{-1}$) by 8.8 times and $\text{Pt}/\text{Cs}_3\text{Bi}_2\text{I}_9$ ($2.47 \text{ mmol h}^{-1} \text{ g}^{-1}$) by 2.5 times. Notably,

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† Electronic supplementary information (ESI) available. See DOI: <https://doi.org/10.1039/d3cc05781j>



this activity level represents a new record among Bi-based perovskite photocatalysts under visible-light conditions. This heightened activity is attributed to the superior HER active sites provided by MoS₂ QDs as co-catalysts, combined with the unique crystal structure of Cs₃Bi₂I₉, ensuring the creation of MoS₂ QDs/Cs₃Bi₂I₉ heterojunctions of a type II nature.

We employed a modified solvothermal approach to synthesize high-quality Cs₃Bi₂I₉ microcrystals (method details in the supporting information).²⁶ Additionally, MoS₂ QDs were synthesized *via* a solvothermal treatment of bulk MoS₂ in isopropanol, followed by a sequence of centrifugal separation.²⁷ The MoS₂ QDs/Cs₃Bi₂I₉ heterostructure composites were obtained by ultrasonic mixing Cs₃Bi₂I₉ microcrystals into the isopropanol dispersion of MoS₂ QDs followed by solvent evaporation. The zeta potential of Cs₃Bi₂I₉ microcrystals and MoS₂ QDs is 10.57 and -9.39 mV, respectively (Fig. S1, ESI†). Thus, the negatively charged MoS₂ QDs were electrostatic self-assembly onto the surface of the positively charged Cs₃Bi₂I₉ after ultrasonication and solvent evaporation, as illustrated in Fig. 1a. The resulting composite contained MoS₂ QDs with a mass percentage of 5.4 wt%. Pristine Cs₃Bi₂I₉, as illustrated in Fig. S2 (ESI†), exhibited a regular hexagonal prism shape with sizes ranging from 500 nm to 1 μ m. The XRD pattern (Fig. S3, ESI†) confirms its

excellent crystallinity, with peak positions matching the standard perovskite structure of Cs₃Bi₂I₉ (JCPDS#23-0847). TEM images of MoS₂ QDs revealed their size to be between 2 nm and 5 nm (Fig. S4, ESI†), and XPS spectra illustrated the presence of both the 1T phase and 2H phase (Fig. S5, ESI†).²⁸ After incorporating MoS₂ QDs, the resulting MoS₂ QDs/Cs₃Bi₂I₉ composites maintained their hexagonal prism shape (Fig. 1b) and crystallinity. TEM and HRTEM images revealed tight attachment of MoS₂ QDs (yellow circles) to the surface of Cs₃Bi₂I₉ crystals (Fig. 1c and Fig. S6, ESI†).

In the inset image of Fig. 1d, Cs₃Bi₂I₉ exhibited a vibrant red color, while the color of MoS₂ QDs/Cs₃Bi₂I₉ was slightly deepened. Their UV-vis diffuse reflectance spectra (UV-vis-DRS) further indicated extended light absorption in the range of 600–800 nm due to the presence of MoS₂ QDs. In comparison to pristine Cs₃Bi₂I₉, the measured band gap of MoS₂ QDs/Cs₃Bi₂I₉ was reduced to 1.81 eV from the Kubelka-Munk plot (Fig. S7, ESI†). The presence of MoS₂ QDs in the Cs₃Bi₂I₉/MoS₂ QDs composite was further confirmed by XPS survey spectra, including the Bi 4f and I 3d regions (Fig. 1e, f and Fig. S8, ESI†). The high-resolution Bi 4f and I 3d peaks of MoS₂ QDs/Cs₃Bi₂I₉ shifted towards lower binding energies (0.26 eV for Bi 4f and 0.40 eV for I 3d), respectively, compared to pristine Cs₃Bi₂I₉. These shifts could be attributed to the strong interfacial interaction between MoS₂ QDs and Cs₃Bi₂I₉, as reported in previous studies.^{20–22,29}

The photocatalytic hydrogen evolution performance of MoS₂ QDs/Cs₃Bi₂I₉ (5 mg) and the comparison samples was measured in a 20 mL ethanol solution containing 0.2 mL of HI and 0.2 mL of H₃PO₂ under visible light irradiation ($\lambda > 420$ nm, 300-W Xe lamp). H₃PO₂ is used as a sacrificial reagent in the reaction system to reduce I₃⁻ ions to form I⁻. The generated H₂ increased linearly with irradiation time, reaching a peak hydrogen evolution rate of 6.09 mmol h⁻¹ g⁻¹ over a 3-hour reaction period. The apparent quantum efficiency (AQE) of MoS₂ QDs/Cs₃Bi₂I₉ for H₂ evolution at 500 nm was determined to be 4.71%.³⁰ When the MoS₂ QDs/Cs₃Bi₂I₉ composite was replaced with an equal amount of pristine Cs₃Bi₂I₉, while maintaining other experimental conditions, the average achieved H₂ evolution rate dramatically dropped to 0.69 mmol h⁻¹ g⁻¹. This observation indicates that the enhanced photocatalytic activity of MoS₂ QDs/Cs₃Bi₂I₉ originates from the incorporation of MoS₂ QDs. Furthermore, Pt-loaded Cs₃Bi₂I₉ (Pt/Cs₃Bi₂I₉) and bulk MoS₂-modified Cs₃Bi₂I₉ exhibited H₂ evolution rates of 2.47 (Fig. S9, ESI†) and 1.93 mmol h⁻¹ g⁻¹, respectively, which were notably lower than that of MoS₂ QDs/Cs₃Bi₂I₉, highlighting the superior cocatalytic performance of MoS₂ QDs over Pt and bulk MoS₂. Consequently, the photocatalytic HER activity of MoS₂ QDs/Cs₃Bi₂I₉ is over 8.8 and 2.4 times higher than that of Cs₃Bi₂I₉ and Pt/Cs₃Bi₂I₉, respectively. As illustrated in Fig. 2c, the effect of different mass percentages of MoS₂ QDs in the MoS₂ QDs/Cs₃Bi₂I₉ composite was investigated. Notably, the incorporation of just 0.9 wt% MoS₂ QDs into Cs₃Bi₂I₉ led to a significant increase in the H₂ evolution rate to 3.67 mmol h⁻¹ g⁻¹. With higher MoS₂ QDs loading, the H₂ evolution rate progressively increased, peaking at 6.09 mmol h⁻¹ g⁻¹ for the MoS₂ QDs/Cs₃Bi₂I₉ composite with

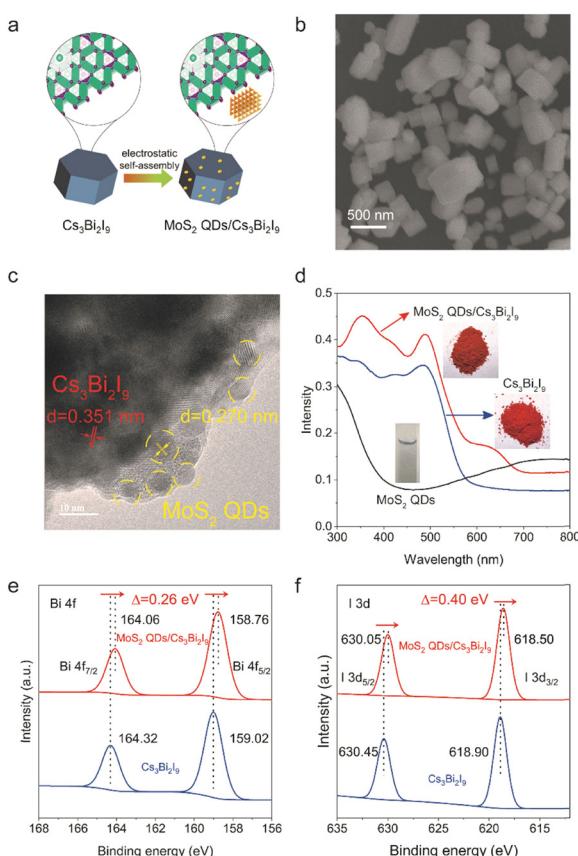


Fig. 1 (a) Schematic illustration of synthetic route for the MoS₂ QDs/Cs₃Bi₂I₉. (b) SEM images of MoS₂ QDs/Cs₃Bi₂I₉. (c) TEM image of the MoS₂ QDs/Cs₃Bi₂I₉. (d) UV-vis diffuse reflectance spectra of Cs₃Bi₂I₉, MoS₂ QDs, and MoS₂ QDs/Cs₃Bi₂I₉ (inset: product pictures) (e) and (f) XPS spectra of Bi 4f and I 3d region of Cs₃Bi₂I₉ and MoS₂ QDs/Cs₃Bi₂I₉.



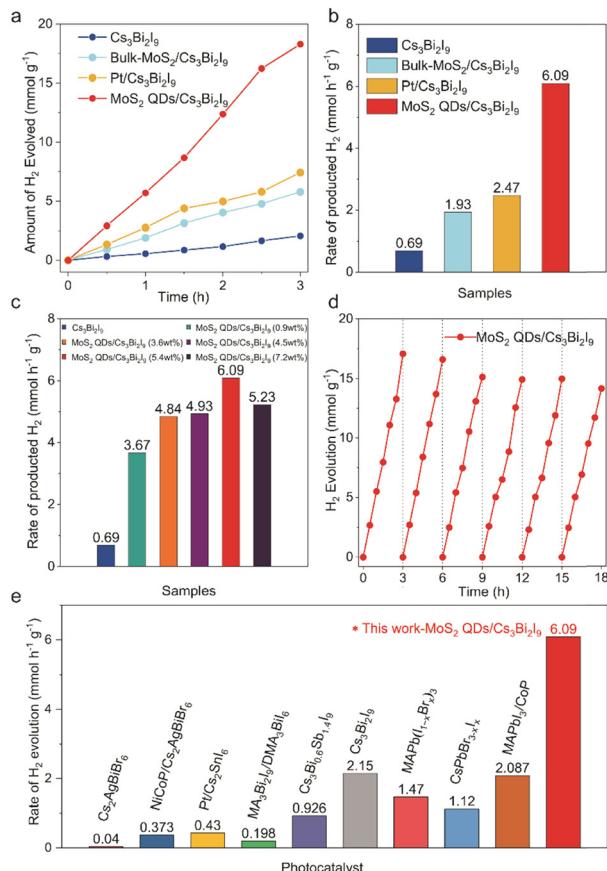


Fig. 2 (a) Time courses of photocatalytic H₂ evolution over Cs₃Bi₂I₉, bulk-MoS₂/Cs₃Bi₂I₉, Pt/Cs₃Bi₂I₉, MoS₂ QDs/Cs₃Bi₂I₉ ($\lambda > 420$ nm). (b) The H₂ generation rate of Cs₃Bi₂I₉, bulk-MoS₂/Cs₃Bi₂I₉, Pt/Cs₃Bi₂I₉, MoS₂ QDs/Cs₃Bi₂I₉. (c) The H₂ generation rate of MoS₂ QDs/Cs₃Bi₂I₉ loading different amounts of cocatalyst (0.9, 3.6, 4.5, 5.4, 7.2 wt%). (d) Cycling measurements of photocatalytic H₂ production of MoS₂ QDs/Cs₃Bi₂I₉ ($\lambda > 420$ nm). (e) Comparison of the hydrogen evolution performance of reported perovskite-based catalysts and this work.

5.4 wt% MoS₂ QDs. However, at 7.2 wt% MoS₂ QDs, the photocatalytic HER activity showed a decline.

In addition to its impressive photocatalytic hydrogen evolution activity, the MoS₂ QDs/Cs₃Bi₂I₉ composite also exhibited remarkable stability (Fig. 2d). Throughout six cycles of 3-hour experiments, the rate of photocatalytic hydrogen evolution remained nearly constant. XRD patterns displayed no alterations in the crystalline phase of the MoS₂ QDs/Cs₃Bi₂I₉ composite after an 18-hour reaction, and SEM analysis confirmed the preservation of its morphology (Fig. S10, ESI[†]). Furthermore, the MoS₂ QDs/Cs₃Bi₂I₉ composite outperformed recently reported Bi-based perovskite photocatalysts and some of Pb-based perovskite photocatalysts in terms of photocatalytic hydrogen evolution (Fig. 2e and Table. S1, ESI[†]).^{7,13,23-25,29,31,32}

Both electrochemical and photoelectrochemical experiments were employed to elucidate the significant effect of MoS₂ QDs in enhancing the photocatalytic hydrogen evolution activity of Cs₃Bi₂I₉. From Fig. 3a, it can be found that the electrocatalytic HER onset potential of MoS₂ QDs could reach

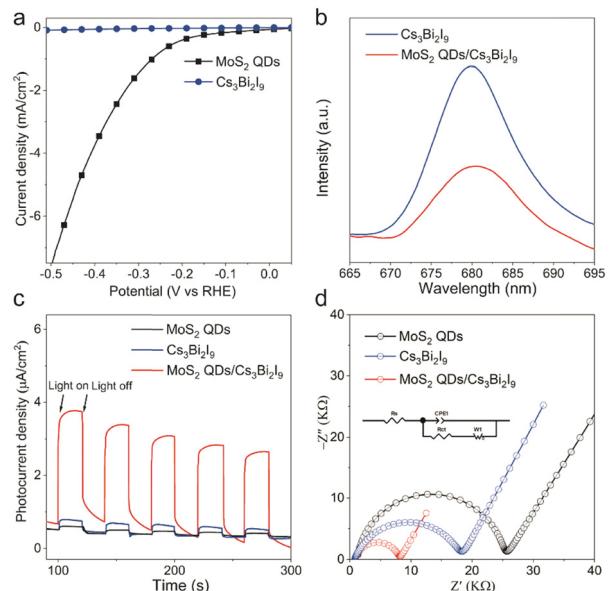


Fig. 3 (a) Electrochemical HER polarization curves of Cs₃Bi₂I₉ and MoS₂ QDs loaded carbon paper recorded in HI solution. (b) PL spectra of Cs₃Bi₂I₉ and MoS₂ QDs/Cs₃Bi₂I₉. (c) Transient photocurrent responses of Cs₃Bi₂I₉, MoS₂ QDs and MoS₂ QDs/Cs₃Bi₂I₉ recorded in 0.1 M tetrabutylammonium hexafluorophosphate ethanol solution under light irradiation. (d) EIS Nyquist plots of Cs₃Bi₂I₉ and MoS₂ QDs/Cs₃Bi₂I₉ under light irradiation.

~ -0.2 V vs. RHE indicating a superior HER activity of MoS₂ QDs compared with pristine Cs₃Bi₂I₉. Moreover, the photoluminescence (PL) emission intensity of MoS₂ QDs/Cs₃Bi₂I₉ exhibited a notable reduction compared to that of pristine Cs₃Bi₂I₉ (Fig. 3b). Time-resolved photoluminescence (TRPL) results show that the average lifetime for MoS₂ QDs/Cs₃Bi₂I₉ is greater than Cs₃Bi₂I₉ (Fig. S11, ESI[†]). This suggests that the association with MoS₂ QDs significantly enhances the separation efficiency of electrons and holes within Cs₃Bi₂I₉, thereby substantially boosting the photocatalytic HER efficacy of MoS₂ QDs/Cs₃Bi₂I₉. Notably, the MoS₂ QDs/Cs₃Bi₂I₉ composite demonstrated the highest photocurrent, directly correlating with its most favorable catalytic H₂ evolution activity (Fig. 3c). Furthermore, for a deeper understanding of the improved HER activity of the photocatalyst, we employed electrochemical impedance spectroscopy (EIS) to characterize and analyze the opto-electronic performance of the photocatalysts in terms of the separation and transfer of photogenerated charges. EIS analysis revealed that the MoS₂ QDs/Cs₃Bi₂I₉ composite displayed significantly lower charge transfer resistance compared to pristine Cs₃Bi₂I₉ (Fig. 3d).

Based on the ultraviolet photoelectron spectroscopy (UPS), we obtained E_{VB} and E_{CB} values of 1.1 V (NHE), -0.92 V (NHE) and 3.54 V (NHE), -0.11 V (NHE) for Cs₃Bi₂I₉ and MoS₂ QDs, respectively (Fig. S12 and S13, ESI[†]). The energy diagram of the MoS₂ QDs/Cs₃Bi₂I₉-based hydrogen generation system through HI splitting is illustrated in Fig. 4.^{10,18,19,33} According to the UPS calculation method, we obtained the Fermi levels (E_f) values of 0.12 V for Cs₃Bi₂I₉ and 0.52 V for MoS₂ QDs. A type II heterojunction forms at the interface of MoS₂ QDs and

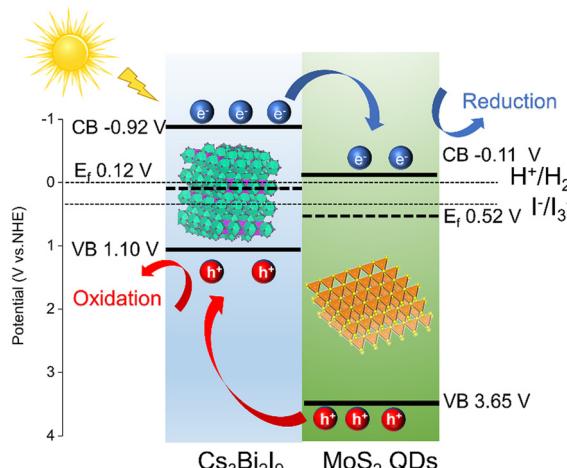


Fig. 4 Schematic band diagram of $\text{Cs}_3\text{Bi}_2\text{I}_9$ and MoS_2 QDs for photo-catalytic HI splitting reactions.

$\text{Cs}_3\text{Bi}_2\text{I}_9$, leading to efficient migration of photogenerated electrons to MoS_2 QDs, thus facilitating the effective separation of photogenerated carriers. Simultaneously, these photogenerated electrons on the surface of MoS_2 QDs diffuse toward active sites where protons undergo efficient reduction to generate H_2 . These heterojunctions play a pivotal role in promoting the separation of charge carriers, thereby contributing significantly to the remarkable enhancement in activity.

In summary, the incorporation of MoS_2 QDs as cocatalysts has been demonstrated to significantly enhance the photocatalytic performance of $\text{Cs}_3\text{Bi}_2\text{I}_9$ for hydrogen evolution. The optimized MoS_2 QDs/ $\text{Cs}_3\text{Bi}_2\text{I}_9$ composite showcases an impressive 8.8-fold increase in the rate of H_2 evolution compared to the unmodified $\text{Cs}_3\text{Bi}_2\text{I}_9$. This study underscores the potential of harnessing zero-dimensional MoS_2 QDs as remarkably efficient cocatalysts, to enhance the efficacy of the photocatalytic HER in Bi-based perovskite photocatalysts. Furthermore, the insights gleaned from this research pave the way for the development of exceptionally active photocatalysts based on lead-free perovskites.

This work was financially supported by the National Natural Science Foundation of China (52102197, 52102325), Excellent Research and Innovation Team Project of Anhui Province (2023AH010001) and Collaborative Innovation Program of Hefei Science Center, CAS (2022HSC-CIP016).

Conflicts of interest

There are no conflicts to declare.

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