# Supporting Information

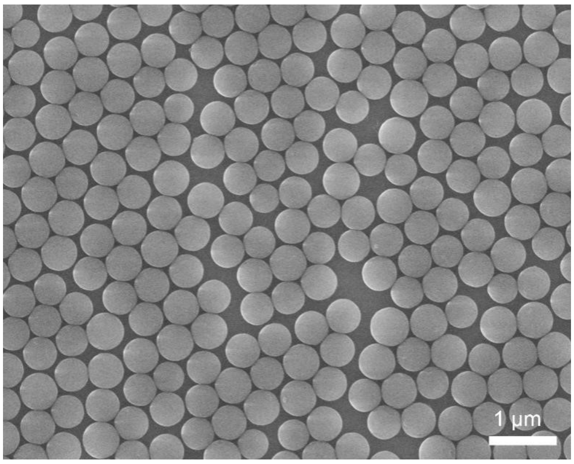


**Fig. S1.** SEM image of $SiO_2$ spheres.

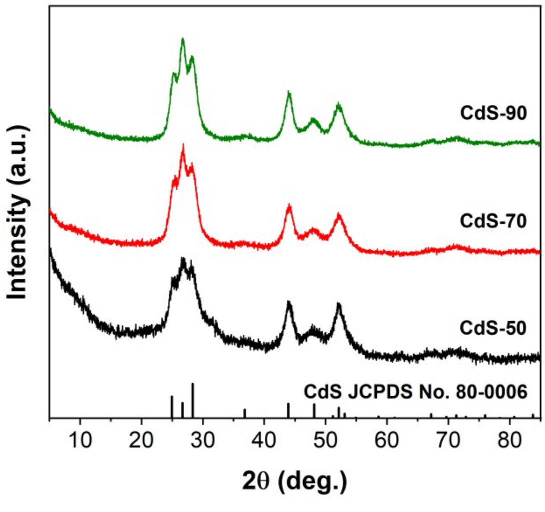


**Fig. S2.** XRD patterns of CdS hollow spheres prepared with different reaction temperatures.

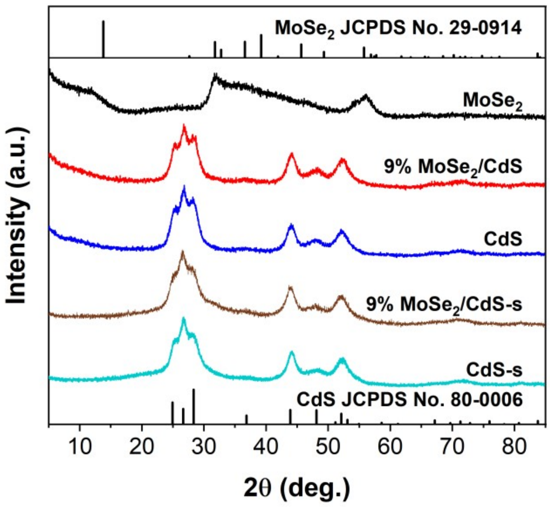


**Fig. S3.** XRD patterns of different samples.

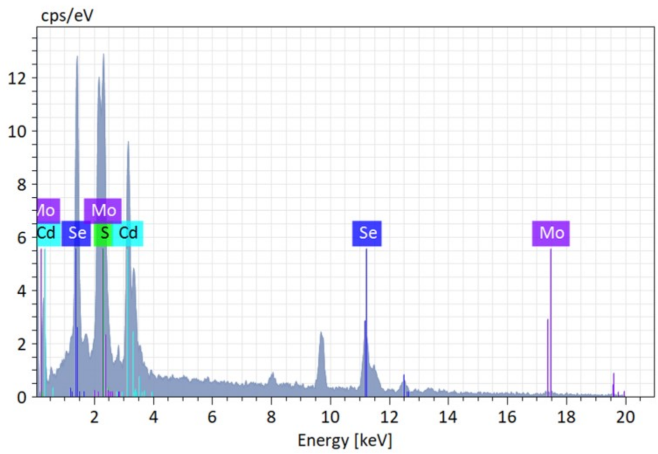


**Fig. S4.** EDX spectrum of 9% $MoSe_2$/CdS composite.

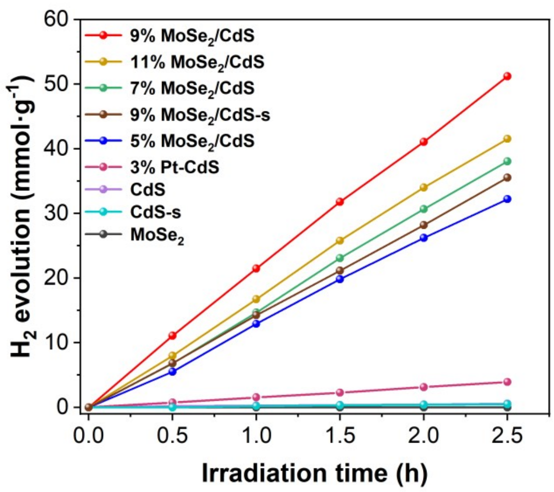


**Fig. S5.** PHE activities of different samples.

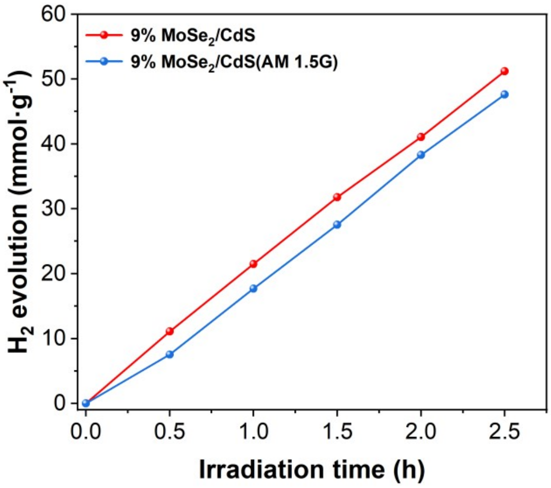


**Fig. S6.** PHE activities of 9% $MoSe_2$/CdS measured with 400-nm cut-off filter and AM 1.5G filter.

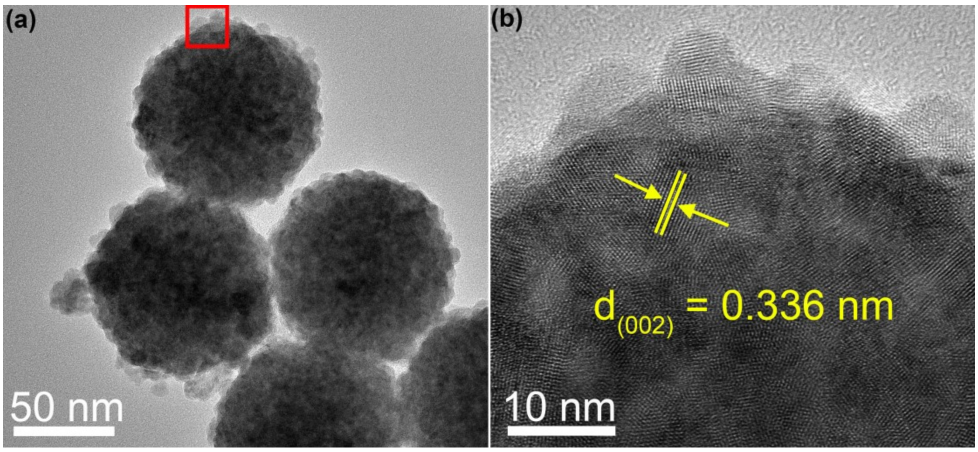


**Fig. S7.** (a) TEM and (b) HRTEM graphs of CdS-s. (b) is amplified from the marked area in (a).

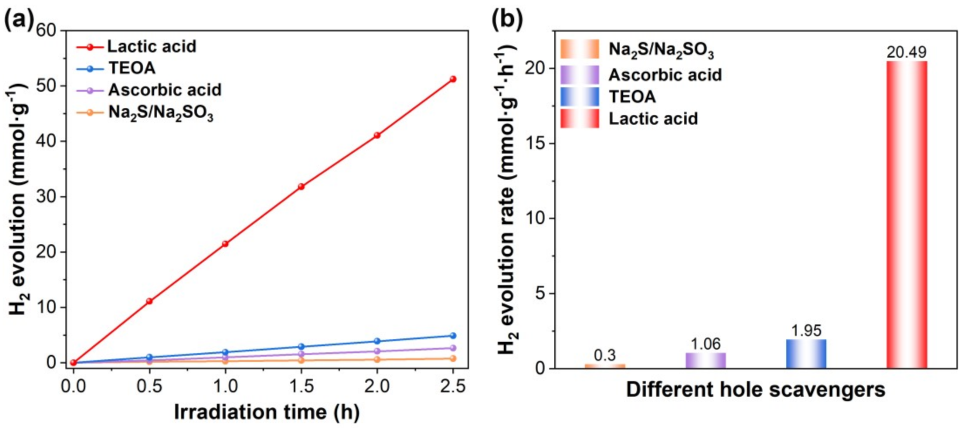


**Fig. S8.** Average PHE rates of 9% $MoSe_2$/CdS measured using different hole scavengers.

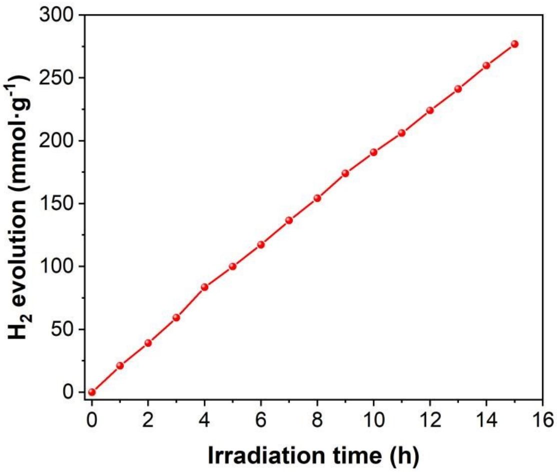


**Fig. S9.** Long-term PHE stability of 9% $MoSe_2$/CdS.

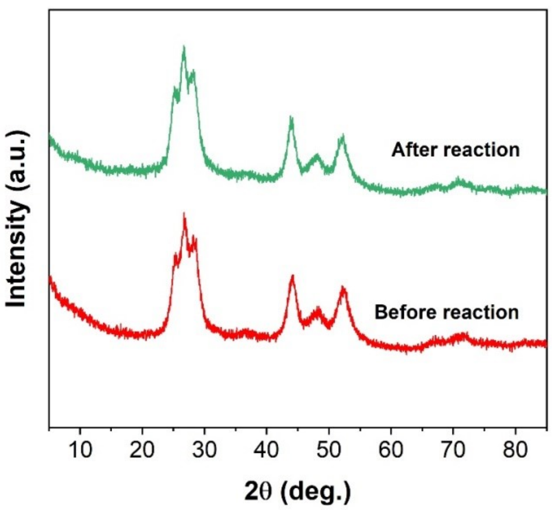


**Fig. S10.** XRD patterns of 9% $MoSe_2$/CdS before and after catalytic test.

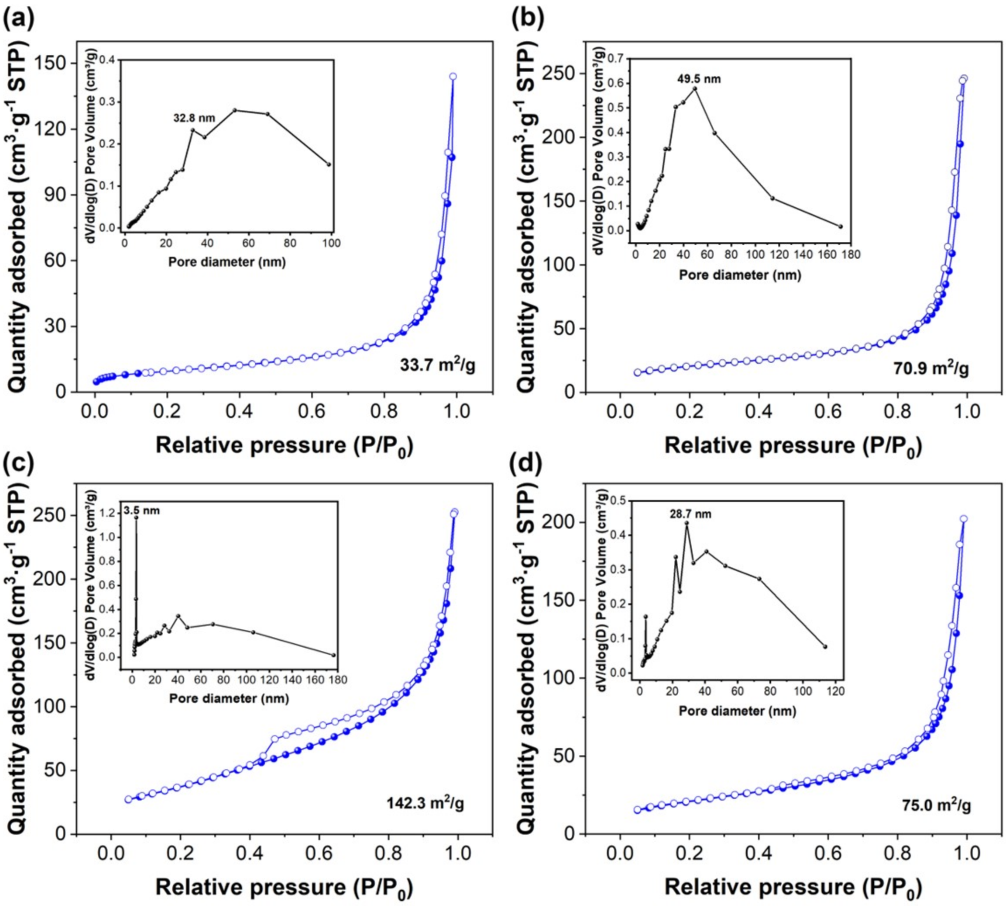


**Fig. S11.** $N_2$ adsorption-desorption isotherms and pore-size distribution of (a) CdS-s, (b) CdS, (c) $MoSe_2$, and (d) 9% $MoSe_2$/CdS.

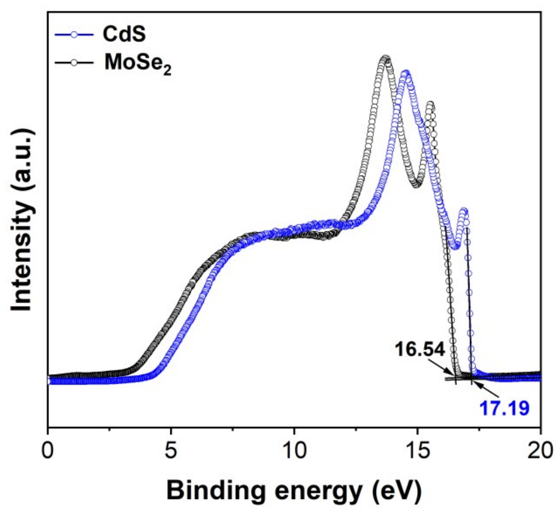


**Fig. S12.** UPS spectra of CdS and $MoSe_2$.

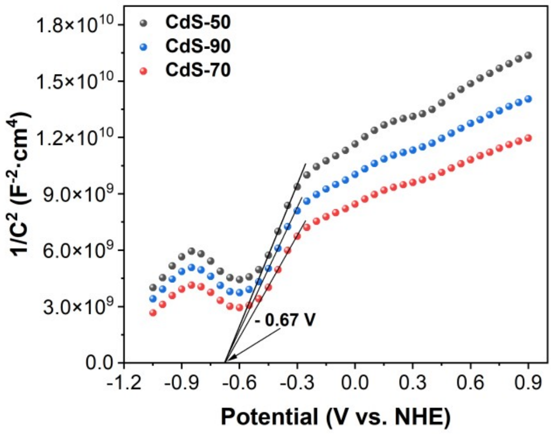


**Fig. S13.** Mott–Schottky curves of CdS hollow spheres with differing synthesis temperatures.

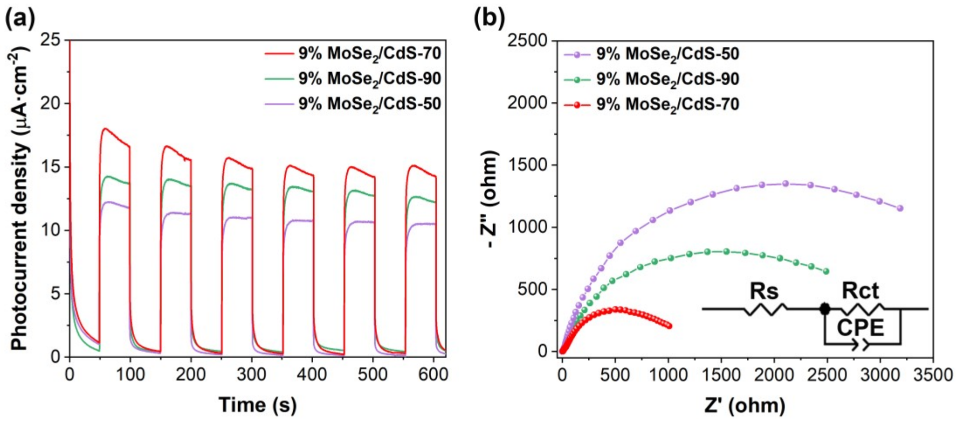


**Fig. S14.** (a) Photocurrent densities and (b) EIS Nyquist curves of 9% $MoSe_2$/CdS-50, 9% $MoSe_2$/CdS-70, and 9% $MoSe_2$/CdS-90.

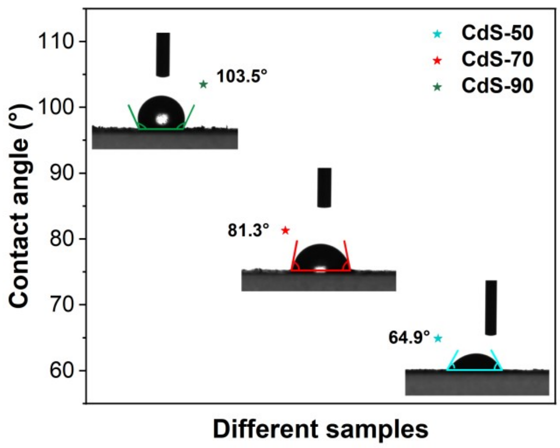


**Fig. S15.** Static water contact angles of CdS hollow spheres with different formation temperatures.

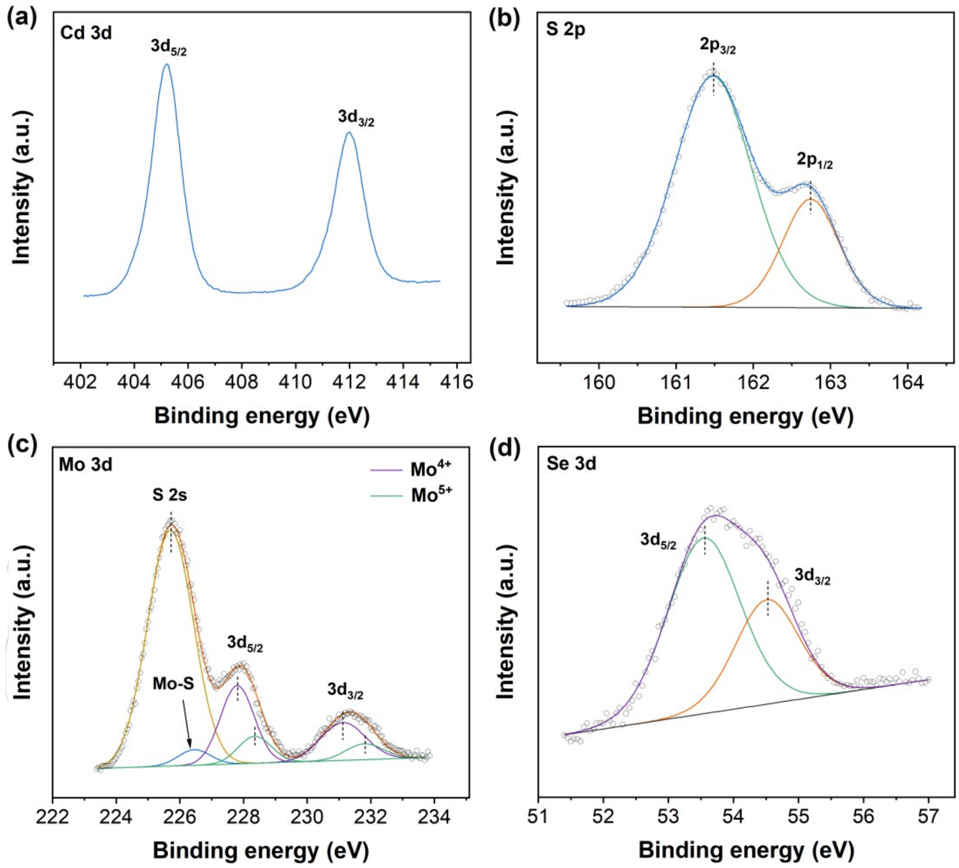


**Fig. S16.** XPS spectra of (a) Cd, (b) S, (c) Mo, and (d) Se elements of 9% $MoSe_2$/CdS after catalytic test.

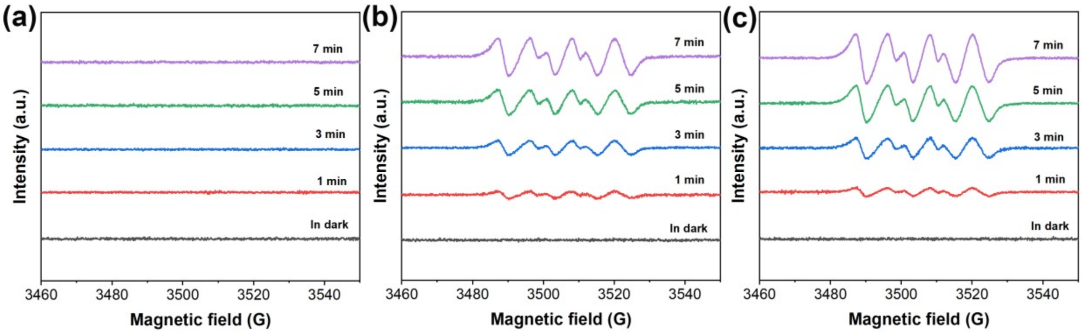


**Fig. S17.** DMPO-$\cdot O_2^-$ EPR signals of (a) $MoSe_2$, (b) CdS, and (c) 9% $MoSe_2$/CdS detected at certain irradiation periods.

**Table S1.** PHE activity comparison on our $MoSe_2$/CdS heterojunction as well as CdS- and $MoSe_2$-based photocatalysts documented in literatures.

| Photocatalyst | Hole scavenger (aqueous solution) | Light source (Xe lamp) | Maximum rate ($mmol\cdot g^{-1}\cdot h^{-1}$) | AQY (420 nm) | Reference |
|---|---|---|---|---|---|
| **$MoSe_2$/CdS** | Lactic acid | $\lambda > 400$ nm | 20.49 | 10.36% (475 nm) | This work |

| | | | | | |
|---|---|---|---|---|---|
| **CdS/Ni-based MOF** | Lactic acid | AM 1.5G filter | 4.5 | 6.36%<br>11.83% (365 nm) | 1 |
| **$Cu_{2-x}S$/CdS** | $Na_2S/Na_2SO_3$ | $\lambda \geq 420$ nm | 5.75 | 0.8% | 2 |
| **Pt, Au/CdS** | TEOA | $\lambda > 380$ nm | 6.71 | 3.72% (380 nm) | 3 |
| **$NiPS_3$/CdS** | Lactic acid | AM 1.5G filter | 18.24 | 12.11% (475 nm) | 4 |
| **$ZnIn_2S_4$/CdS** | $Na_2S/Na_2SO_3$ | $\lambda > 400$ nm | 5.68 | 4.8% | 5 |
| **H-$CeO_2$/H-CdS** | Lactic acid | $\lambda \geq 420$ nm | 8.36 | 56.5% | 6 |
| **CdS/Pt/$NaTaO_3$** | Lactic acid | AM 1.5G filter | 19.84 | 7.9% (350 nm) | 7 |
| **Au/$ZnWO_4$/CdS** | $Na_2S/Na_2SO_3$ | 420-800 nm | 5.48 | 2.62% (450 nm) | 8 |
| **0D/2D CdS/$MoO_{3-x}$** | Lactic acid | 350-W Xe lamp | 7.44 | 8.8%<br>14.3% (450 nm) | 9 |
| **Cd/CdS** | $Na_2S/Na_2SO_3$ | $\lambda > 420$ nm | 10.6 | 12.1% | 10 |
| **CdS/rGO** | $Na_2S/Na_2SO_3$ | 420-760 nm | 2.17 | - | 11 |
| **CdS@$TiO_2$/Mxene** | Lactic acid | - | 16.2 | - | 12 |
| **$PtP_2$@C/CdS** | Lactic acid | 3-W 420-nm<br>LED lamps | 9.76 | 41.67% | 13 |
| **CdS-$MoS_2$-$CoO_x$** | TEOA | 300-W Xe lamp | 7.4 | 7.6% | 14 |
| **CdS/WC** | Lactic acid | $\lambda \geq 420$ nm | 9.18 | 14.3% | 15 |
| **CdS/graphene QDs/Pt single atoms** | Lactic acid | $\lambda \geq 420$ nm | 13.49 | 35.5% | 16 |
| **1D CdS/2D MXene** | Lactic acid | $\lambda > 420$ nm | 8.87 | 27.8% (450 nm) | 17 |
| **$MA_{1-x}FA_xPbI_3$/$MoSe_2$** | perovskite-saturated | AM 1.5G filter | 12.53 | 10.29% | 18 |
| **$Cd_{0.7}Zn_{0.3}S$/$MoSe_2$** | $Na_2S/Na_2SO_3$ | $\lambda \geq 420$ nm | 0.67 | 1.96% (450 nm) | 19 |
| **$MoSe_2$/N-doped C/$ZnIn_2S_4$** | TEOA | $\lambda > 400$ nm | 10.31 | - | 20 |
| **CdS/$MoSe_2$/UiO-66-$NH_2$** | TEOA | 500-W Xe lamp | 0.426 | - | 21 |
| **$MoSe_2$/$Mn_{0.5}Cd_{0.5}S$** | $Na_2S/Na_2SO_3$ | $\lambda > 420$ nm | 5.6 | 67.04%<br>71.16% (400 nm) | 22 |
| **$MoSe_2$/g-$C_3N_4$** | TEOA | $\lambda > 400$ nm | 4.0 | - | 23 |
| **$Ta_3N_5$/$MoSe_2$** | $Na_2S/Na_2SO_3$ | 300-W Xe lamp | 0.81 | - | 24 |
| **$TiO_2$/$MoSe_2$** | Lactic acid | 300-W Xe lamp | 0.69 | - | 25 |
| **$MoSe_2$/CdS** | Lactic acid | $\lambda \geq 420$ nm | 4.7 | 15.6% (450 nm) | 26 |
| **CdS-$Cu_{1.81}S$** | $Na_2S/Na_2SO_3$ | 300-W Xe lamp | 2.71 | 11.32% | 27 |
| **$Ag_2S$/CdS/$Cd_2SO_4(OH)_2$** | $Na_2S/Na_2SO_3$ | 300-W Xe lamp | 9.69 | 7.60% | 28 |
| **CdS-Pd** | TEOA | 300-W Xe lamp | 0.95 | 33.92%<br>27.49% (500 nm) | 29 |
| **Pt single sites-$ZnIn_2S_4$** | TEOA | $\lambda > 420$ nm | 17.5 | 50.4% | 30 |
| **CoP@$ZnIn_2S_4$** | TEOA | $\lambda > 420$ nm | 7.4 | 7.6% | 31 |
| **Mo-$ZnIn_2S_4$@$NiTiO_3$** | TEOA | $\lambda > 420$ nm | 14.06 | 44.1% | 32 |
| **$ZnIn_2S_4$/P-doped Co/C** | TEOA | $\lambda > 420$ nm | 2.1 | - | 33 |
| **$FeS_2$@$ZnIn_2S_4$** | TEOA | AM 1.5G filter | 5.05 | 13.4% | 34 |
| **Zn-doped $Ni_2P$/g-$C_3N_4$** | TEOA | AM 1.5G filter | 1.077 | 8.9% | 35 |
| **F/C co-doped g-$C_3N_4$** | TEOA | $\lambda > 420$ nm | 3.87 | 15.4% | 36 |
| **B-doped g-$C_3N_4$/Ni-** | TEOA | 60-W 420-nm | 2.19 | 1.8% | 37 |

| | | | | | |
|---|---|---|---|---|---|
| **MOF-74** | | LED lamp | | | |
| **Cu-coordinated g-$C_3N_4$** | TEOA | $\lambda > 420$ nm | 6.53 | - | [38] |
| **NiS/g-$C_3N_4$** | TEOA | 420-nm LED lamps | 0.244 | - | [39] |

**Table S2.** Fitting parameters for EIS Nyquist plots of $MoSe_2$, CdS, and 9% $MoSe_2$/CdS. (Rs: series resistance, Rct: charge transfer resistance, CPE: constant phase angle element)

| Sample | Rs (Ω) | Rct (Ω) | CPE-T (F) | CPT-P |
|---|---|---|---|---|
| CdS | 20.56 | 10125 | $1.64 \times 10^{-4}$ | 0.808 |
| $MoSe_2$ | 29.22 | 7149 | $1.33 \times 10^{-4}$ | 0.825 |
| 9% $MoSe_2$/CdS-70 | 28.42 | 889 | $1.73 \times 10^{-4}$ | 0.809 |
| 9% $MoSe_2$/CdS-90 | 22.82 | 3007 | $1.13 \times 10^{-4}$ | 0.877 |
| 9% $MoSe_2$/CdS-50 | 22.07 | 4342 | $1.04 \times 10^{-4}$ | 0.868 |

**Table S3.** Average diameters (calculated by Scherrer's formula), specific surface areas, and static water contact angles of CdS with differing preparation temperatures.

| Sample | Average diameter (nm) | Specific surface area ($m^2$/g) | Water contact angle ($^o$) |
|---|---|---|---|
| CdS-50 | 4.7 | 74.1 | 64.9 |
| CdS-70 | 4.8 | 70.9 | 81.3 |
| CdS-90 | 5.1 | 68.4 | 103.5 |

**Table S4.** XPS peak fitting data for $MoSe_2$, CdS, and 9% $MoSe_2$/CdS with and without light irradiation.

| Species | $MoSe_2$ | CdS | $MoSe_2$/CdS | light-irradiated $MoSe_2$/CdS |
|---|---|---|---|---|
| **Mo-S** | - | - | 226.4 eV | 226.7 eV |
| **$Mo^{4+}$** | 228.13-231.23 eV | - | 227.80-231.13 eV | 227.93-231.18 eV |
| **$Mo^{5+}$** | 229.1-232.3 eV | - | 228.4-231.9 eV | 228.68-232.14 eV |
| **$Se^{2-}$** | 53.85-54.7 eV | - | 53.6-54.5 eV | 53.8-54.7 eV |
| **$Cd^{2+}$** | - | 404.7-411.4 eV | 405.3-412.0 eV | 405.0-411.7 eV |
| **$S^{2-}$** | - | 161.2-162.4 eV | 161.6-162.8 eV | 161.4-162.6 eV |

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