

Supporting Information

Exposing high-activity (111) facets CoO octahedral Loading

MXene Quantum dots of Efficient

and Stable Photocatalytic H₂ Evolution

Lan Ding*, Siyang Wang, Yaoyao Tang, Xinyi Chen, Hongjun Zhou*

State Key Laboratory of Heavy Oil Processing Beijing Key Laboratory of Biogas

Upgrading Utilization, China University of Petroleum-Beijing, Beijing 102249, China

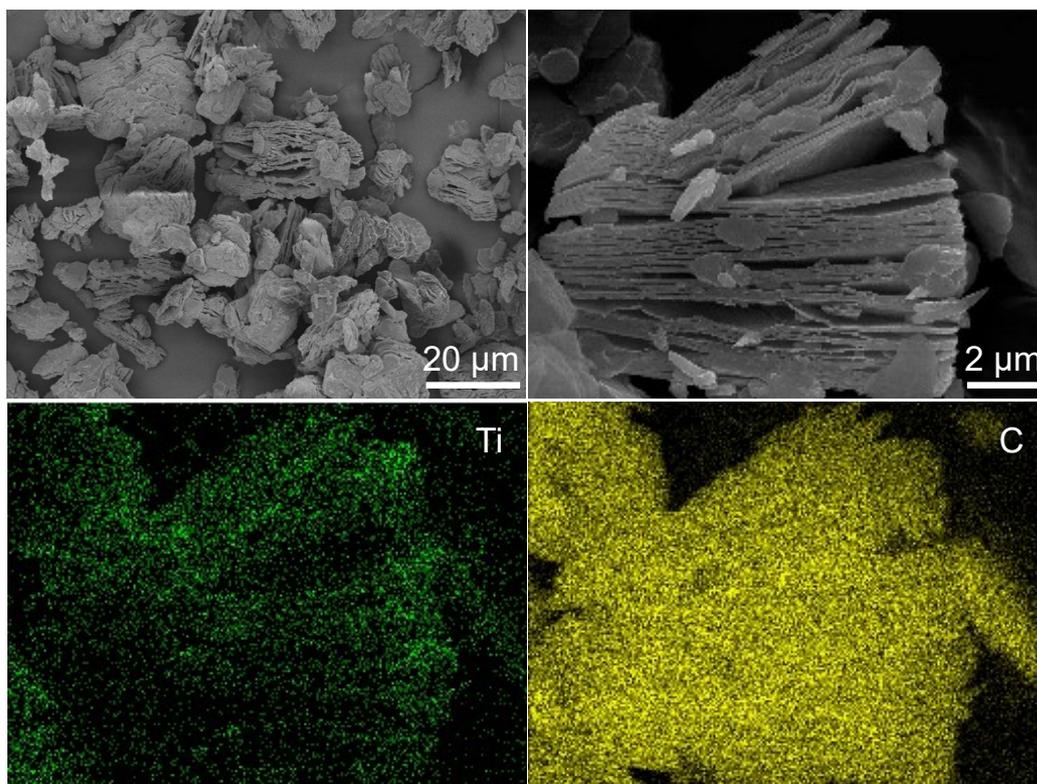


Fig. S1. SEM image and elements scanning for Ti₃C₂ MXene.

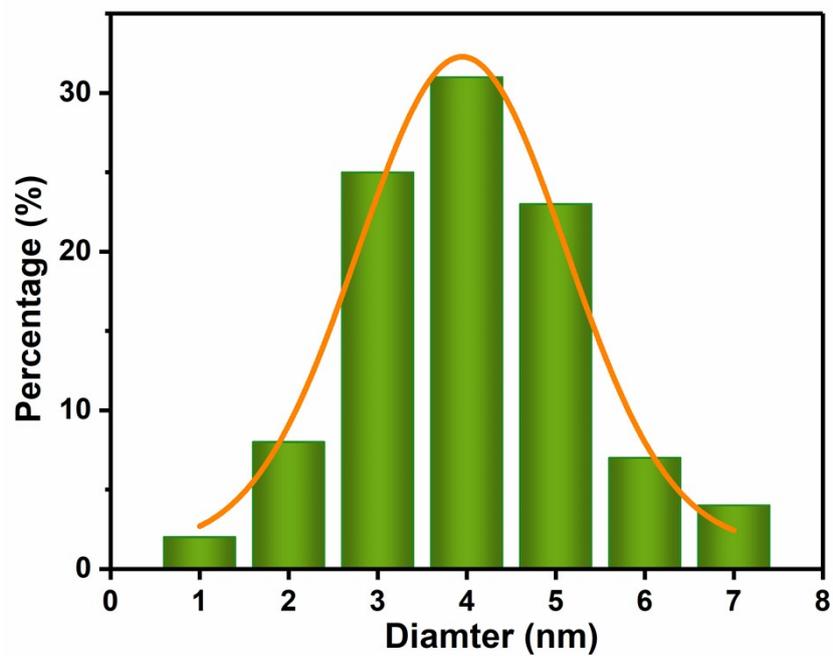


Fig. S2. Diameter distribution for N-MQDs.

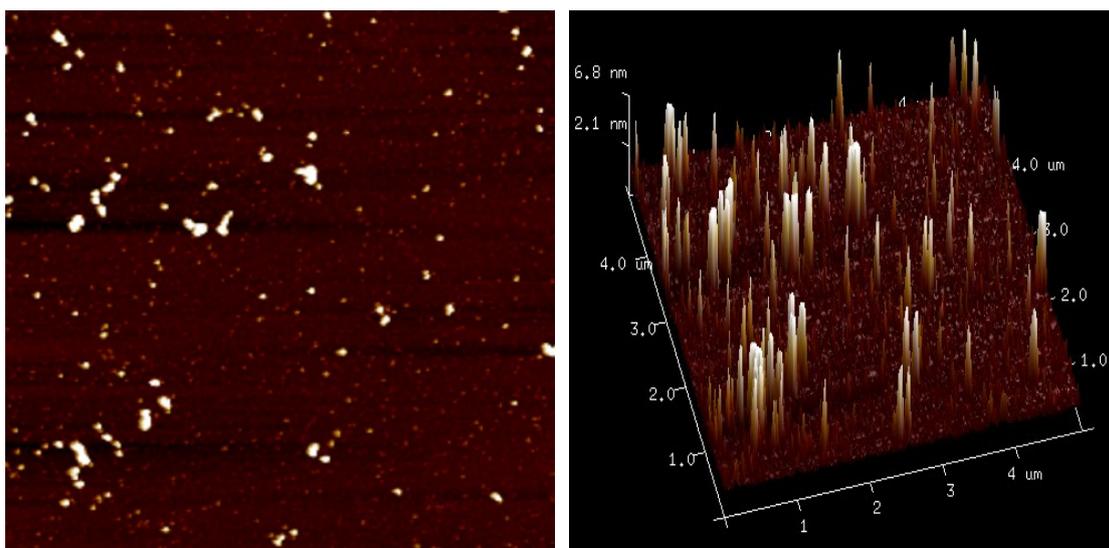


Fig. S3. AFM image and 3D AFM rendered image for N-MQDs

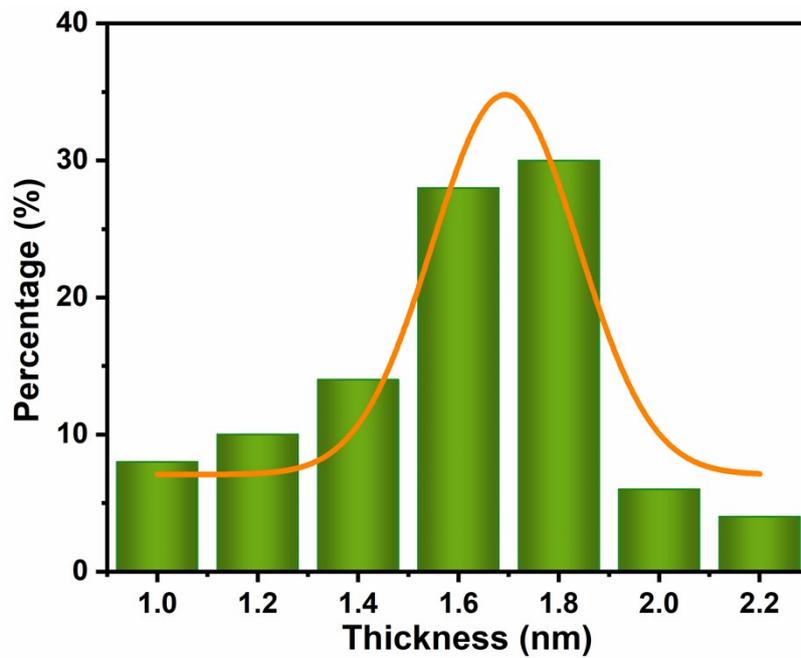


Fig. S4. The height size distribution for N-MQDs.

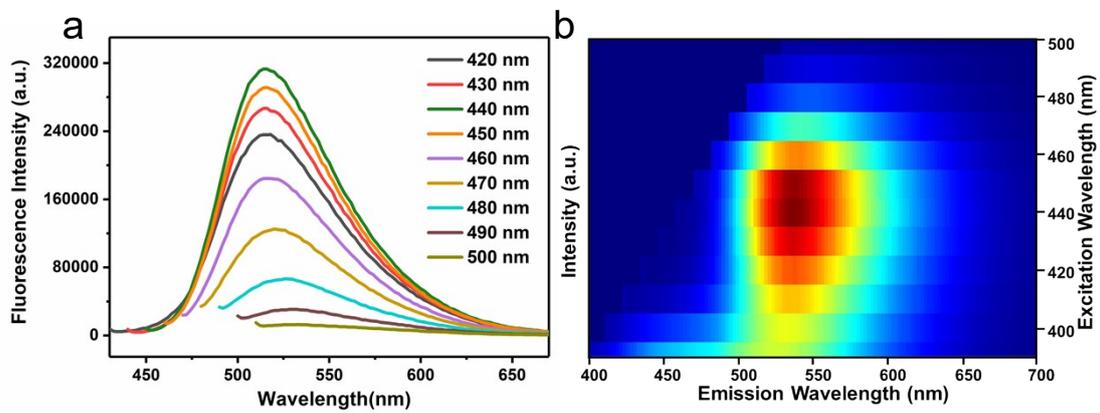


Fig. S5. (a) PL spectra for N-MQDs. (b) excitation-emission matrix.

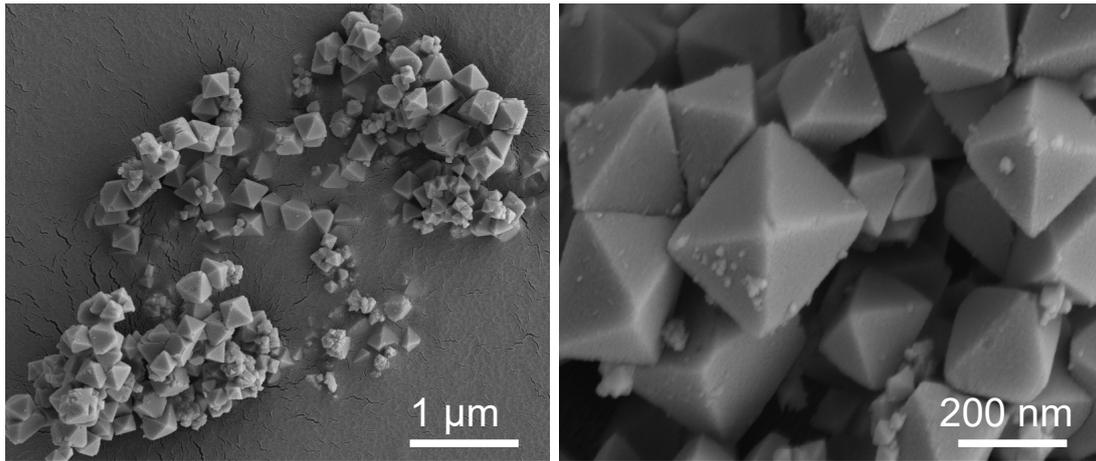


Fig. S6. SEM images for octahedral CoO.

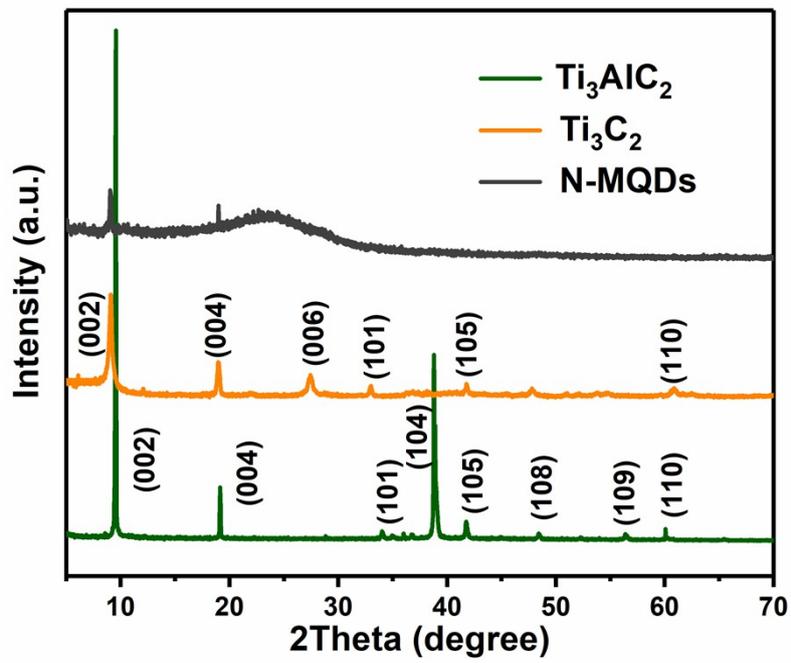


Fig. S7. XRD patterns for Ti₃AlC₂ powder, Ti₃C₂ MXene, and N-MQDs.

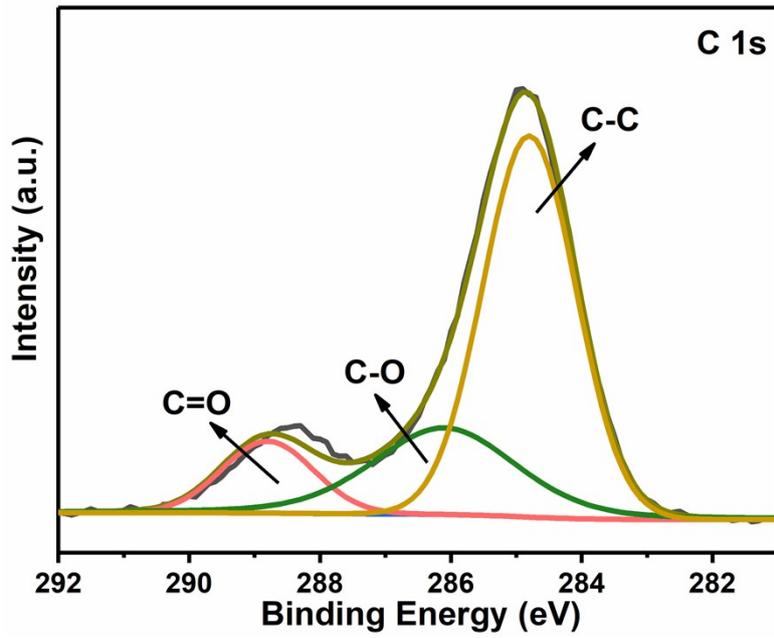


Fig. S8. C 1s XPS spectra for CoO.

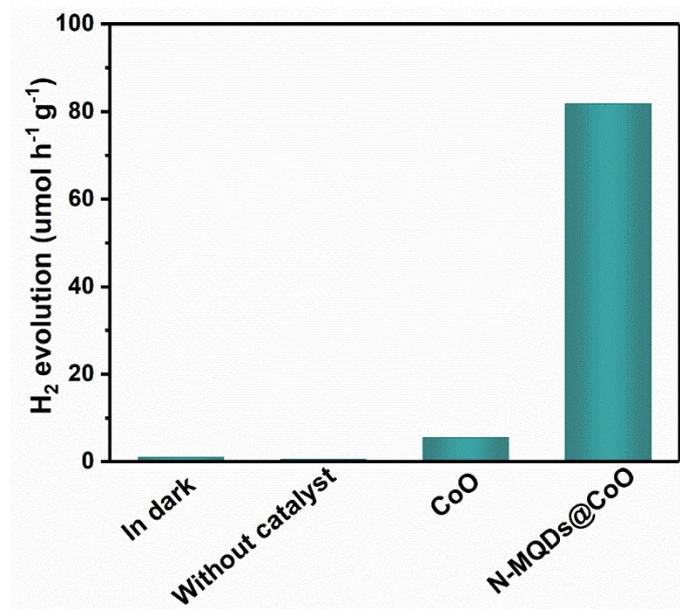


Fig. S9. The comparison of H₂ production rates with different environments.

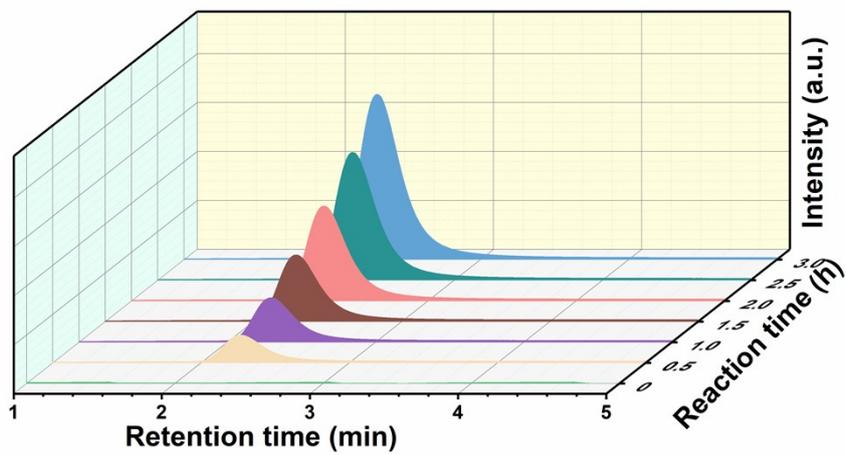


Fig. S10. Time-dependent gas chromatograms H₂ production for N-MQDs@CoO.

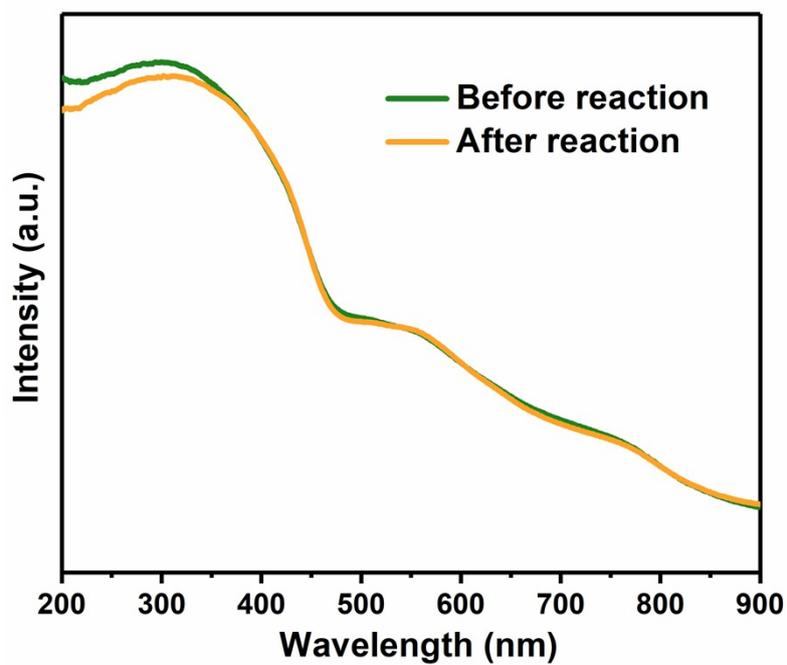


Fig. S11. (a) UV-visible DRS for the recycled catalyst after 4 cycles.

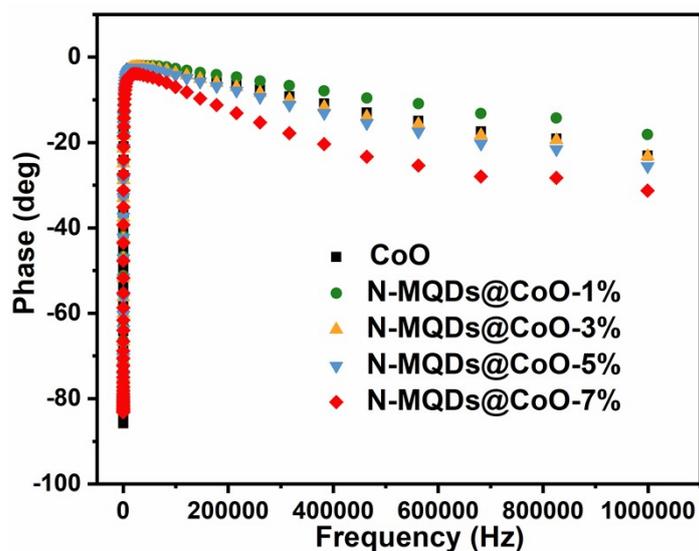


Fig. S12. The Bode-phase for pure CoO and N-MQDs@CoO.

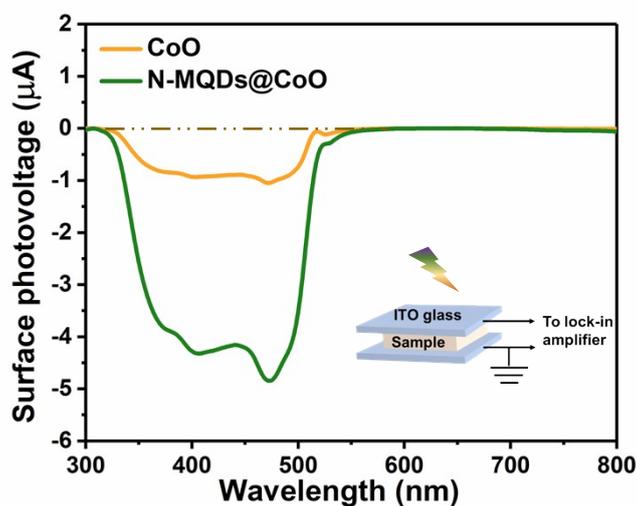


Fig. S13. The SPV spectrum for pure CoO and N-MQDs@CoO.

Table S1. Entries in the table are fitted from EIS results

Sample	R_s/Ω	R_{ct}/Ω	C_p
CoO	27.72	23660	4.41×10^{-4}
N-MQDs@CoO-1%	18.65	16360	2.48×10^{-3}
N-MQDs@CoO-3%	9.02	4375	2.11×10^{-4}
N-MQDs@CoO-5%	4.48	1715	1.32×10^{-4}

N-MQDs@CoO-7%	7.45	1044	1.91×10^{-3}
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Table S2. The average fluorescence lifetimes for CoO and N-MQDs@CoO

Sample	lifetime τ (ns)	Pre-exponential Factors B	Average lifetimes τ (ns)	X_2
CoO	$\tau_1=2.01$	$B_1=3133.26$	2.19	1.168
	$\tau_2=8.17$	$B_2=36.55$		
N-MQDs@CoO-1%	$\tau_1=3.26$	$B_1=998.73$	4.52	1.098
	$\tau_2=9.94$	$B_2=2042.98$		
N-MQDs@CoO-3%	$\tau_1=5.50$	$B_1=921.90$	6.39	1.046
	$\tau_2=10.09$	$B_2=2059.96$		
N-MQDs@CoO-5%	$\tau_1=7.22$	$B_1=2906.34$	8.43	1.063
	$\tau_2=15.98$	$B_2=109.41$		
N-MQDs@CoO-7%	$\tau_1=7.27$	$B_1=2788.07$	6.81	1.086
	$\tau_2=12.54$	$B_2=274.42$		

Table S3. Comparison for different CoO-based materials

Photocatalyst	Light source	Catalyst dosage (mg)	H ₂ evolution rate	Ref.
N-MQDs@CoO	≥ 420 nm	10	81.6 μmol g ⁻¹ h ⁻¹	This work
CoO@MoS ₂	>400 nm	50	21.4 μmol g ⁻¹ h ⁻¹	[1]
CoO/NiCo-LDH	>420 nm	40	1.5 mmol g ⁻¹ h ⁻¹	[2]
CoO/g-C ₃ N ₄	>400 nm	50	50.2 μmol g ⁻¹ h ⁻¹	[3]
CoP/CoO	>420 nm	50	43.4 μmol g ⁻¹ h ⁻¹	[4]
a-CoO/GO	>400 nm	50	21.1 μmol g ⁻¹ h ⁻¹	[5]
rGO@CoO	≥ 420 nm	50	830 μmol h ⁻¹ g ⁻¹	[6]
CDs/CoO	>400 nm	50	33.4 μmol g ⁻¹ h ⁻¹	[7]

References

- [1] Shi, W., Guo, F., Li, M., Shi, Y., Shi, M., Yan, C., 2019. Constructing 3D sub-micrometer CoO octahedrons packed with layered MoS₂ shell for boosting photocatalytic overall water splitting activity. *Appl. Surf. Sci.* 473, 928-933.
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- [4] Guo, F., Huang, X., Chen, Z., Sun, H., Chen, L., 2021. Anchoring CoP nanoparticles on the octahedral CoO by self-phosphating for enhanced photocatalytic overall water

splitting activity under visible light. *Chin. J. Chem. Eng.* 40, 114-123.

[5] Lin, Z., Du, C., Yan, B., Yang, G., 2019. Two-dimensional amorphous CoO photocatalyst for efficient overall water splitting with high stability. *J. Catal.* 372, 299-310.

[6] Selvarajan, R., Vadivel, S., Saranya, A., Baraneedharan, P., Jayavel, R., 2022. Facile synthesis of rGO@CoO nanocomposites electrode material for photocatalytic hydrogen generation and supercapacitor applications. *Inorg. Chem. Commun.* 139, 109345.

[7] Shi, W., Guo, F., Zhu, C., Wang, H., Li, H., Huang, H., Liu, Y., Kang, Z., 2017. Carbon dots anchored on octahedral CoO as a stable visible-light-responsive composite photocatalyst for overall water splitting. *J. Mater. Chem. A* 5, 19800-19807.