Supporting information

Insight into the factors influencing the photocatalytic H_2 evolution performance of molybdenum sulfide

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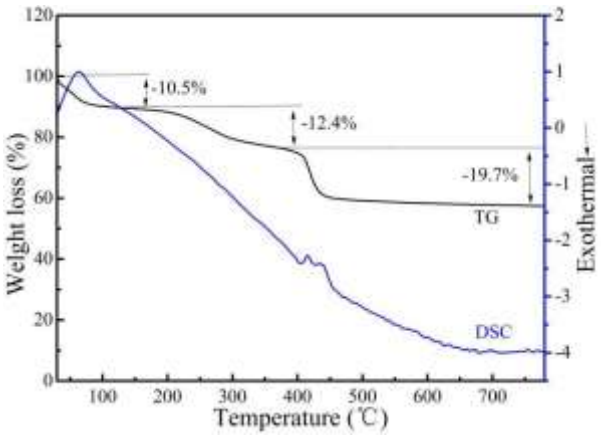

Fig. S1 TG-DSC curves of the amorphous MoS_3 in Ar atmosphere.

Table S1 Atomic ratio of S and Mo obtained by quantitative XPS analysis

Sample	Ratio (S_2^{2-})	Ratio (S^{2-})	Ratio (S_2^{2-}/S^{2-})	S/Mo
300-MoS_{2+x}	57.9%	42.1%	1.4	2.78
350-MoS_{2+x}	55.0%	45.0%	1.2	2.40
400-MoS_{2+x}	--	100%	--	1.84

Table S2 Microstructure of MoS_{2+x} synthesized at different temperatures

Sample	S_{BET} (m^2/g)	Pore volume (cm^3/g)	Pore diameter (nm)
200-MoS_{2+x}	8.6	0.03	6.3
300-MoS_{2+x}	23.6	0.16	22.8
350-MoS_{2+x}	14.4	0.052	12.5
400-MoS_{2+x}	110.7	0.21	7.1
500-MoS_{2+x}	70.3	0.32	14.9

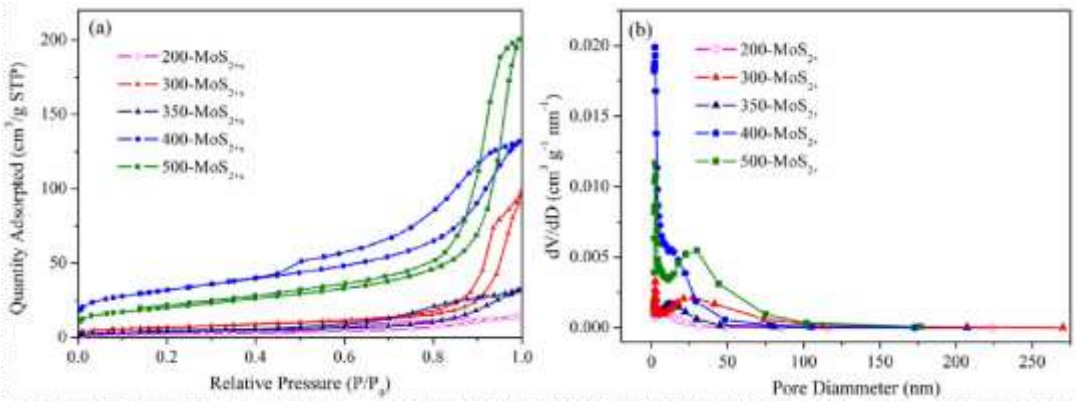

Fig. S2 N_2 adsorption-desorption isotherms (a) and BJH pore-size distribution curves (b) for MoS_{2+x} samples obtained at different temperatures.

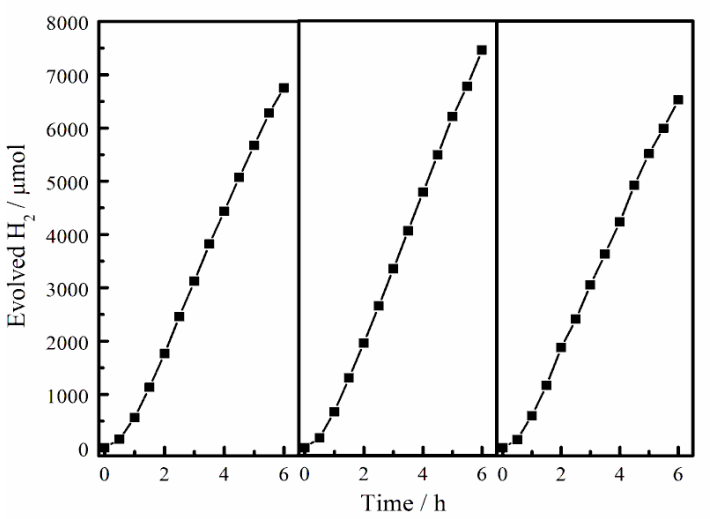

Fig. S3 Recycling test of H_2 evolution over 400-MoS_{2+x} in 200 mL 10 vol% TEOA aqueous solution containing 0.1 g catalyst, 7.5 mM EB, pH 9.0. In the second and third cycles, EB and TEOA were added into the new system again. (Note: the discrepancy with Fig. 5 in H_2 evolution activity is because that the sample was not the same batch.)

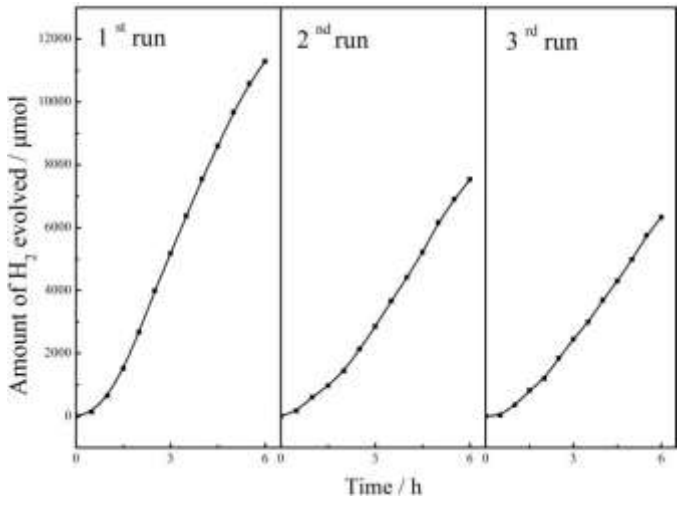

Fig. S4 Recycling test of H_2 evolution over 300-MoS_{2+x} in 200 mL 10 vol% TEOA aqueous solution containing 0.1 g catalyst, 7.5 mM EB, pH 9.0. In the second and third cycles, EB and TEOA were added into the new system again.

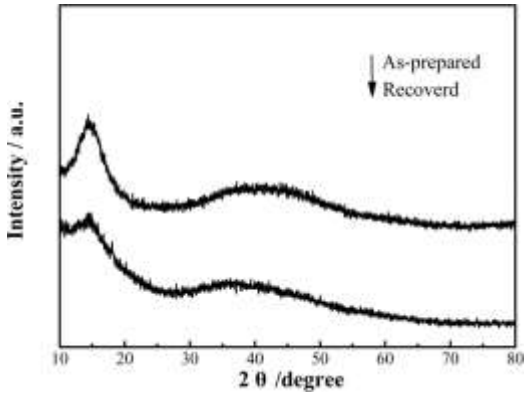

Fig. S5 XRD patterns of 300-MoS_{2+x} samples as-prepared and recovered after H_2 evolution reaction.

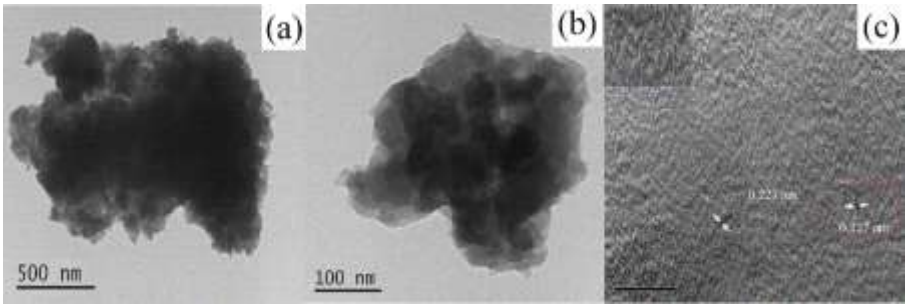

Fig. S6 TEM (a, b) and HRTEM (c) images of 300-MoS_{2+x} recovered after H_2 evolution reaction.

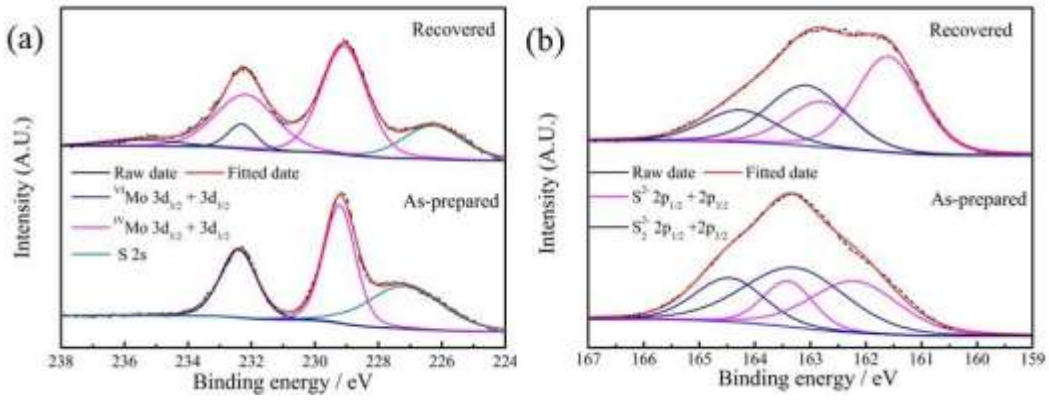

Fig. S7 High resolution XPS spectra of Mo 3d (a) and S 2p (b) for the 300-MoS_{2+x} samples as-prepared and recovered after reaction.

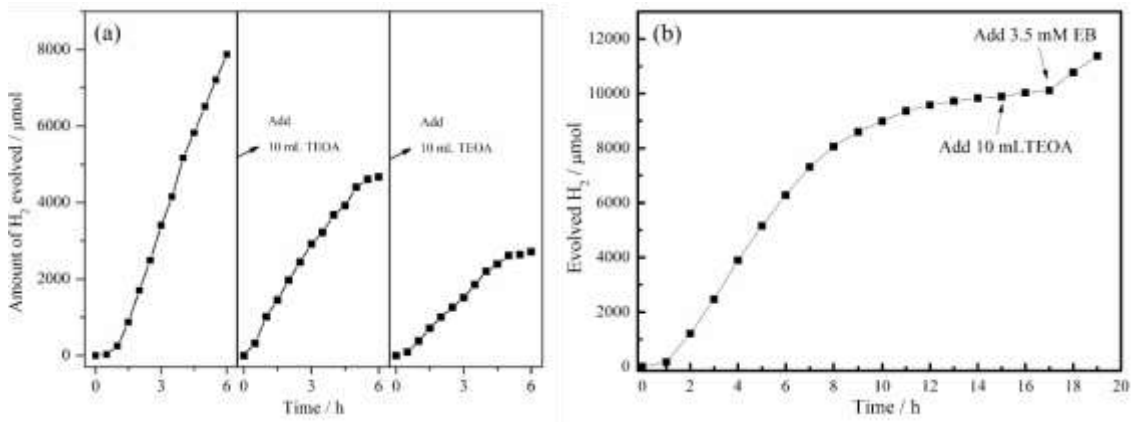

Fig. S8 (a) Recycling test of H_2 evolution over 300-MoS_{2+x} in 200 mL of TEOA aqueous solution containing 0.1 g catalyst, 7.5 mM EB, pH 9.0. In the second and third cycles, only 10 mL TEOA were readded into the original system. (b) Time course of hydrogen production over 300-MoS_{2+x} in 200 mL of TEOA aqueous solution containing 0.1 g catalyst, 7.5 mM EB, pH 9.0. At the time of 15 h and 17 h, 10 mL TEOA and 3.3 mM EB were added again, respectively. (Note: the discrepancy with Fig. S4 in H_2 evolution activity is because that the sample was not the same batch.)

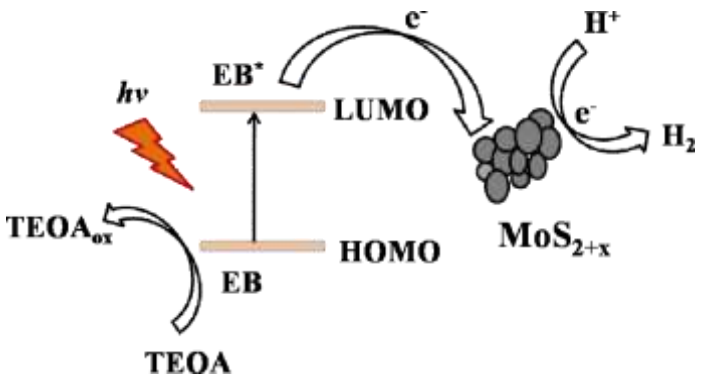

Fig. S9 Photocatalytic hydrogen production mechanism of MoS_{2+x} sensitized by EB.