

Supplementary Information

Superior Dye Adsorbent towards Hydrogen Evolution Reaction Combining Active Sites and Phase-Engineering of (1T/2H) MoS₂/ α -MoO₃ Hybrid Heterostructured Nanoflowers

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Sample	2H-MoS ₂ (%)	1T-MoS ₂ (%)	α -MoO ₃ (%)
12 hr	40.8	46.8	12.2
16 hr	50.5	36.2	13.2
20 hr	70.6	21.9	7.3

Table S1: Calculated percentages of desired phases of MoS₂ and α -MoO₃

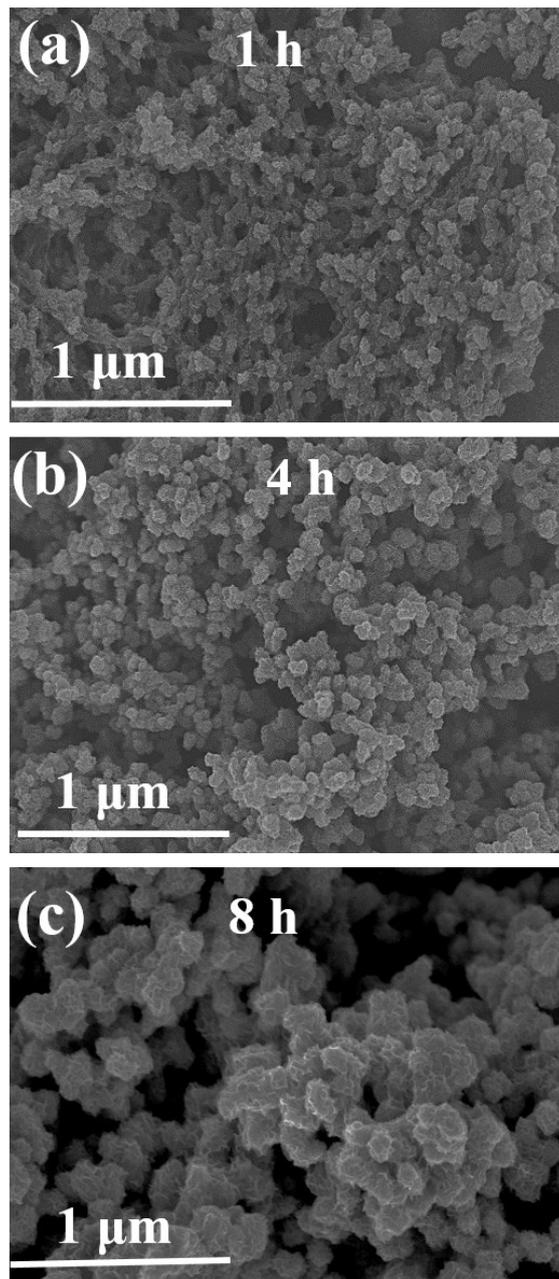


Fig. S1 SEM images of the sample prepared at 1h (a), 4h (b) and 8h (c).



Sample Details

12 h

Sample Name: mc12 dilution 1
SOP Name: mansettings.nano
General Notes:
File Name: 20180503.dts Dispersant Name: ethanol
Record Number: 4 Dispersant RI: 1.362
Material RI: 1.59 Viscosity (cP): 0.9930
Material Absorbance: 0.010 Measurement Date and Time: 2018/05/31 7:10:41:32

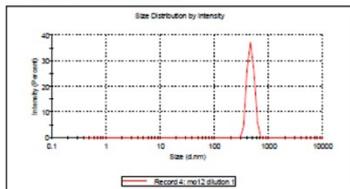
System

Temperature (°C): 30.0 Duration Used (s): 70
Count Rate (kcps): 159.9 Measurement Position (mm): 3.00
Cell Description: Disposable micro cuvette (4... Attenuator: 11

Results

Z-Average (d.nm)	Peak 1: Size (d.nm)	% Intensity	±1 Dev (d.nm)
455.7	455.4	100.0	66.72
PDI: 0.416	Peak 2: 0.000	0.0	0.000
Intercept: 0.884	Peak 3: 0.000	0.0	0.000

Result quality Refer to quality report



Sample Details

16 h

Sample Name: mc16 1
SOP Name: mansettings.nano
General Notes:
File Name: 20180503.dts Dispersant Name: ethanol
Record Number: 3 Dispersant RI: 1.362
Material RI: 1.59 Viscosity (cP): 0.9930
Material Absorbance: 0.010 Measurement Date and Time: 2018/05/31 7:10:37:42

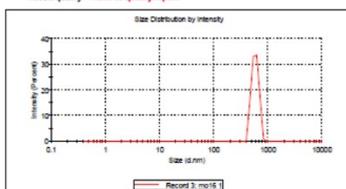
System

Temperature (°C): 30.0 Duration Used (s): 30
Count Rate (kcps): 250.3 Measurement Position (mm): 3.00
Cell Description: Disposable micro cuvette (4... Attenuator: 11

Results

Z-Average (d.nm)	Peak 1: Size (d.nm)	% Intensity	±1 Dev (d.nm)
564.2	562.5	100.0	83.83
PDI: 0.303	Peak 2: 0.000	0.0	0.000
Intercept: 0.887	Peak 3: 0.000	0.0	0.000

Result quality Refer to quality report



Sample Details

20 h

Sample Name: mc20 dilution 1
SOP Name: mansettings.nano
General Notes:
File Name: 20180503.dts Dispersant Name: ethanol
Record Number: 6 Dispersant RI: 1.362
Material RI: 1.59 Viscosity (cP): 0.9930
Material Absorbance: 0.010 Measurement Date and Time: 2018/05/31 7:10:49:05

System

Temperature (°C): 30.0 Duration Used (s): 60
Count Rate (kcps): 110.0 Measurement Position (mm): 3.00
Cell Description: Disposable micro cuvette (4... Attenuator: 11

Results

Z-Average (d.nm)	Peak 1: Size (d.nm)	% Intensity	±1 Dev (d.nm)
590.7	525.4	100.0	90.30
PDI: 0.113	Peak 2: 0.000	0.0	0.000
Intercept: 0.969	Peak 3: 0.000	0.0	0.000

Result quality Refer to quality report

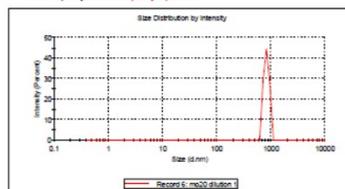


Fig. S2 Calculated size distribution by DLS for samples prepared at 12 h, 16 h and 20 h.

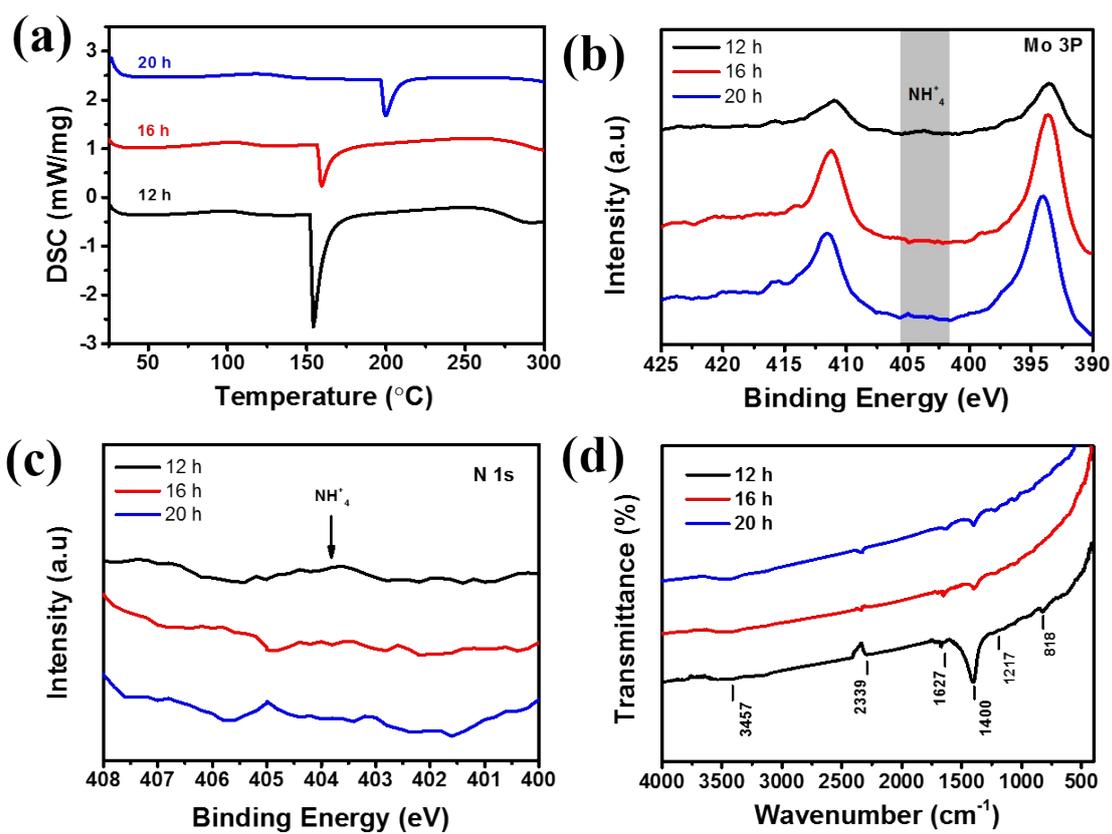


Fig. S3 (a) DSC (b and c) XPS and (d) FTIR spectra of (1T/2H) MoS₂/α-MoO₃

heterostructured nanoflowers prepared at 12, 16 and 20 h, respectively.

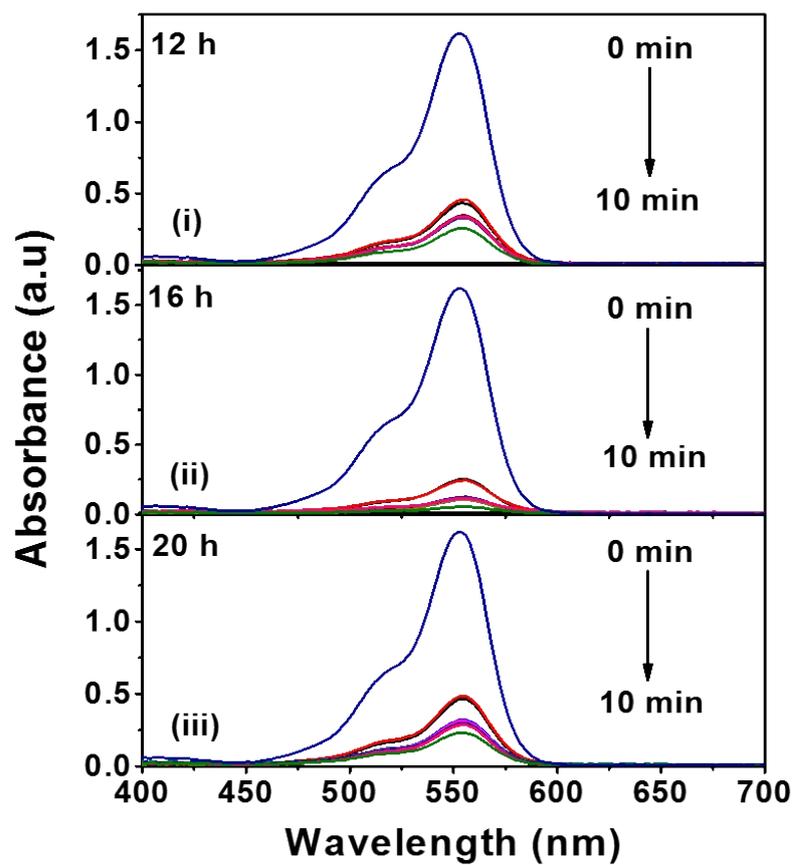


Fig. S4 UV-vis absorption spectra of samples with different reaction time. 12 hr (i), 16 hr (ii) and 20 hr (iii).

0 min → 10 min

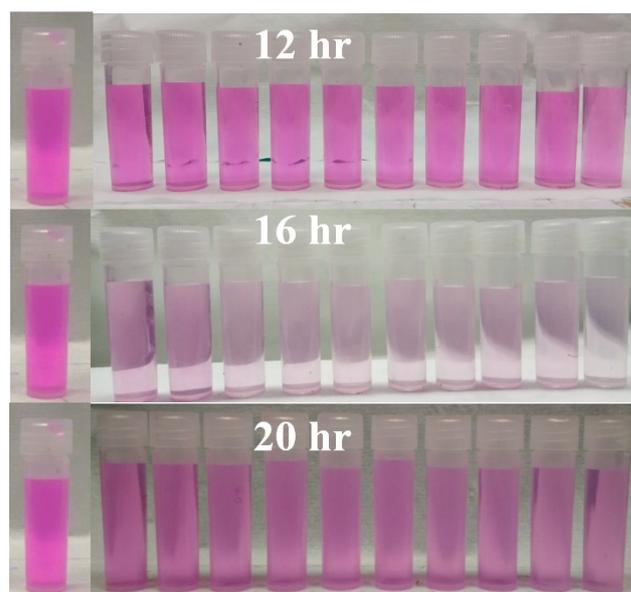


Fig. S5 Photographic images of RhB after adsorption with respect to time.

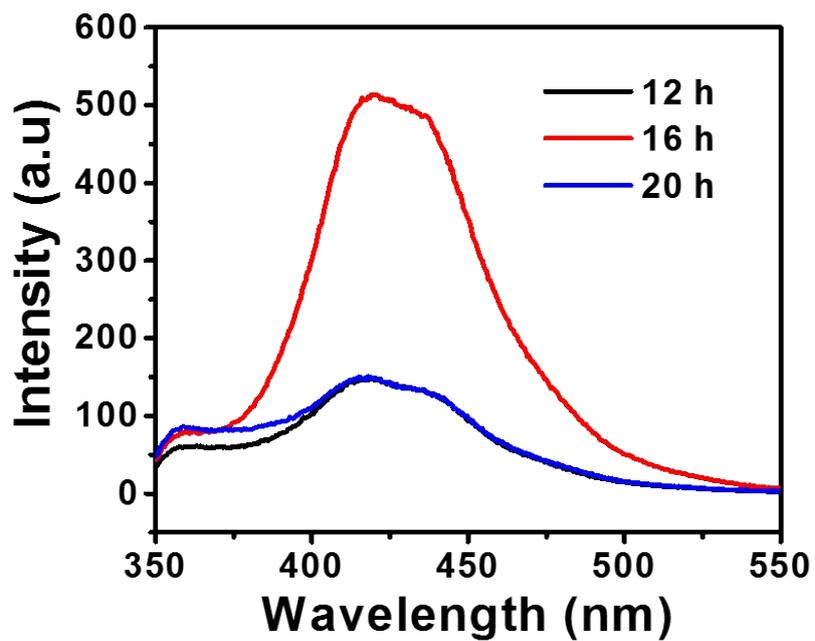


Fig. S6 The evolution of $\cdot\text{OH}$ radicals by fluorescence spectra of TA under mild sonication with all the three samples.

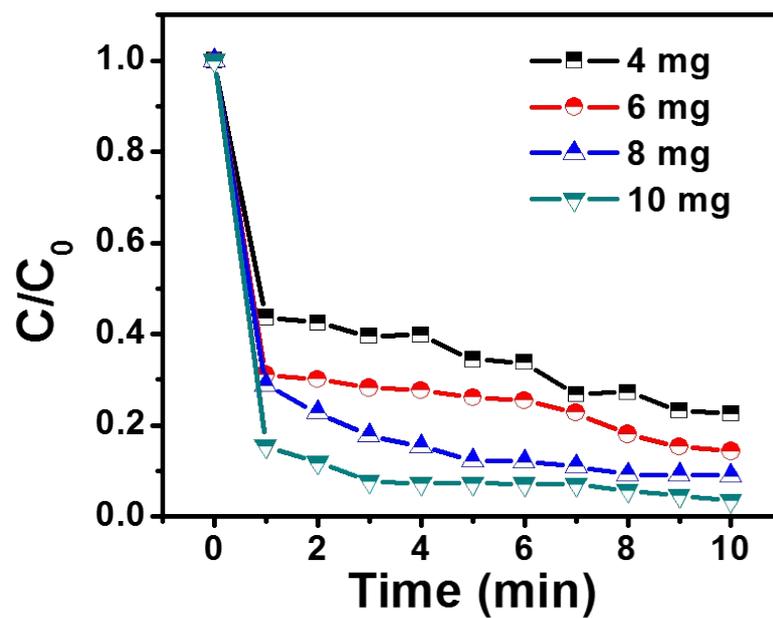


Fig. S7 Adsorbent quantity dependent of 16 hr sample towards degradation of RhB solution with initial concentration of 47.9 mg L^{-1} .

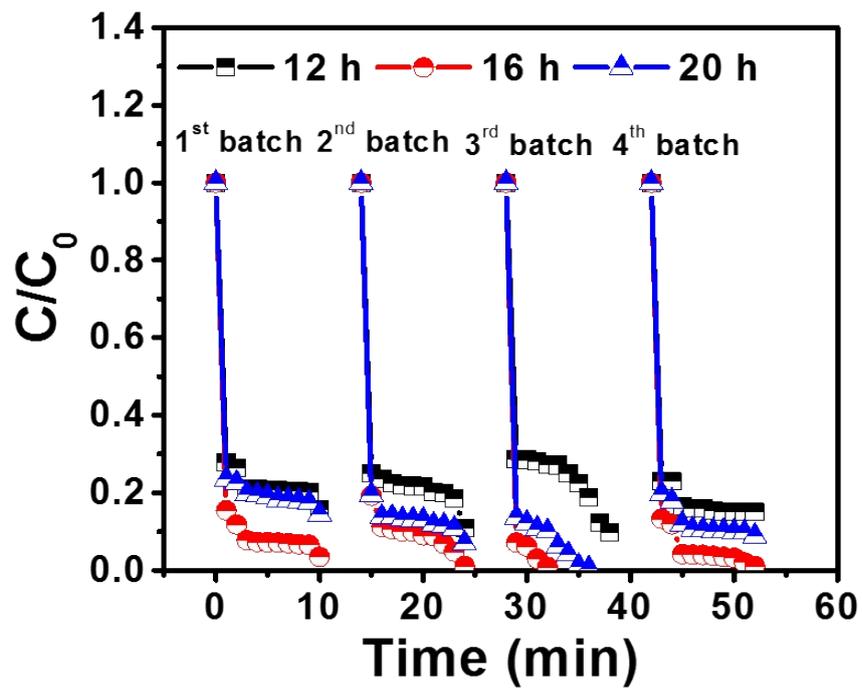


Fig. S8 Reproducibility of adsorption showing for all the three samples prepared over four batches.

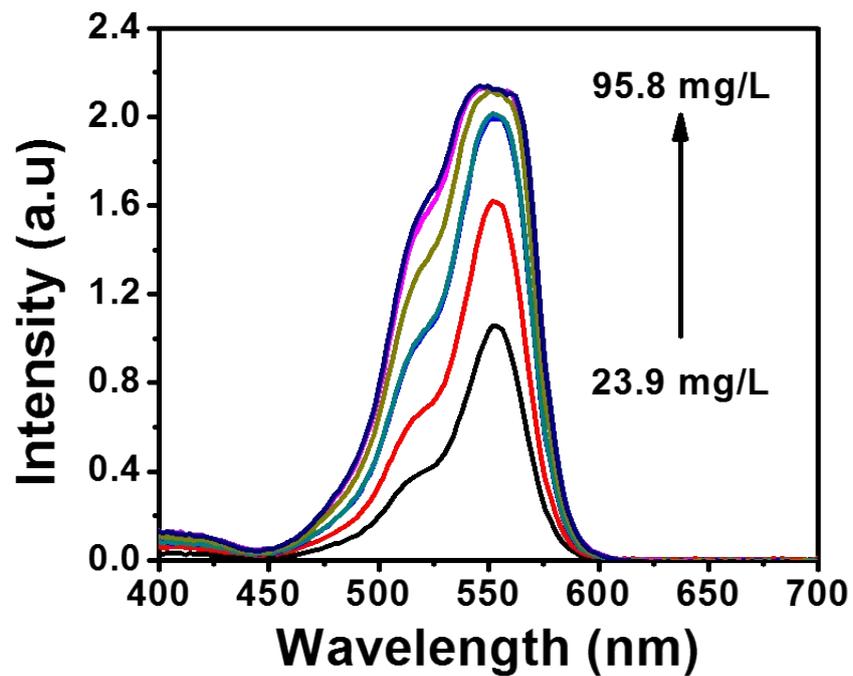


Fig. S9 UV-vis absorption spectra of RhB solution to the increase of its initial concentration.

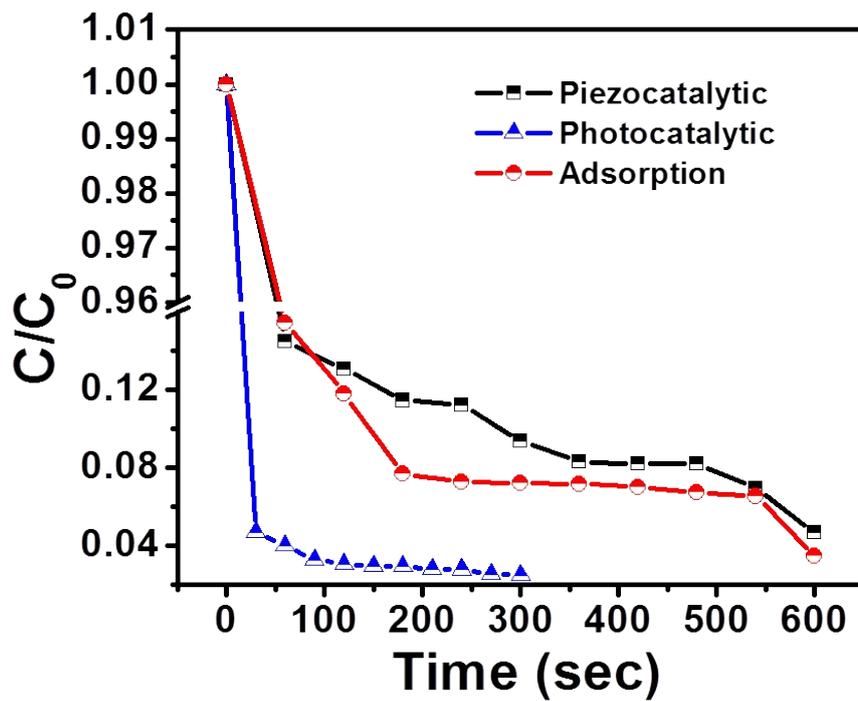


Fig. S10 Comparison of adsorption process with piezo-catalytic and photocatalytic method of dye degradation of RhB solution by utilizing 16 hr sample as adsorbent.

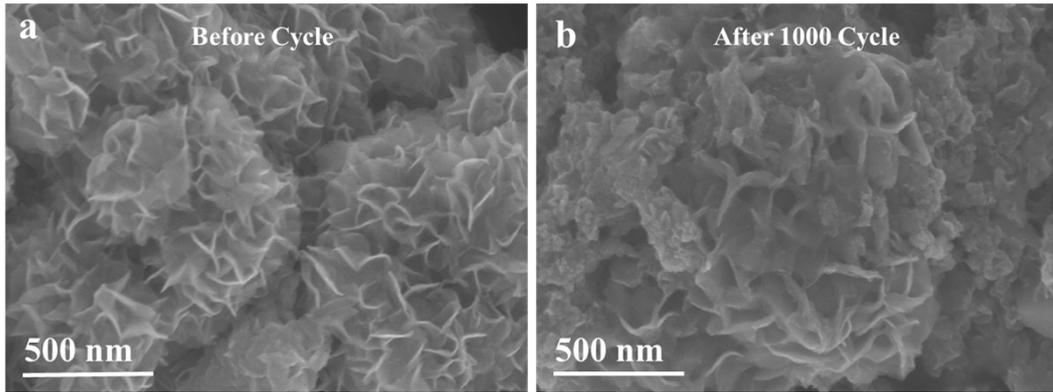


Fig. S11 (a) SEM image of 12 h sample before cycle and (b) after 1000 cycle.