Supporting Information

Synthesis of TiO₂ Nanosheet Photocatalysts from Exfoliation of TiS₂ and Hydrothermal Treatment

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Supporting Table

Table SI. Kinetic fitting results for MB degradation by TiO_2 -based photocatalysts. The data were fit to a first-order model, which can be described as: $\ln (C_{initial}/C) = kt$, where $C_{initial}$ is the initial concentration of MB (5 ppm), C is the concentration of MB at irradiation time t, and *k* is the rate constant.

Photocatalyst	k (min ⁻¹)	r ²
P25	0.185	0.991
TiO ₂ nanosheets	0.103	0.992
P25 + CB	0.071	0.957
P25 + CB + PVDF	0.071	0.978

Supporting Figures



FIG. S1. Lithiation voltage profile of bulk TiS₂. The electrode was charged galvanostatically using a 0.05 C rate until the potential reached 0.9 V vs. Li/Li⁺, as indicated by the arrow. Then, the electrode was held at 0.9 V for 2 h, followed by a 1.5 h rest at open circuit. If the voltage was higher than 1 V vs. Li/Li⁺ after this relaxation period, the electrode was charged galvanostatically again using a 0.05 C rate until the potential reached 0.9 V vs. Li/Li⁺, followed by another 2 h potentiostatic hold at 0.9 V. These steps were repeated until the open circuit voltage was less than 1 V vs. Li/Li⁺ to ensure sufficient lithiation to weaken the van der Waals interactions between the TiS₂ interlayers and enable exfoliation of the nanosheets after applying ultrasonic frequencies.



FIG. S2. Photographs of (a) dispersed $Li_x TiS_2$ nanosheets obtained after electrochemical lithiation of bulk TiS_2 , and (b) TiO_2 nanosheets obtained after hydrothermal treatment.



FIG. S3. Raman spectroscopy analysis of prepared TiO_2 nanosheets. (a) Optical microscopy image with blue spot localized on a TiO_2 nanosheet and red spot on carbon residue on the background. (b) Corresponding Raman spectrum from the red and blue spots in (a). The peak at 150 cm⁻¹ is from the E_g mode of anatase while the large peak at 520 cm⁻¹ is from the crystalline Si substrate.



FIG. S4. SEM images of as-received, bulk TiS₂.



FIG. S5. XRD pattern of (a) as-received, bulk TiS_2 , (b) as-prepared TiO_2 nanosheets obtained after hydrothermal treatment of TiS_2 nanosheets, and (c) as-prepared bulk TiO_2 obtained after hydrothermal treatment of bulk TiS_2 . The reference patterns for TiS_2 and TiO_2 were obtained from JCPDS No.15-0853 and JCPDS No. 21-1272, respectively.



FIG. S6. XPS analysis of P25 TiO₂ (aligning to the C 1*s* peak at 284.7 eV). The Ti $2p^{3/2}$ and Ti $2p^{1/2}$ peaks are found at 587.3 eV and 464.4 eV. In the O 1s spectrum, the peak at 529.2 is assigned to oxygen in the TiO₂ lattice (e.g., Ti-O-Ti) while the peak at 531.01 eV is attributed to surface hydroxide.



FIG. S7. Diffuse reflectance spectra obtained for TiO_2 nanosheets, bulk TiS_2 , bulk TiO_2 and P90 TiO_2 . Bulk TiS_2 refers to the as-obtained TiS_2 powder. Bulk TiO_2 refers to the TiO_2 obtained after hydrothermal treatment of the bulk TiS_2 powder (without electrochemical lithiation and exfoliation).



FIG. S8. Change in UV-vis absorbance of MB (5 ppm initial concentration) in aqueous suspension of TiO_2 nanosheets under UV irradiation (0 – 40 min).



FIG. S9. SEM image of product obtained after electrochemical reduction of carbon-black free TiS_2 electrode followed by sonication. In this case, the binder used was carboxymethyl cellulose.