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Fabrication and Enhanced Solar Hydrogen Production of 2D/2D COF/$SnNb_2O_6$ Nanosheets

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Experimental section

Characterization. The phase and crystallinity of the samples were characterized by an XRD-6100 X-ray diffractometer. The morphology of the samples was characterized by JEOL JEM 2100F transmission electron microscopy (TEM) and high-resolution projection electron microscopy (HRTEM). Qualitative and quantitative analysis of sample elements using Escalab 250Xi energy dispersive X-ray spectrometer (EDS); specific surface area and pore size distribution of samples using TriStar II 3020 specific surface area and pore analyzer; ESCA PHI500 X-ray photoelectron spectroscopy The instrument (XPS) analyzes the chemical elements and valence states in the sample; the UV-Vis absorption spectroscopy (UV-Vis DRS) was used to test the UV-visible absorption properties of the sample; the CHI660E electrochemical workstation is used to test the photocurrent of the sample.

Photocatalytic H_2 production test. Photocatalytic hydrogen production experiments were performed in a 250 mL Pyrex flask connected to a closed gas circulation and evacuation system for sampling. A 300 W Xe lamp equipped with a 420 nm filter was used as the visible light source, and the xenon lamp was placed at 10 cm in the reactor with an irradiation intensity of 171 $mW \cdot cm^{-2}$. In a typical run, 10 mg of the photocatalyst and 50 ml aqueous solution containing 20 vol% methanol (the mixed solution pH = 6), where methanol is used as a sacrificial agent to remove photo-generated holes, and Pt nanoparticle promoters are removed from H_2PtCl_6 aqueous solution by in-situ photodeposition strategy load onto the catalyst surface. Before the lamp is turned on, the suspension is ultrasonically dispersed for 5 min, and then N_2 is introduced into the reactor for 20 min to remove dissolved oxygen in the suspension to ensure an oxygen-free environment. During the reaction, the reaction solution was kept under stirring, and circulating water was passed to keep the reaction system at room temperature. The temperature of the solution in the reaction system was maintained at 5 °C by circulating cooling water. The above solution is continuously stirred to maintain the homogeneity of the suspension under the photocatalytic reaction. At a given time interval, the content of H_2 was analyzed by gas chromatography (GC-7900, CEAULIGHT, China, TCD, Nitrogen as a carrier gas, and 5Å molecular sieve column). All the determined H_2 amounts were the average data of three independent measurements.

Supplementary Tables and Figures

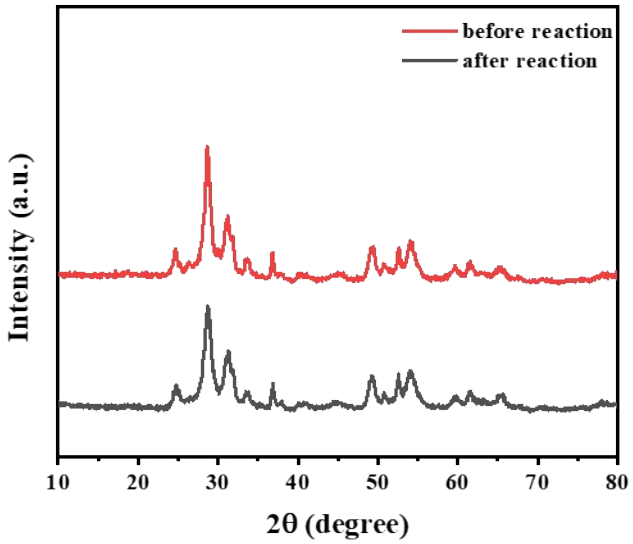

Fig. S1. XRD patterns of TpPa-2-COF/$SnNb_2O_6$ (10:90) nanosheets before and after the photocatalytic reaction

Table 1. Comparison of $SnNb_2O_6$-based photocatalysts for solar hydrogen evolution behavior

Catalyst	Light source	Sacrificial agent	H_2 evolution rate (μmol h^{-1})	Ref.
TpPa-2-COF/$SnNb_2O_6$	300 W Xe lamp (λ>420 nm)	Methanol (20 vol%)	7.66	This work
CNQDs/$SnNb_2O_6$	300 W Xe lamp (λ>420 nm)	Methanol (20 vol%)	5.25	[1]
MoS_2/$SnNb_2O_6$	300 W Xe lamp (λ>400 nm)	Methanol (20 vol%)	12.9	[2]

$SrTiO_3/SnNb_2O_6$	300 W Xe lamp (λ>420 nm)	Methanol (20 vol%)	5.72	[3]
$NaNbO_3/SnNb_2O_6$	300 W Xe lamp (λ>420 nm)	Methanol (20 vol%)	10	[4]
$SnNb_2O_6$ nanoplates	300 W Xe lamp (λ>420 nm)	Methanol (12.5 vol%)	1.84	[5]

Table S2. Corresponding fitted parameters of EIS data using the equivalent circuit in inset in Fig. 4b.

Samples	R_s (Ω cm^2)	R_{ct} (Ω cm^2)
$SnNb_2O_6$	**29.77**	**883.6**
TpPa-2-COF	**27.87**	**226.1**
5：95	**27.41**	**155**
10：90	**19.07**	**119.8**
15：85	**23.97**	**193.6**
20：80	**26.32**	**240.75**

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