

Supplementary Information for

A novel solution-phase approach to nanocrystalline niobates: selective syntheses of $\text{Sr}_{0.4}\text{H}_{1.2}\text{Nb}_2\text{O}_6 \cdot \text{H}_2\text{O}$ nanopolyhedrons and SrNb_2O_6 nanorods photocatalysts

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Experimental Section

Characterization of samples

The as-prepared samples were characterized by powder X-ray diffraction (XRD) on a Bruker D8 Advance X-ray diffractometer operated at 40 kV and 40 mA with Ni-filtered Cu K α irradiation ($\lambda=1.5406$ Å). The amounts of Sr and Nb in the products were measured by an inductively coupled plasma optical emission spectrometer (ICP-OES) (Ultima2, Jobin Yvon Co., France). Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) images were recorded using a JEOL model JEM 2010 EX microscope at an accelerating voltage of 200 kV. The Brunauer–Emmett–Teller (BET) surface area was measured with an ASAP2020M apparatus (Micromeritics Instrument Corp.). UV–vis diffuse reflectance spectra (UV–vis DRS) were obtained by using a UV–vis spectrophotometer (Varian Cary 500) and the data were converted to Kubelka-Munk (KM) functions. Barium sulfate was used as a referent. Electron spin resonance (ESR) signals of spin-trapped paramagnetic species with 5,5-dimethyl-1-pyrroline N-oxide (DMPO) were recorded with a Bruker A300 spectrometer. The settings for ESR spectrometer were center field 3507.0 G, sweep width 80.0 G, microwave frequency 9.85 Hz, power 6.34 mW, modulation frequency 100 kHz, and modulation amplitude 1 G. Also, a 200 W mercury Xe lamp (Hamamatsu Corp., L9566-02) with a set of cutoff filters and an emission at 254 nm was used as a light source.

Evaluation of Photocatalytic Activities

The photocatalytic activities of the samples were evaluated by the degradation of methyl orange (MO) in an aqueous solution under ultraviolet irradiation. The reaction bath was a quartz tube. And three 4 W UV lamps with a wavelength centered at 254 nm (Philips, TUV 4W/G4T5) were used as the illumination source. Reaction suspensions were prepared by adding the powder (160 mg) to 160 mL of an MO solution (20 mg L⁻¹). The suspensions were stirred in the dark for 120 min to ensure adsorption-desorption equilibrium prior to irradiation. All the reaction processes are in present of O₂ flow (60 mL min⁻¹). During irradiation, 3 mL of the suspension was removed at given time intervals for subsequent MO concentration analysis following centrifugation. The MO concentration was analyzed using an UV-vis spectrophotometer (Varian Cary 50) and determined by monitoring the changes of the main absorbance maximized at 464 nm. The TOC values of the MO solutions have been measured with a total organic carbon (TOC-V_{CPH}, Shimadzu, Japan).

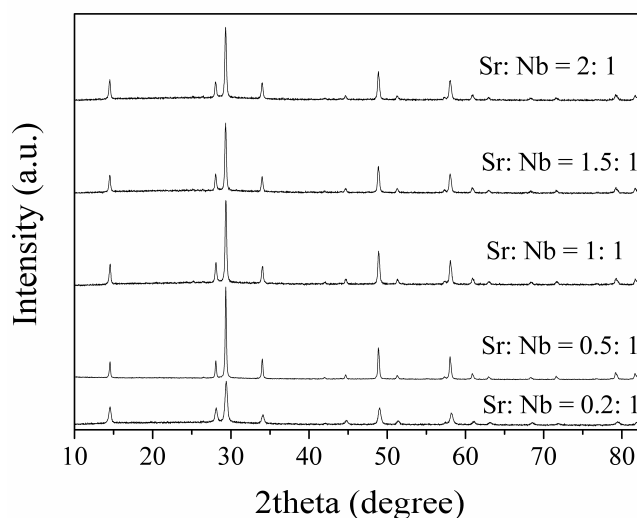


Figure S1 XRD patterns of the as-prepared samples with different molar ratio of Sr/Nb under pH 10

Table S1 The elemental analysis of the products by ICP-OES

Sample	Stoichiometric value/% (wt)		Experimental value/% (wt)		Sr/Nb (mole ratio)
	Sr	Nb	Sr	Nb	
SN1-HT	10.43	55.30	10.29	56.63	1.0: 5.2
SN2-HT	23.72	50.30	23.65	50.85	1.0: 2.0

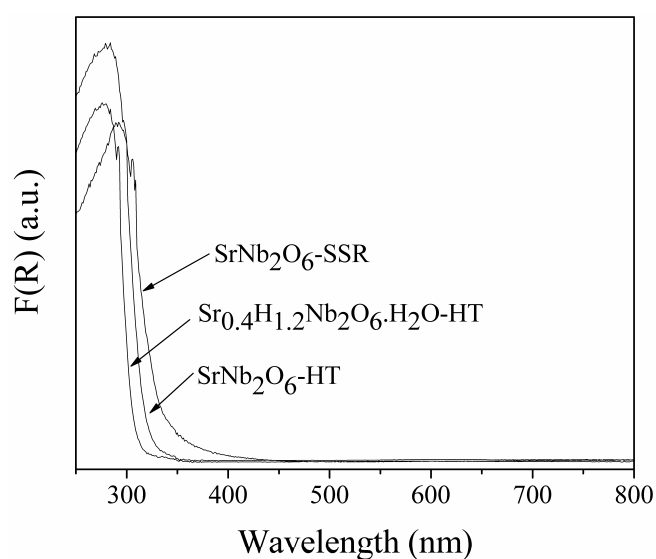


Figure S2. UV-visible diffuse reflection spectrum of products obtained by hydrothermal reactions or solid state reactions.

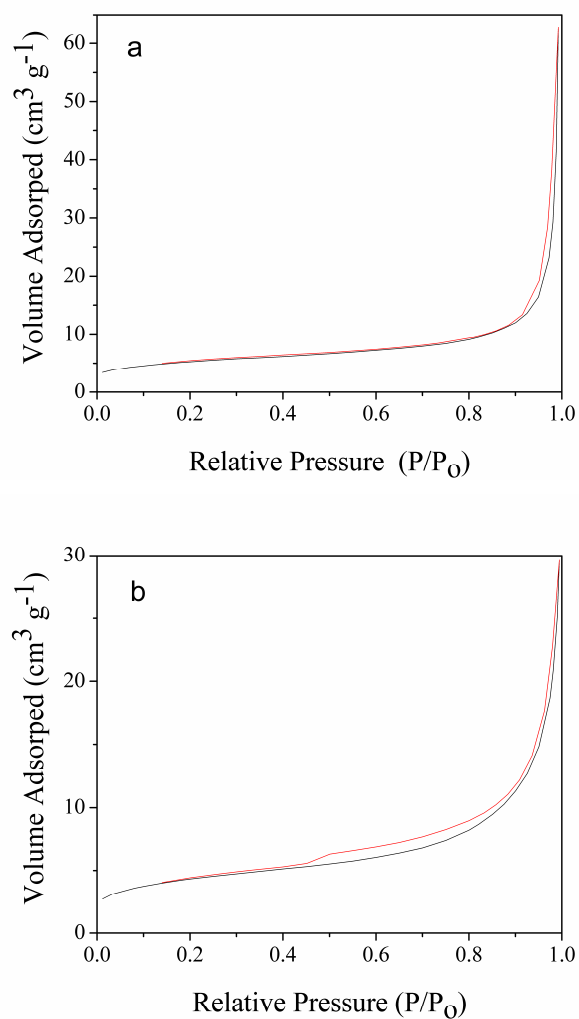


Figure S3. Nitrogen adsorption-desorption isotherm of $\text{Sr}_{0.4}\text{H}_{1.2}\text{Nb}_2\text{O}_6 \cdot \text{H}_2\text{O-HT}$ (a) and $\text{SrNb}_2\text{O}_6\text{-HT}$ (b) powders.

Table S2 The TOC values of the methylorange solutions in present of different catalysts for different reaction times.

Sample	TOC value/mg L ⁻¹		
	0h	2h	3h
SN1-HT	8.8	2.2	1.2
SN2-HT	8.8	3.3	0.9
No catalyst	8.8	8.8	8.8

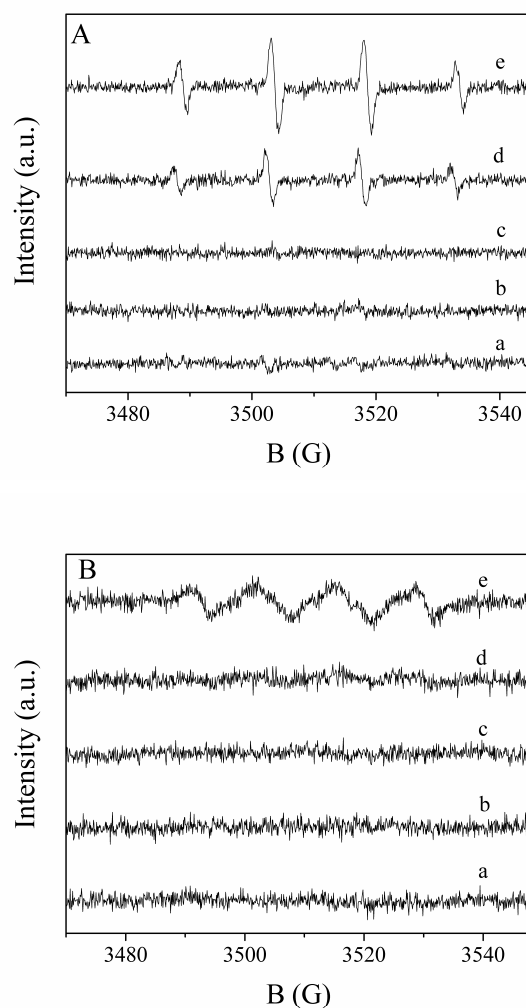


Figure S4. ESR spectra observed for as-preparation samples under UV irradiation for 90 s. The activity species are $\cdot\text{OH}$ (A) and $\cdot\text{O}_2^-$ (B). (a) Blank-light, (b) $\text{Sr}_{0.4}\text{H}_{1.2}\text{Nb}_2\text{O}_6\cdot\text{H}_2\text{O}$ -HT-dark, (c) SrNb_2O_6 -HT-dark, (d) $\text{Sr}_{0.4}\text{H}_{1.2}\text{Nb}_2\text{O}_6\cdot\text{H}_2\text{O}$ -HT-light, (e) SrNb_2O_6 -HT-light.

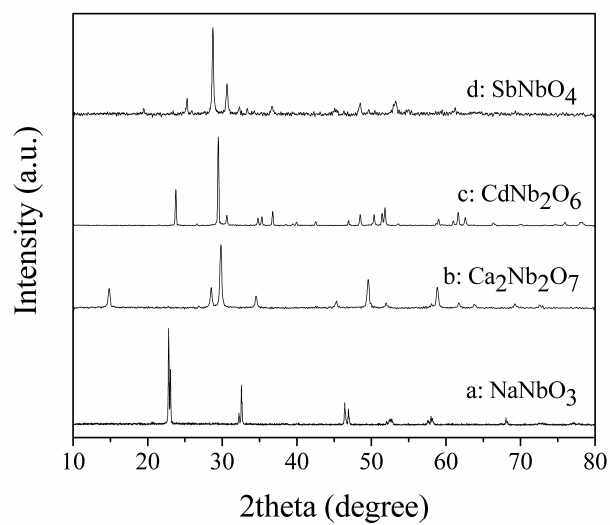


Figure S5. XRD patterns of different hydrothermal samples under 180°C , 48 h, and different pH values: (a) pH 12, (b) pH 10, (c) pH 10, (d) pH 2.