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Constructing novel $NaLiTi_3O_7$/g-C_3N_4 Z-scheme photocatalyst to facilitate the separation of charge carriers and the hydrogen production performance

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1.Characterization of photocatalytic samples

The prepared samples were characterized by X-ray powder diffraction (XRD) on a Rigaku D/max 2500 PC powder diffractometer. The scan rate and scan range were 2°/min and 10-80°, respectively. Thermogravimetric analysis (TGA) was performed on a Seiko TG/DTA 7300 instrument. Scanning electron microscopy (SEM) was performed on a Hitachi S-4800 field emission scanning electron microscope with an acceleration voltage of 10kV. Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HR-TEM) were performed on a Tecnai F20 transmission electron microscope with an acceleration voltage of 200 kV. FT-IR spectra in the mid-infrared range (400-4000 cm^{-1}) were recorded on a Nicolet 5700 FT-IR instrument. X-ray photoelectron spectroscopy (XPS) analysis was performed on a Kratos AXIS-Ultra instrument using monochromatic Al-Kα radiation (225 W, 15 mA, 15 kV). The UV-visible diffuse reflectance spectra (UV-DRS) were recorded in the range of 300-800 nm on a UV-Vis spectrometer (U-4100, Hitachi). In this case, $BaSO_4$ was used as the reflectance standard material. The apparent quantum efficiency (AQE) was measured underillumination ofa 300 W Xe lamp with bandpass filter (365, 420 and 500 nm) system. The quantum efficiency is calculated by the following equation:

$$\mathrm{AQE} = \frac{2MNAhc}{AIt\lambda} \times 100\%$$

where M is the amount of hydrogen molecules, N_A is the Avogadro's constant, h is thePlanck constant, c is the light velocity, I is the intensity of the light, A is the irradiationarea, t is the reaction time, and λ is the wavelength of light.

2. Photocatalytic experiment for water splitting

The photocatalytic H_2-production experiments were performed in an outer irradiation type photoreactor (50 mL quartz glass). A 100 mg catalyst sample was suspended in lactic acid solution (10 vol%). Note that lactic acid is used as a sacrificial electron donor.There was 1 wt% Pt photodeposited on the surface of the catalyst using H_2PtCl_6 as a precursor. Before the reaction, the mixture is degassed under vacuum to remove CO_2 and O_2. The released gas was observed only under irradiation and analyzed using an online gas chromatograph (Techcomp 7900, TCD, Ar carrier). During the reaction, the temperature is maintained at 20℃ by a constant temperature cooling circulation pump.

3. Photochemical properties test

The Versa STAT 3 electrochemical workstation of Princeton University was used to analyze the photoelectric properties of the sample. Firstly, 50 mg samples were continuously stirred and dispersed in 5 mL ethanol solution for 1 h to achieve uniform dispersion. The dispersion is then evenly sprayed onto the FTO glass using an art spray gun. Finally, the FTO glass was calcined in a tubular furnace for 2 hours under N_2 atmosphere at 350℃, and the heating rate was maintained at 5℃min^{-1} to prevent the decline of the sample during the test. The electrochemical workstation is a three-electrode system, the photoanode is sample coated FTO glass, the electrode is platinum plate, the reference electrode is Ag/AgCl, and the electrolyte is Na_2SO_4 aqueous solution. The electrolyte is purified with nitrogen before use to degas the solution to prevent the presence of air from affecting the test results.

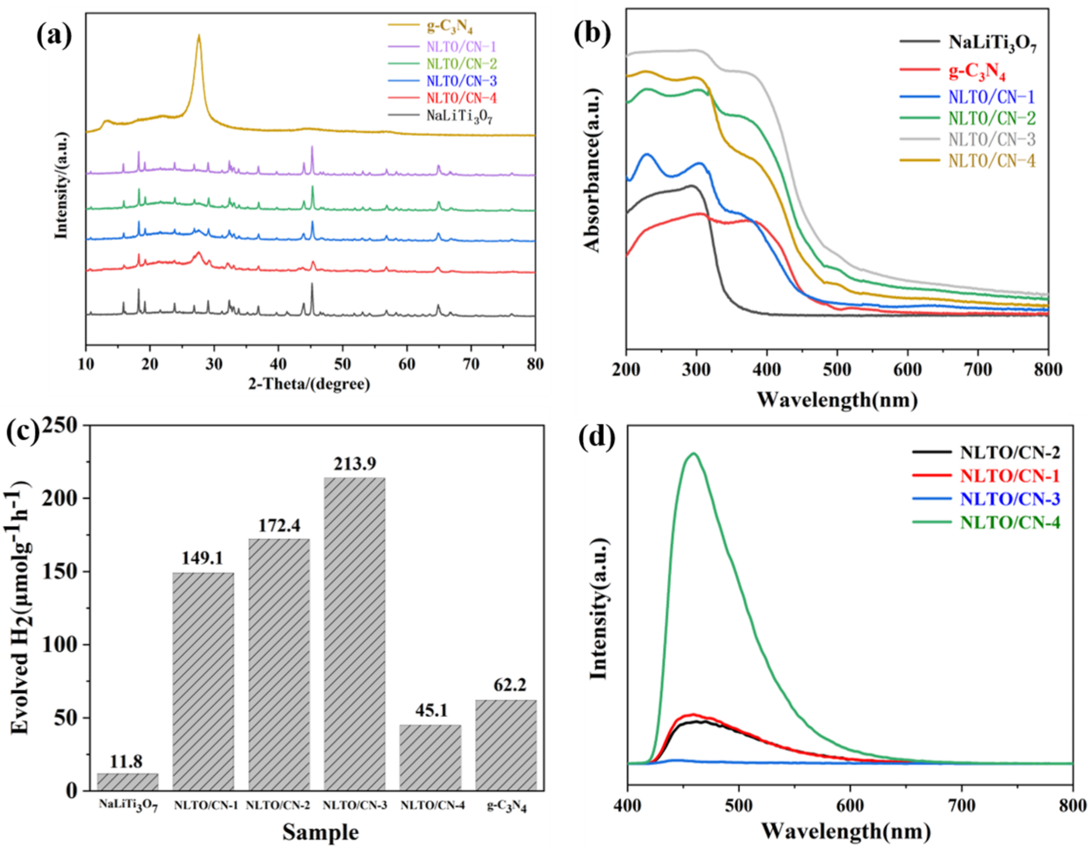

Figure S1 (a) XRD patterns of pure $NaLiTi_3O_7$, pure g-C_3N_4, NLTO/CN-1, NLTO/CN-2, NLTO/CN-3, and NLTO/CN-4 (b) UV-vis DRS diagram of pure $NaLiTi_3O_7$, pure g-C_3N_4, NLTO/CN-1, NLTO/CN-2, NLTO/CN-3, and NLTO/CN-4 (c) Hydrogen production performance of pure $NaLiTi_3O_7$, pure g-C_3N_4, NLTO/CN-1, NLTO/CN-2, NLTO/CN-3, and NLTO/CN-4 (d) Fluorescence properties of NLTO/CN-1, NLTO/CN-2, NLTO/CN-3, and NLTO/CN-4