Electronic Supplementary Information (ESI)

Experimental

1. Preparation of porous g-C₃N₄

2 g Dicyandiamide (Alfa Aesar, 99% purity) was placed in a "ship-shape" crucible (6 cm×3 cm ×1 cm), and covered with another "ship-shape" crucible, forming a semi-closed system, whose "window" can be controlled by the position excursion of the two crucibles. Thereafter, the "semi-closed system" was heated at 550 °C for 4h in a muffle furnace (30 cm×20 cm×10 cm) with the ramping rate of 2.2 °C min⁻¹. Thus, the resulting yellow solid that was obtained from semi-closed system with the different sizes of window (cm²) was named as CNP*n*, where *n* represent the size of window.

2. Characterization

X-ray diffraction (XRD) pattern of sample was recorded on an ARL XTRA diffractometer (power 40 kV, 40 mA) using Cu-K α radiation in the 2-theta range from 6° to 90°. N₂ sorption isotherms were measured on a Micromeritics ASAP 2020 system at -196 °C, and the sample was evacuated at 300 °C for 4 h prior to test. The Brunauer–Emmett–Teller (BET) method was utilized to calculate the specific surface areas of sample using adsorption branches acquired at a relative pressure (p/p_0) or 0.99. The pore size distribution was derived from the desorption branch of the isotherm using the Barrett-Joyner-Halanda (BJH) algorithm. Transmission electron microscopic (TEM) images were gotten on a JEM-2100 (JEOL) electron microscope operating. Fourier Transform infrared spectra were recorded by a VERTEX 70 FTIR spectrometer with a resolution of 2 cm⁻¹. UV-Visible diffuse reflection spectra was measured on Shimadzu UV2550 and converted from reflection to absorbance using Kubelka-Munk method. Photoluminescence (PL) measurement was curried out at room temperature with a Varian Cary Eclips luminescence spectrometer, and the excitation wavelength was 350 nm. Elemental Analysis was performed on Heraeus-CHN-O-Rapid and all the samples were dried at 373 K over night before test. TG–DTA analysis of sample was conducted on the thermobalance (STA-499C, Netzsch)

Photocatalytic activity of $g-C_3N_4$ materials for methyl orange (MO) photodegradation was evaluated in a Pyrex reactor. 0.1 g sample was dispersed in MO aqueous solution (100 mL, 10 mg L⁻¹). The light irradiation system contains a Xe lamp with cutoff filter (420 nm) for visible light and a water bath to eliminate the temperature effect. Before the experiments of MO photodegradation, the reaction solutions were first stirred in dark for 30 min to reach the adsorption–desorption equilibrium of MO on catalysts. The concentration of MO with the time of light irradiation was detected by Shimadzu UV2550.



Figure S1. FT-IR spectra of CNP0 and CNP18 samples.



Figure S2. Nitrogen sorption isotherms (a) and pore size distributions (b) of these CNPn samples.



Figure S3. UV-vis diffuse reflection spectra of g-C₃N₄ samples.



Figure S4. TG–DTA of dicyandiamide from room-temperature to 550 $^{\circ}$ C at a heating rate of 10 $^{\circ}$ C min⁻¹.



Figure S5. Wide-angle XRD patterns of the products obtained at different temperature.