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Supporting Information

Stucture disorder of graphitic carbon nitride induced by liquid-assisted grinding for enhanced photocatalytic conversion

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Section S1: Experimental methods:

Synthesis of CN. The CN was prepared by a facile thermal treatment of dicyandiamide. Typically, 10 g of dicyandiamide powder was put into a covered crucible, and then heated at a rate of 5 °C min^{-1} to reach the temperature of 550 °C and maintained at this temperature in air for 2 h.

General Procedure for LAG-CN-x by Ball Milling CN in the Presence of Organic Solvent. The LAG-CN-x samples were carried out in a planetary ball-mill machine (QM-3SP2) in the presence of organic solvents with different polarities at 300 rpm. In the typical fabrication, 1 g of CN powders were added into an agate capsule containing agate balls of 5 mm in diameter. 20 ml of acetonitrile, CCl_4, ethanol, IPA, DMSO, or DI water was added into the above capsule as solvents assisted grinding. The container was fixed in the planetary ball-mill machine and agitated with 300 rpm for 12 h in all cases. The resultant products was collected by centrifugation, with deionized water and ethanol, and dried at 80 °C under vacuum for 24 h.

Characterization. The structures of the power samples were investigated by x-ray diffractometer (XRD, Bruker D8 Advanced Diffractometer operating with Cu Kα radiation). The angular range was 2θ = 5-65 °, with a speed of 3 °/min. Infrared transmission was obtained with a Fourier transform infrared (FTIR) spectrophotometer Spectrum (Nicolet). The optical absorbance spectra of the samples were performed on using a UV-vis spectrometer (CARY 500). Surface binding elements were analyzed with X-ray photoelectron spectroscopy (XPS, USA Thermo ESCALAB 250). All binding energies were referenced to C1s peaks (284.8 eV) arising from surface hydrocarbons (or possible adventitious hydrocarbon). Prior peak deconvolution, X-ray satellites, and inelastic background (Shirley-type) were subtracted for all

spectra. Transmission electron microscopy (TEM, JEM 2100, 200 kV) were used to characterize the morphology and structure of the obtained products. Element analysis was conducted by an elemental analyzer (vario EL Ⅱ; Elementar Analysensyteme, Germany). The photoluminescence measurements were performed in an Edinburgh instruments (FLSP 920) system operated at room temperature.

Photocatalytic test. The photocatalytic reaction was performed in a pyrex glass cell connected to a glass closed gas circulation system. The as-obtained products were loaded with 3 wt% Pt and calacinated at 180 °C for 30 min (ramp: 2 °C min^{-1}). H_2 evolution analysis was performed by dispersing 50 mg of catalyst power in an aqueous solution containing triethanolamine (100 mL, 10 vol.%) as the sacrificial electron donor. The reactant solution was evacuated several times to remove air completely before the reaction. A 300 W Xe lamp with a 420 nm cutoff filter was used as the light source. A flow of cooling water was used during the reaction to maintain the temperature of the reactant solution at 15 °C. The amount of H_2 evolved was analyzed by gas chromatography (TECHCOMP, 7890Ⅱ).

The apparent quantum yield (QY, %) for hydrogen evolution was measured using a similar experimental setup, with a 420 nm band-pass filter. The computing computing methods are as follows:

$$\text{QY (\%)} = N_e/N_p = 2N_{H_2}/N_p \qquad (1)$$

Here N_e stands for the number of reacted electrons, N_{H_2} is the number of evolved H_2 molecules and N_p is the number of incident photos.

Section S2: Crystal and chemical structures.

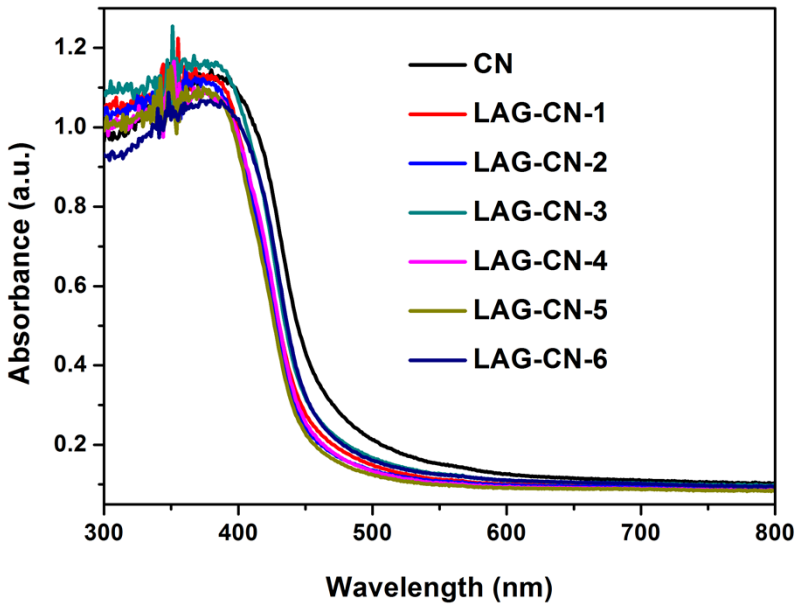

Fig. S1. UV-vis adsorption spectra of as-prepared CN and LAG-CN-x samples.

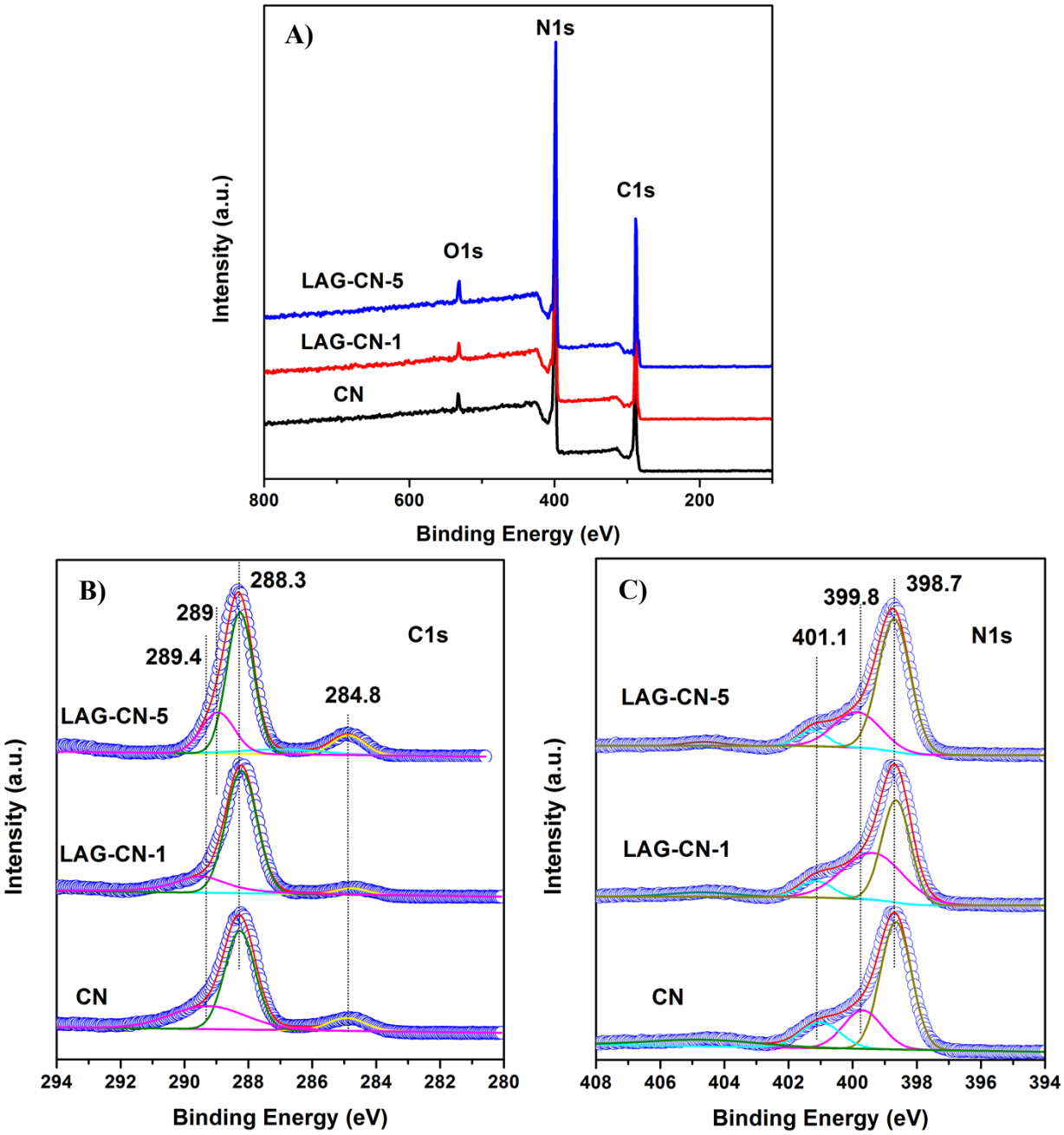

Fig. S2. XPS survey spectrum (A) and the corresponding high-resolution spectra of C1s (B), N1s(C) that are obtained from the CN, LAG-CN-1 and LAG-CN-5.

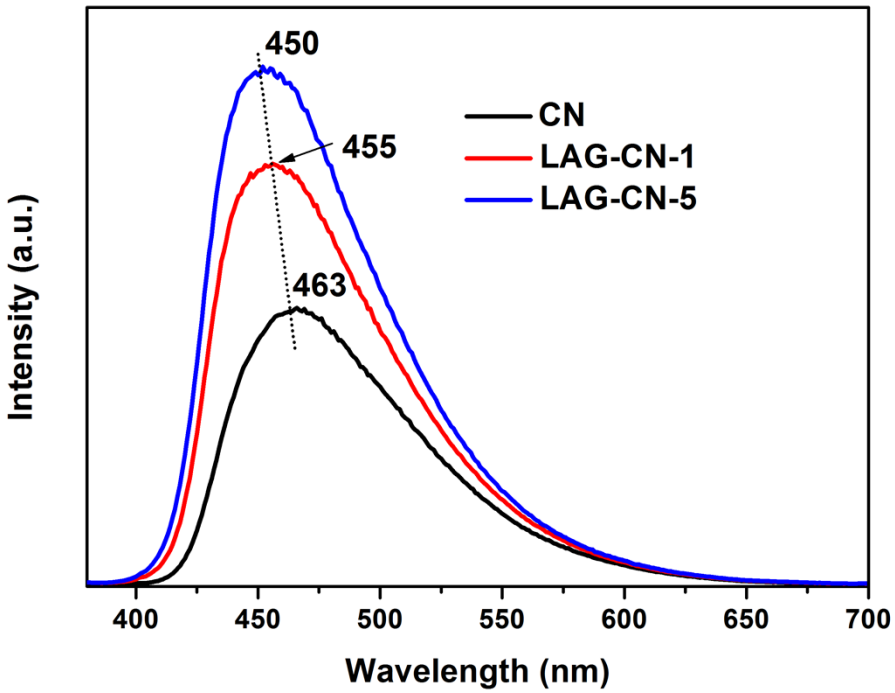

Fig. S3. Fluorescence emission spectra of the as-prepared samples. The wavelength of excitation light is 365 nm.