Support Information Experimental

Preparation of g-C₃N₃

2 g of melamine chloride and a roll of copper foil were placed in a reaction kettle lined with para-polyphenylene (PPL) and then heated at 260°C for 24h.

After the reaction, the polymer generated on the copper foil was scraped off and then washed three times with ethanol and pure water (80°C) in a centrifuge tube and centrifuged. The centrifuged samples were repeatedly washed with HNO₃ solution at a concentration of 10 wt%. The samples were then washed with HCl solution at a concentration of 10 wt%, deionized water and anhydrous ethanol, respectively, by ultrasonication three times in sequence. After drying, the brown product of g-C₃N₃ obtained ^[13].

Preparation of $g-C_3N_3/H_2SO_4$ solution

3 g g-C₃N₃ and 10 ml H_2SO_4 were mixed and stirred until completely dissolved, and the resulting solution was a dark brown solution. The concentration of protonated g-C₃N₃ in the dissolved solution was 300 mg-ml⁻¹. In all subsequent studies, concentrated sulfuric acid refers to 98 wt% sulfuric acid if not otherwise specified.

Preparation of g*- C₃N₃ recrystallization

Deionized water was added to the above brown solution until no more crystalline products were precipitated, filtered and then thoroughly washed with deionized water and dried at 60 °C. The product was denoted as $g^*-C_3N_3$.

Characterizations

The material phase structure was characterized by Shimadzu XRD-6000. The pore structure and specific surface area were characterized by Brunauer-Emmett-Teller (BET, Autosorb-IQ). The elemental valence states were tested by X-ray photoelectron spectroscopy (XPS) (Thermo Scientific Co. Ltd.). Functional groups were tested by Fourier transform infrared spectroscopy (FT-IR, Shimadzu IR Prestige-21). Basic physical properties of materials were tested by UV-Vis (Shimadzu UV-2450) absorption



spectroscopy and photoluminescence (PL, Fluoromax-4, Horiba Jobin Yvon) spectroscopy.

Fig. S1 Schematic diagram of the dissolution process of g-C₃N₃ in concentrated sulfuric acid

The sulfuric acid used in this work was a concentrated sulfuric acid at 98 wt% concentration. We added the effect of concentration on freezing point, when an additional 10 wt% of deionized water was added, there was no freezing at -40°C and no precipitation was produced. However, when an additional 20 wt% of deionized water was added, the solution had frozen. See the figure below for details. As shown in the figure below.



S2 10 wt% (a) and 20 wt% (b) of deionized water was added (-40°C)