

Electronic Supplementary Information (ESI)

Direct conversion of urea into graphitic carbon nitride over mesoporous TiO₂ spheres under mild condition

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Experimental Section

Synthesis of titanium glycolate precursor: The titanium glycolate precursor was prepared at room temperature by following the previously reported procedures.²³ Typically, the mixture of titanium *n*-butoxide (2 mL) and ethylene glycol (50 mL) was poured into a solution containing acetone (170 mL) and water (2.7 mL) and was stirred at room temperature to form the titanium glycolate precursor. After 1 h, the white precipitate was washed several times with ethanol and dried in an oven at 60 °C.

Light-driven synthesis of mesoporous TiO₂ spheres: The titanium glycolate precursor (4.0 g) was dispersed in water (400 mL) and then exposed to the UV-light irradiation for 2 h. After the irradiation, the solid sample was harvested and dried in air. The obtained product was the mesoporous TiO₂ spheres described in the title of this communication and was designated as TiO₂-UV. The UV-light source used in the experiment was a 400 W high-pressure mercury lamp (main output at 313 nm).

Control experiments: a) The TiO₂-1 sample was prepared by calcining the titanium glycolate precursor at 500 °C for 2 h.²² b) The TiO₂-2 sample was prepared through *in situ* hydrolysis of the titanium glycolate precursor at 100 °C for 2 h.²³

Formation of C₃N₄ from urea: The formation of C₃N₄ from urea was achieved via heating the mixture of TiO₂-UV (0.1 g) and urea (0.5-3 g) in air at about 300 °C for 2 h. To prepare pure C₃N₄, the resulting orange-tan powder was treated with HF (5.0 wt%) for 24 hours at room temperature to remove TiO₂. Subsequently the sample was treated with HNO₃ acid (3 mol/L) at 80 °C for 1 h to remove the byproducts (cyanuric acid).

For comparison, control experiments were performed by replacing TiO₂-UV with TiO₂-1, TiO₂-2, P25, MCM-41, SBA-15, or NaOH, while other reaction conditions were identical. These control experiments all failed to yield C₃N₄.

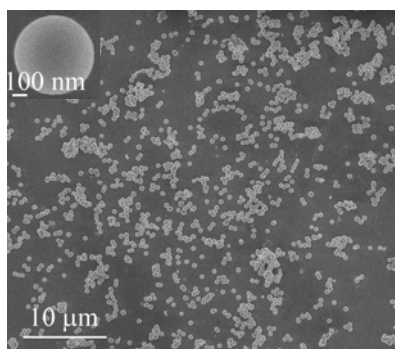


Figure S1. SEM images of the titanium glycolate precursor.

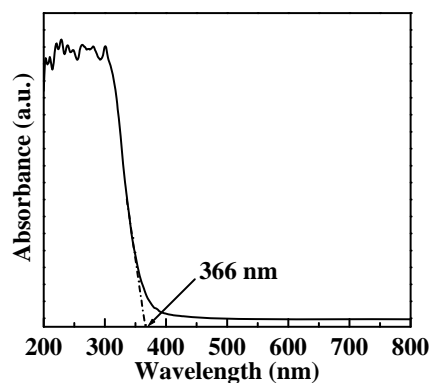


Figure S2. The UV/vis absorption spectrum of the titanium glycolate precursor obtained from the diffuse reflectance one through Kubelka-Munk transformation.

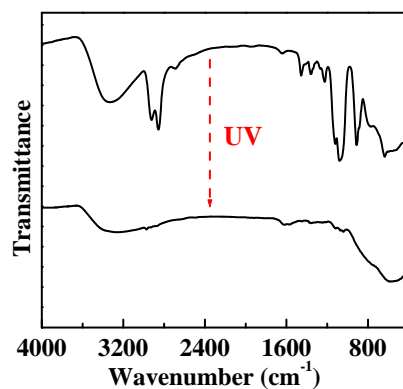


Figure S3. IR spectra of the titanium glycolate precursor before and after UV irradiation.

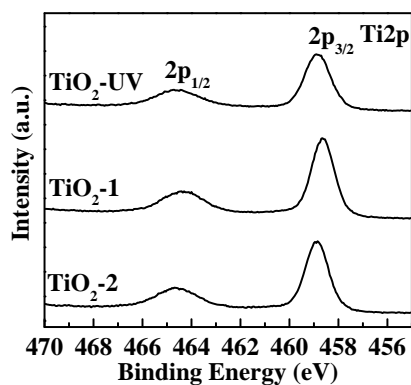


Figure S4. High-resolution XPS spectra of Ti2p for TiO₂-UV, TiO₂-1 and TiO₂-2.

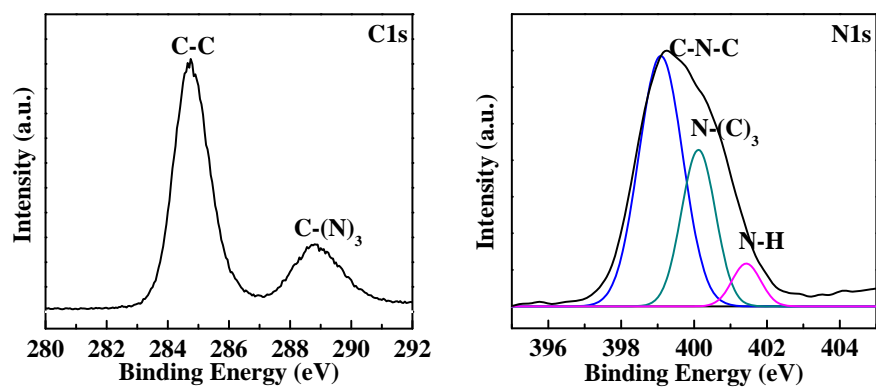


Figure S5. High-resolution XPS spectra of C1s (a) and N1s for the as-prepared C₃N₄ sample. The peak at 284.8 eV in the C1s spectrum is due to adventitious elemental carbon from the XPS instrument itself.