Electronic Supplementary Material (ESI)

Template-free synthesis of nanocage-like $g-C_3N_4$ with high surface area and nitrogen defects for enhanced photocatalytic H₂ activity

Mao Wu, ^a Yansheng Gong, ^{a, b,} * Tao Nie, ^a Jin Zhang, ^a Rui Wang, ^a Huanwen Wang^a and Beibei He^{a, **}

^a Faculty of Materials Science and Chemistry, China University of Geosciences, Wuhan 430074, China

^b Engineering Research Center of Nano-Geomaterials of Ministry of Education, China University of Geosciences, 388 Lumo Road, Wuhan 430074, China

*Corresponding authors:

E-mail addresses: gongysh@cug.edu.cn (Y.S. Gong);

babyfly@mail.ustc.edu.cn (B.B. He)



Fig. S1. SEM images of the commercial dicyandiamide.



Fig. S2. FTIR spectra of the pressor treated by HCl and without treated.



Fig. S3. (a) SEM, (b) TEM images, and (c) photocatalytic H₂ evolution of the carbon nitride prepared via the precursor without HCl-treated.





Fig. S4. Atomic concentrations for C, N and O elements of bulk g-C₃N₄, DCN-550 and DCN-600 derived from the EDX spectra.



Fig. S5. (a) SEM images and (b) XRD spectra of DCN-550 after four cycles of photocatalytic reaction.



Fig. S6.TG curves of the precursor for the samples with and without HCl-treatment..



Fig. S7. Solid-state 13C MAS NMR spectra of the intermediate products at three

different stages.



Fig. S8.TEM images of DCN-550 after loaded with 5 wt.% Pt.

Table S1. Atomic concentrations for C and N elements of bulk g-C₃N₄, DCN-550 and DCN-600 derived from the organic elemental analysis (OEA).

sample	C (wt.%)	N (wt.%)	C/N atomic ratio
Bulk g-C ₃ N ₄	34.98	61.67	0.66
DCN-550	36.40	58.63	0.72
DCN-600	35.96	61.58	0.68

Table S2. Atomic concentrates for C, N and O elements of bulk g-C₃N₄, DCN-550 and DCN-600 derived from the XPS spectra.

Sample	C (At%)	N (At.%)	O (Wt.%)	C/N
Bulk g-C ₃ N ₄	40.7	58.1	1.40	0.700
DCN-550	43.6	54.31	2.54	0.803
DCN-650	42.21	56.21	1.92	0.751

Table S3. XPS N1s spectra results of bulk g-C₃N₄, DCN-550 and DCN-600.

Sample	N-H	N-(C)3	C-N=C
Bulk g-C ₃ N ₄	11.19%	23.88%	64.93%
DCN-550	10.51%	19.81%	69.68%
DCN-650	9.37%	19.85%	70.78%

Sample	τ ₁ [ns]	$\tau_2 [ns]$	B ₁	B ₂	τ_{av} [ns]
Bulk g-C ₃ N ₄	2.2423	8.0063	585.963	165.415	5.13
DCN-550	1.8120	8.7063	701.689	248.494	6.15
DCN-600	1.8316	8.4693	926.859	302.265	5.82

Table S4. PL decay fitting data of bulk $g-C_3N_4$, DCN-550 and DCN-600.