Hierarchical macro-mesoporous $g-C_3N_4$ with inverse opal structure and vacancy for high-efficiency solar energy conversion and environmental remediation

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TEXT

Method of detection of degradation intermediates

Ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) Determination of the photodegradation intermediates of LVX were carried out on a UPLC–MS/MS system equipped with a UItiMate 3000 UPLC (Thermo Scientific) and a Q Exactive Plus MS/MS mass analyzer (Thermo Fisher Scientific). The UPLC column was a Hypersil Gold C18 column (2.1×100 mm) from Thermo Scientific. 0.1% of formic acid aqueous solution and acetonitrile were used as mobile phases A and B, respectively. The eluent flow rate kept 0.2 mL/min. An injection volume of 10 µL was used in analysis and column temperature at 302 K (30 °C). Linear gradient elution was as follow: the initial 93% A was reduced to 50% A over 25 min. Then mobile phase A returned to 93% in 30 min. A positive ionization mode electro-spray interface was used. The other parameters were set as the following: the ESI was done at 4 kV at temperature of 350°C; the gas (N₂) flow rate was 12 L min⁻¹, and nebulizer was 35 psi.

FIGURES



Figure S1. SEM images of (a) Three-dimensional ordered self-assembly silica microspheres, (b) Bulk CN, (c) Nv CN, and (d) IO CN.



Figure S2. (a) Nitrogen adsorption–desorption isotherms and (b) BJH pore size distribution of different samples.



Figure S3. The TEM of Nv CN.



Figure S4. The corresponding Tauc plots of Bulk CN and Nv MM CN samples.



Figure S5. Mott-Schottky plots (the Mott-Schottky measurement was conducted at 1000 Hz in the dark)



Figure S6. MS spectra of the NOR and possible intermediates



Figure S7. (a), (b) TEM and (c) SEM of Nv MM CN after five cycles, (d) XRD patterns of Nv MM CN before and after reaction.

TABLES			
Sample	C/N ratio		
Bulk CN	0.67		
Nv CN	0.69		
IO CN	0.71		
Nv IO CN	0.74		

 Table S1.
 Elemental Analysis of different samples.

Sample	τ1 (ns)	Rel %	τ2 (ns)	Rel %	τ (ns)
Bulk CN	0.14	95.33	5.04	4.67	1.175
Nv CN	0.08	98.05	5.28	1.95	1.106
IO CN	0.11	97.65	5.11	2.35	0.987
Nv IO CN	0.10	97.88	4.75	2.12	0.971

Table S2. Time-Resolved Fluorescence Decay Spectra of different samples

Sample	Pt loading	Light Source	HER µmol h ⁻¹ g ⁻¹	Reference
Nv MM CN	3 wt %	AM 1.5	12864	This work
Nv MM CN	3 wt %	420 nm	1019	This work
CNC0.1	1 wt %	420 nm	212.8	1
gC ₃ N ₄ -425	1 wt %	420 nm	779	2
p-CN2	3 wt %	420 nm	396	3
CN-0.1	3 wt %	420 nm	277	4
CCN/T-1.5	1 wt %	AM 1.5	625.5	5
Pt/C ₃ N ₄	3.5 wt %	AM 1.5	~2400	6
HC-CN	3 wt %	420 nm	808.5	7
DCDA- based g- C ₃ N ₄	3 wt %	Without filter	1350	8
MCN-560	3 wt %	420 nm	530	9
UGCNPs	3 wt %	420 nm	1365	10

Table S3. Summary of g-C $_3N_4$ -based photocatalysts for hydrogen evolution rate (HER).

Compounds	Formula	m/z	Proposed structure
NOR	C ₁₆ H ₁₈ FN ₃ O ₃	320	F CH
NOR1	C ₁₆ H ₁₆ FN ₃ O ₅	350	
NOR2	$C_{16}H_{18}FN_3O_4$	336	
NOR3	C ₁₅ H ₁₈ FN ₃ O ₄	324	
NOR4	C ₁₅ H ₁₆ FN ₃ O ₄	322	
NOR5	$C_{13}H_{12}N_2O_4$	261	
NOR6	C ₁₂ H ₁₁ FN ₂ O ₃	251	
NOR7	C ₁₀ H ₇ NO ₄	206	

Table S4. The possible intermediates products of NOR degradation¹¹⁻¹⁴

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