

## Supporting Information

### **Defect engineering synthesis of oxygen doped carbon nitride microtubules for enhanced visible-light-driven photocatalysis**

Li Zhou,<sup>a</sup> Menghan Xu,<sup>a</sup> Wenjie Zhang,<sup>a</sup> and Xiaoquan Yao,<sup>\*a</sup>

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Department of Applied Chemistry, School of Material Science and Technology, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, P. R. China.

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\*Corresponding author:

E-mail addresses: yaoxq@nuaa.edu.cn (X. Yao)

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## **Experimental section:**

### **2.1. Chemicals and materials**

In this research, Melamine (MA,  $C_3H_6N_6$ , 99%), Hydrogen peroxide ( $H_2O_2$ , 30%) and Acetonitrile ( $C_2H_3N$ , 99%) were provided by Beijing Inno Chem Science & Technology Co., Ltd. Tetracycline ( $C_{22}H_{24}N_2O_8$ , abbreviated as TC, 98%) and chloroplatinic acid hydrate ( $H_2PtCl_6 \cdot 6H_2O$ , 99.9%) were purchased from Aladdin. Triethanolamine ( $C_6H_{15}NO_3$ , 99%), L-Ascorbic acid ( $C_6H_8O_6$ , 99%), and isopropanol ( $C_3H_8O$ , 99%) were purchased from Macklin Co., Ltd. All other chemical reagents involved were of analytical grade and employed without further purification. Meanwhile, deionized water ( $>18.2 \text{ M}\Omega \cdot \text{cm}$ ) was obtained from a Milli-pore Milli-Q system (High-tech, Shanghai) and used throughout the experiments.

All instruments and characterisations adopted in this experiment are detailed in the “Supporting Information” section.

### **2.2 Measurement and Characterization**

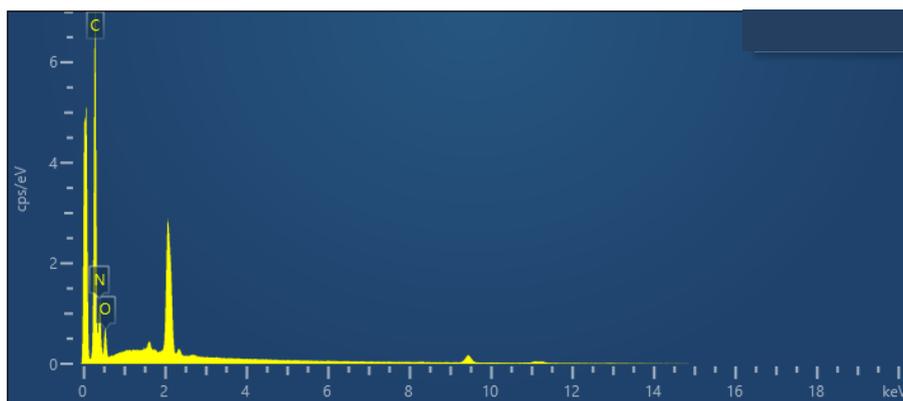
The crystal phase, photoelectric properties, elemental compositions, and morphologies were performed using an X-ray diffraction (XRD) spectroscope (Bruker D8 ADVANCE), UV–vis spectroscopy (JP Shimadzu Co., Ltd), scanning electron microscope (SEM) (Mira 4, Tescan) with energy-dispersed spectroscopy (EDS, Oxford Ultim Max65), and X-ray photoelectron spectroscopy (XPS) (250Xi, Thermo Fisher). Fourier transform infrared spectra (FT-IR) were analyzed by Thermo Scientific Nicolet iS20. The specific surface areas were determined with a surface area analyzer (ASAP 2020 Micropore System, Micromeritics Instrument Corporation, USA) by the Brunauer-Emmett-Teller (BET) method. Steady-state photoluminescence (PL) and time-resolved photoluminescence (TRPL) spectra were recorded using a spectroscopic instrument (FLS1000, Edinburgh Instruments Ltd., UK) with an excitation wavelength of 375 nm at room temperature. A 375 nm laser in timecorrelated single-photon counting (TCSPC) mode was used to measure the decay lifetime of the PL spectra. The generation of superoxide radicals ( $\bullet O_2^-$ ) was verified by electron paramagnetic resonance EPR (Bruker E500, Bruker BioSpin GmbH)

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measurements using 5,5-dimethyl-1-pyrroline N-oxide (DMPO) as the spin-trapping agent.

**Photoelectrochemical Measurement:** Photoelectrochemical tests were conducted in a conventional three-electrode cell at room temperature and pressure using an electrochemical workstation (CHI660D, Shanghai, China). The electrolyte used was 0.5 M Na<sub>2</sub>SO<sub>4</sub>, with the Ag/AgCl reference electrode, the Pt counter electrode, and the working electrode of the samples under investigation. 2 mg of the sample was added to 180 μL of ethanol and 20 μL of Nafion and mix with ultrasound. The solution was mixed and drop-cast onto FTO glass with a fixed area (1 cm<sup>2</sup>). The electrode was dried to obtain the working electrode for subsequent testing. Transient photocurrent measurements were recorded at a potential of 0.4 V (versus Ag/AgCl) under irradiation from a 300 W xenon lamp equipped with a simulated solar filter.

**Photocatalytic hydrogen evolution from water splitting:** In the photodeposition process of Pt (0.5 wt %) on the sample, 40 mg of the sample was dispersed through ultrasonication in a methyl alcohol aqueous solution (V<sub>water</sub>:V<sub>methanol</sub>=55 mL : 15 mL) within a reaction cell. Subsequently, a solution of H<sub>2</sub>PtCl<sub>6</sub>·6H<sub>2</sub>O at a concentration of 0.5 M was added to the reaction cell. The system was then connected to the evaluation system. The evaluation of photocatalytic overall water splitting performance, was conducted in a closed gas circulation system equipped with an overhead-irradiation-type glass vessel. Prior to each test, all the air should be removed and 1 kPa Ar was injected for facilitating the detection of little generated gases. A 300 W Xe lamp was adopted as the irradiation source (equipped with a 420 nm cut-off filter). During each reaction, the suspension was kept at 25 °C, and the evolved gases were analyzed by gas chromatography (GC-2014c).



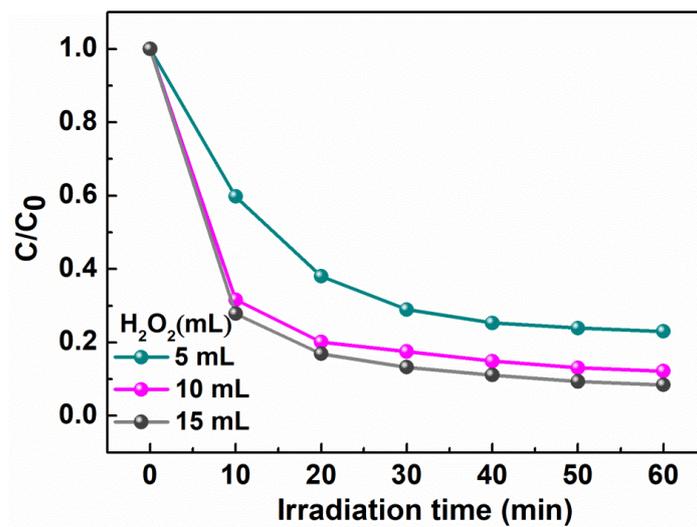
**Fig S1.** The EDX element distribution mapping of C、 N and O in OCN

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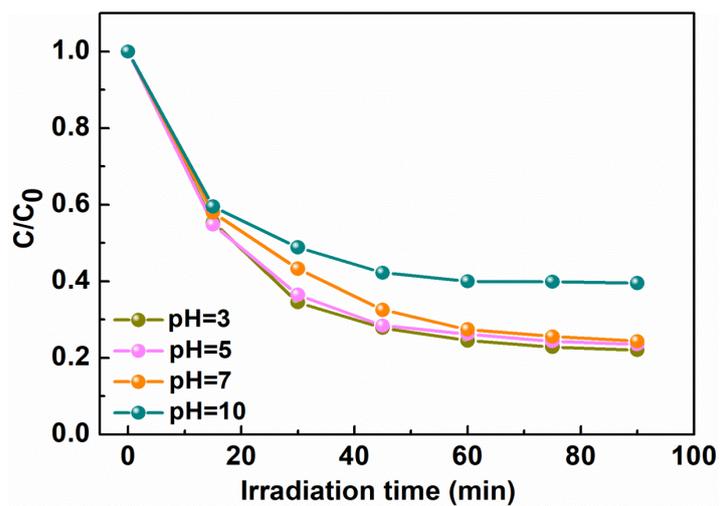
**Table S1.** Element content table of OCN

<b>Element</b>	<b>Wt%</b>	<b>Wt% Sigma</b>	<b>At%</b>
C	51.09	0.36	55.6
N	38.32	0.40	35.76
O	10.59	0.20	8.65
total	100.00		100.00

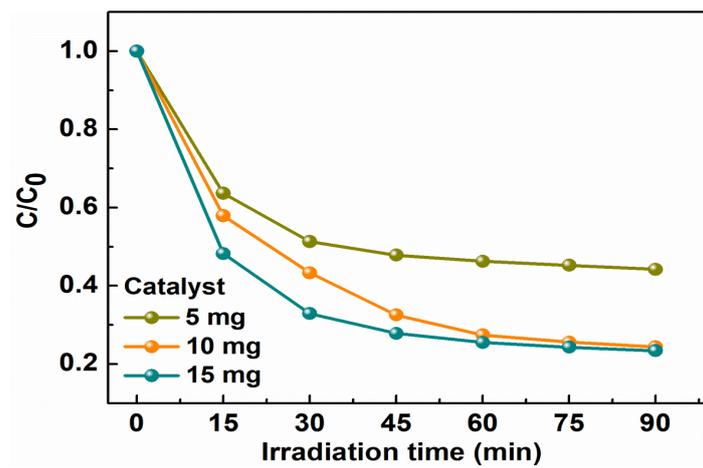
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**Fig S2.** Photocatalytic degradation of TC by OCN synthesized with different amounts of hydrogen peroxide



**Fig S3.** Photocatalytic degradation of tetracycline by pH of different solutions



**Fig S4.** Photocatalytic degradation of tetracycline by catalysts of different quality

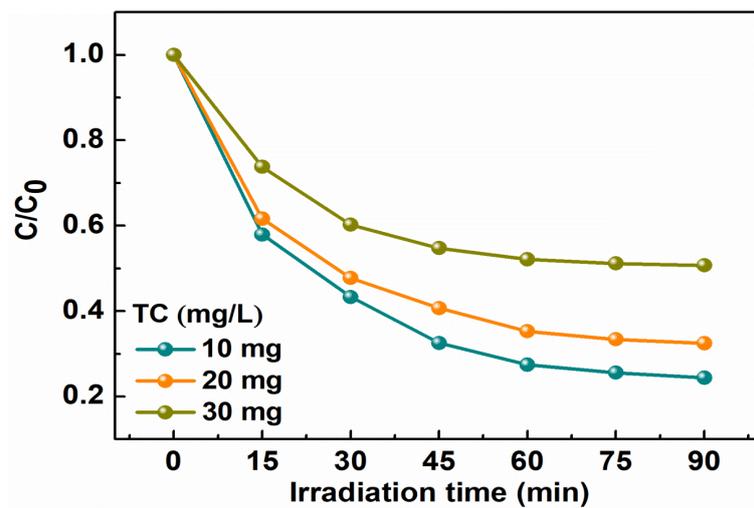
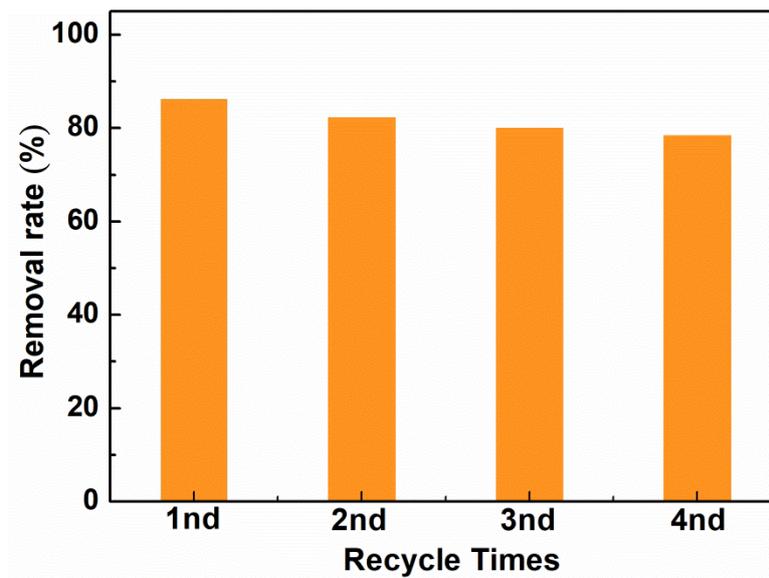


Fig S5. Photocatalytic degradation of tetracycline at different mass concentrations

**Table S2.** Comparison of photocatalytic degradation efficiency of tetracycline with OCN and other g-C<sub>3</sub>N<sub>4</sub> samples

<b>Catalyst</b>	<b>Dosage (mg)</b>	<b>Light source</b>	<b>Initial amount</b>	<b>Time (min)</b>	<b>Removal (%)</b>
GG-N <sup>[1]</sup>	2000	100W UV mercury lamp	50 mg/L	180	91.57
Ag/g-C <sub>3</sub> N <sub>4</sub> <sup>[2]</sup>	50	300W, Xenon ( $\lambda \geq 420$ nm)	20 mg/L	120	83
BiVO <sub>4</sub> /g-C <sub>3</sub> N <sub>4</sub> <sup>[3]</sup>	20	300W, Xenon	20 mg/L	60	56
CeNCN <sup>[4]</sup>	10	300W, Xenon ( $\lambda \geq 420$ nm)	10 mg/L	60	80.09
pg-C <sub>3</sub> N <sub>5</sub> <sup>[5]</sup>	40	300W, Xenon ( $\lambda \geq 420$ nm)	30 mg/L	60	83
OCN	10	300W, Xenon ( $\lambda \geq 420$ nm)	20 mg/L	60	87.7



**Fig S6.** Stability experiments of photocatalytic degradation of tetracycline of OCN

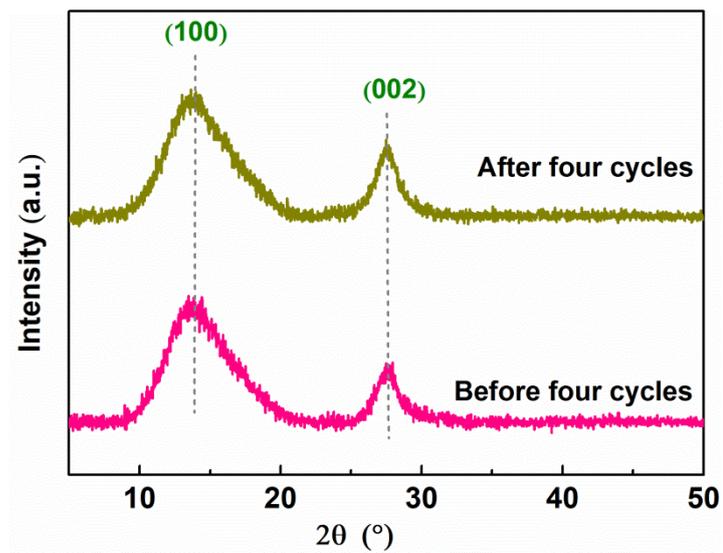
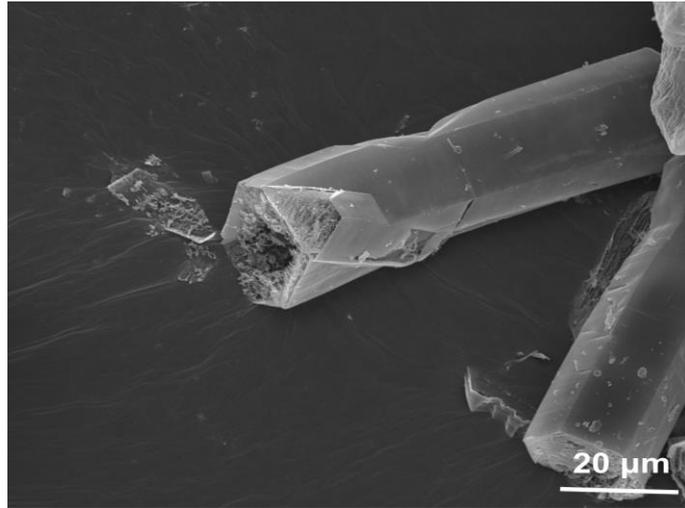
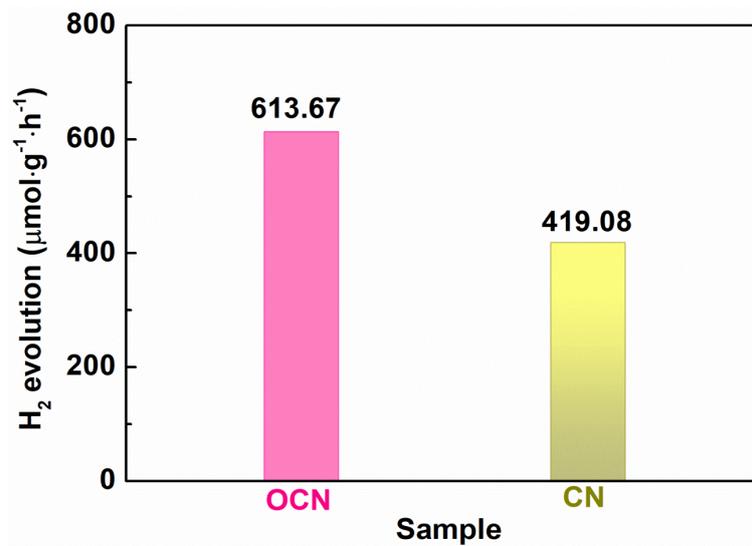


Fig S7. XRD patterns after cycles of OCN



**Fig S8.** SEM images of OCN after cycles



**Fig S9.** The rate of hydrogen production from photocatalytic water.

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## Reference

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