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Electronic Supplementary Information For

Visible-Light Responsive Electrospun Nanofiber Based on Polyacrylonitrile-Splited Graphitic Carbon Nitride

Tiefeng Xu, Fei Wu, Yan Gu, Yi Chen, Jinting Cai, Wangyang Lu, Hongguang Hu, Zhexin Zhu, and Wenxing Chen**

National Engineering Lab for Textile Fiber Materials & Processing Technology
(Zhejiang), Zhejiang Sci-Tech University, Hangzhou 310018, China

E-mail: luwy@zstu.edu.cn(W. Lu); wxchen@zstu.edu.cn(W. Chen)

Materials.

Urea (99%), Polyacrylonitrile (PAN) (AR), N, N-dimethylformamide (DMF) (99.5%), Rhodamine B (RhB) (AR), perchloric acid (AR), NaOH (96%), isopropanol (IPA) (99.7%), KI (99.99%), KBr (AR), benzoquinone (BQ) (97%)

Synthesis of g-C_3N_4.

30 g urea was contained in a semi-closed quartz crucible then was directly heated to 550 °C for 3 h at a heating rate of 2.5 °C/min in a tube furnace. The resultant pale yellow powder was g-C_3N_4 and collected for use without further treatment.

Synthesis of g-C_3N_4/PAN nanofibers

A series of spinning solution was prepared with different g-C_3N_4 concentration as shown in Table 1. A certain amount of g-C_3N_4 powder was dispersed in DMF and then ultrasound for about 5 h. Subsequently, the PAN powder was added into the dispersion and the mixture was ultrasound for another 5 h to form a homogeneous dispersion. The as-prepared spinning solutions were injected into a 10 mL hypodermic syringe and the solution syringe rate was 1 mL/h. The tip-to-collector distance was 15 cm with electrospinning voltage of 20 kV.

Analysis of intermediates

The intermediates formed during RhB degradation photocatalyzed by g-C_3N_4/PAN nanofiber were identified by UPLC Synapt G2-S HDMS (Waters, USA), which equipped with an electrospray ionization (ESI) source. The ion mode was set both on positive and negative mode. In the positive mode, the MS conditions were as follows: capillary voltage 3 kV, source temperature 120 °C, and desolvation temperature 400 °C. High purity nitrogen gas was used as both cone and desolvation gases at a flow rate of 50 and 900 L/h, respectively. Chromatographic separation was conducted with a BEH Amide column (1.7 μm, 2.1×100 mm) using mobile phases A (0.1% formic acid and water) and B (acetonitrile). The eluent gradient began with 60% A at 0 min, where it was held for 1.0 min, followed by a decrease to 30% A at 7 min, and in the next 1 min it remained at 30% A, finally reaching 60% A at 8.1 min. The flow rate was 0.4 mL/min, and the column oven temperature was set at 30 °C. 8 μL solution was injected using the auto sampler. In the negative mode, the conditions were as follows: capillary voltage 2.5 kV, source temperature 120 °C, and desolvation temperature 400 °C. High purity nitrogen gas was used as both cone and desolvation gases at a flow rate of 50 and 900 L/h, respectively. Chromatographic separation was conducted with the same column using mobile phases A (water) and B (acetonitrile). The gradient began with 5% A at 0 min, where it increased to 20% A and held for 5 min. It followed by a increase to 40% A at 8.5 min and in the next 0.5 min it remained at 40% A, finally reaching 5% A at 9.1 min. The flow rate was 0.3 mL/min, and the column oven temperature was set at 30 °C. The parameters for UPLC Synapt G2-S HDMS analyses were as follow: ESI ion mode, lockmass correction of leucine enkephaline (LE, Tyr-Gly-Gly-Phe-Leu, negative m/z 554.2615, positive m/z 556.2771), and the scan range was set to 50–1000 m/z. For further analyzing the photocatalyzed products of RhB, 100 ml of RhB solution (2×10^{-5} M) in the presence of g-C_3N_4/PAN nanifiber after 180 min of reaction was withdrawn and the water was removed by rotary evaporation. The pretreatment process was as follows: The reaction solution was filtered and concentrated by rotary evaporation. The 2 mL residue was added by HCl to adjust the pH < 2, and was added by NaOH to adjust the pH > 10, respectively. After extracting the solution by dichloromethane three times, Na_2SO_4 was used to remove the water in the extracts, and the dichloromethane was removed by rotary evaporation. The dry residue was then dissolved in 2mL methanol. Methyl esterification was carried out using 0.5 mL of trimethylsulfonium hydroxide at 50°C for 30 min. GC-MS analysis was conducted on an Agilent 7890B gas chromatography interfaced with 5977A mass spectrometer. The DB-5MS column (30 m×0.25 mm×0.25μm) was used, employing Helium as carrier gas. The analysis used an experimentally optimized column temperature program: 35 °C, held for 5 min, raised to 100 °C at a rate of 10 $°C·min^{-1}$, then raised to 280 °C at a rate of 20 $°C·min^{-1}$, giving a 20 min run time.

Table S1. Recipe of the electrostatic spinning solution.

Samples	g-C_3N_4 (g)	PAN (g)	DMF (g)
g-C_3N_4 /PAN	0.00	0.608	7.60
5% g-C_3N_4 /PAN	0.0304	0.608	7.60
10% g-C_3N_4 /PAN	0.0608	0.608	7.60
15% g-C_3N_4 /PAN	0.0912	0.608	7.60

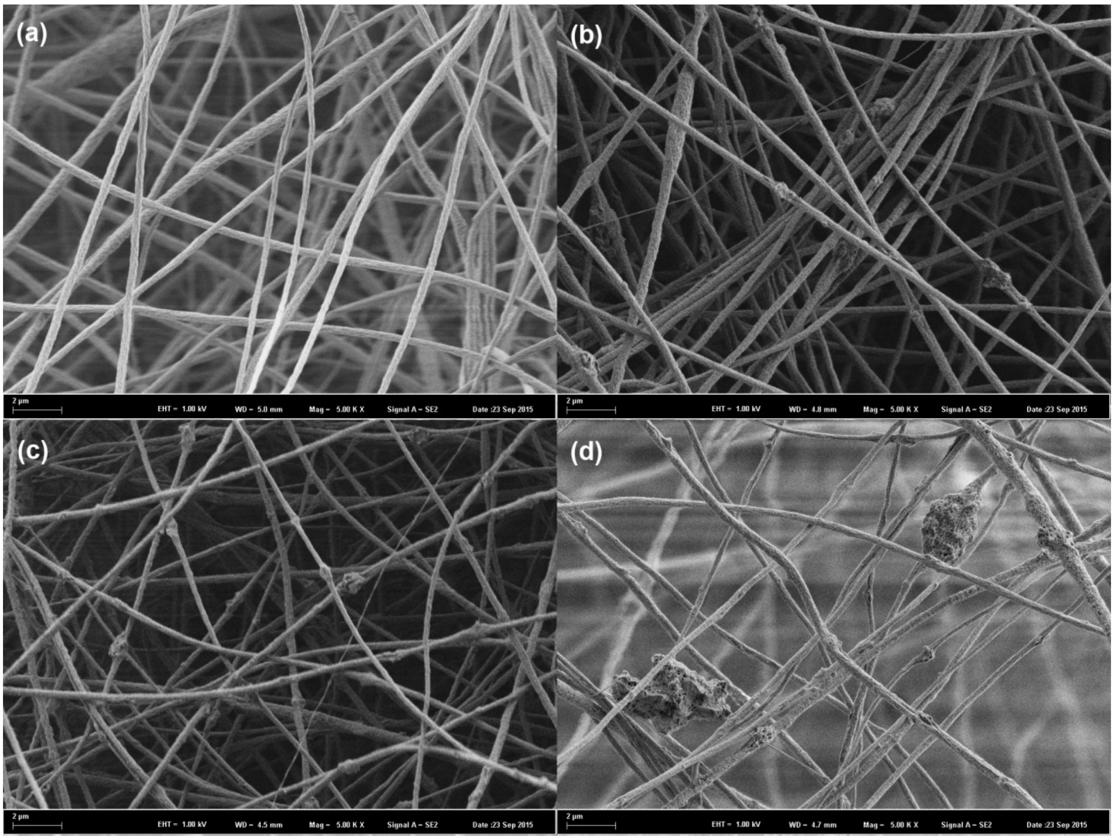


Fig.S1. FESEM images of g-C_3N_4/PAN nanofibers with different g-C_3N_4 concentrations: (a) PAN nanofibers; (b) 5% g-C_3N_4/PAN nanofibers; (c) 10% g-C_3N_4/PAN nanofibers; (d) 15% g-C_3N_4/PAN nanofibers.

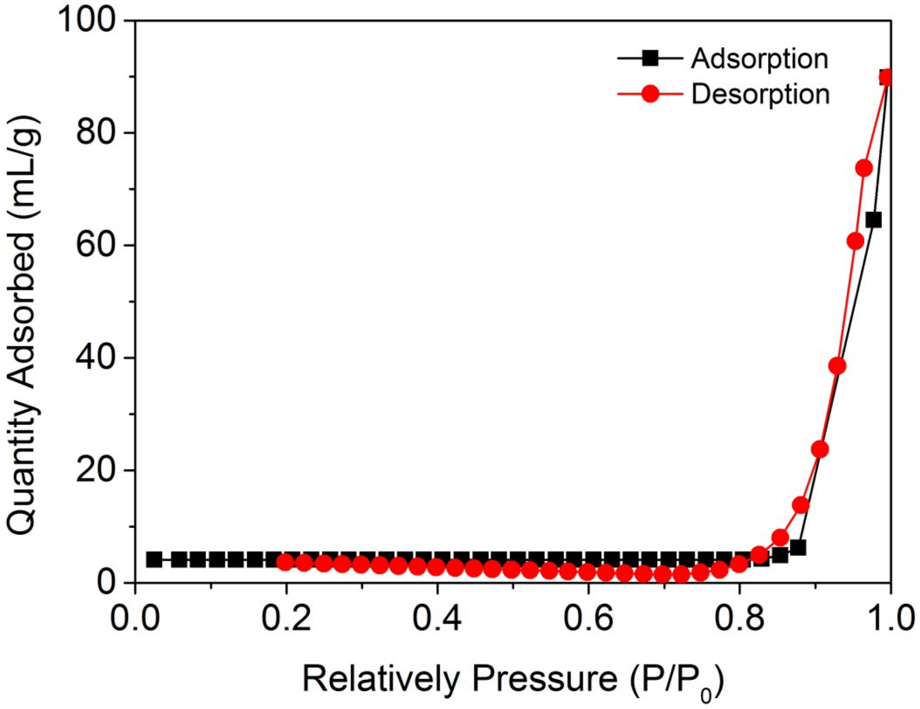


Fig.S2. N_2 adsorption–desorption isotherms of 10% g-C_3N_4/PAN nanofibers.

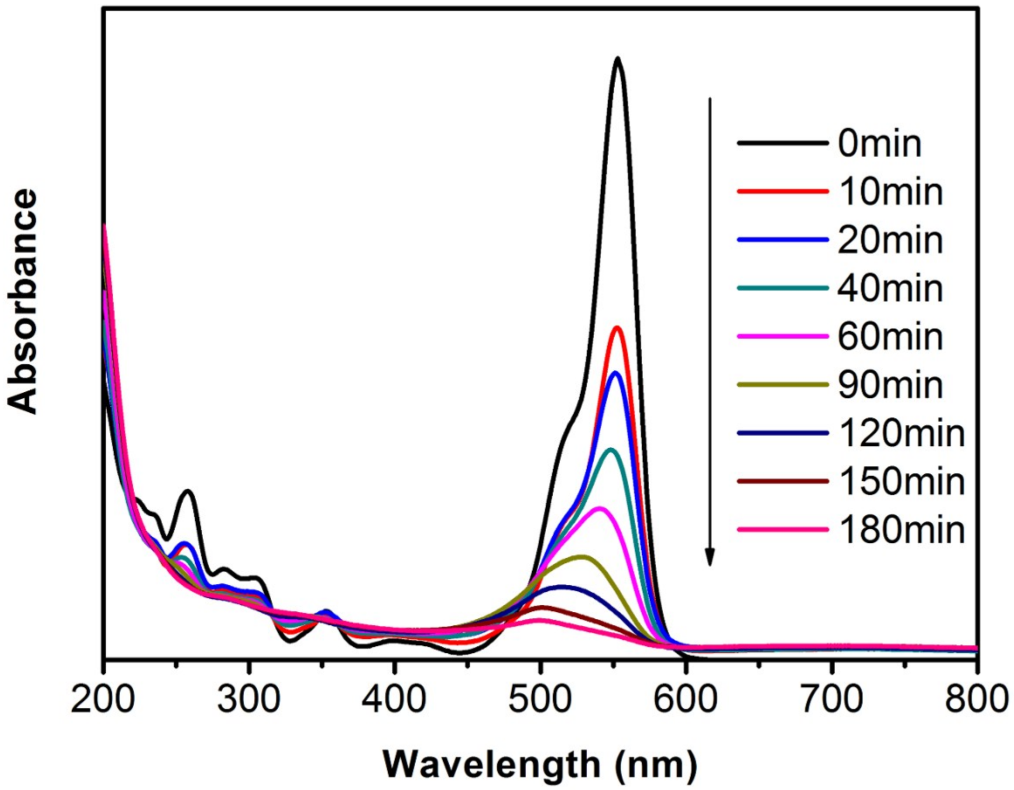


Fig. S3. Spectral changes during photocatalytic degradation of RhB under visible light irradiation (λ>400 nm). (RhB: 2×10^{-5} M, pH 5.6, 10% g-C_3N_4 /PAN: 1 g/L)

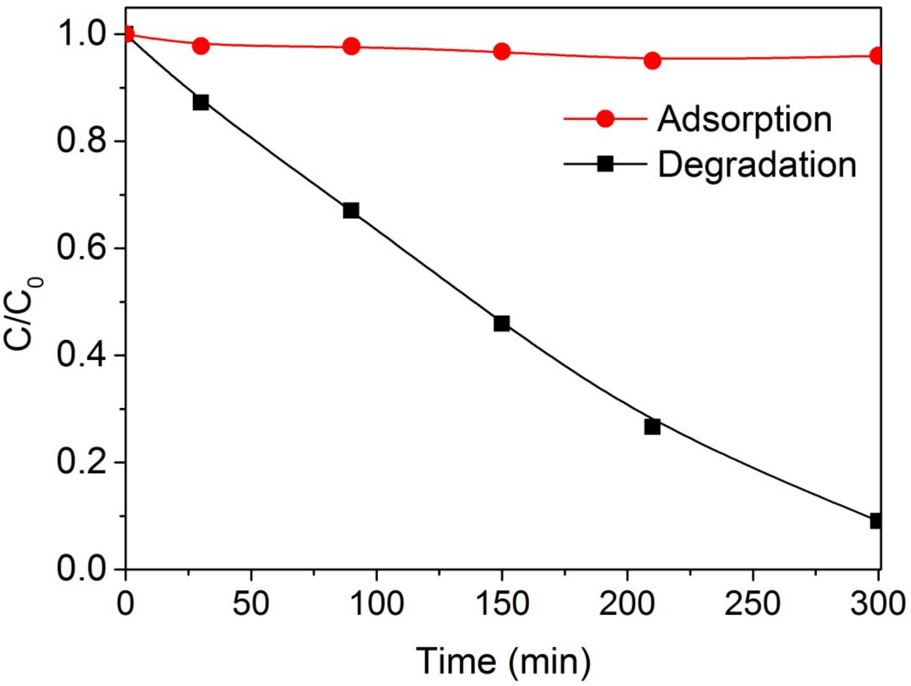


Fig.S4. The adsorbtion and photocatalytic degradation of 4-CP in the presence of g-C_3N_4/PAN nanofibers under visible light irradiation (λ>400 nm). (4-CP: 2×10^{-5} M, pH 11, 10% g-C_3N_4 /PAN: 1g/L.). The change of 4-CP concentration was measured by ultra-performance liquid chromatography with photodiode array detector (UPLC-PDA, Waters, USA).

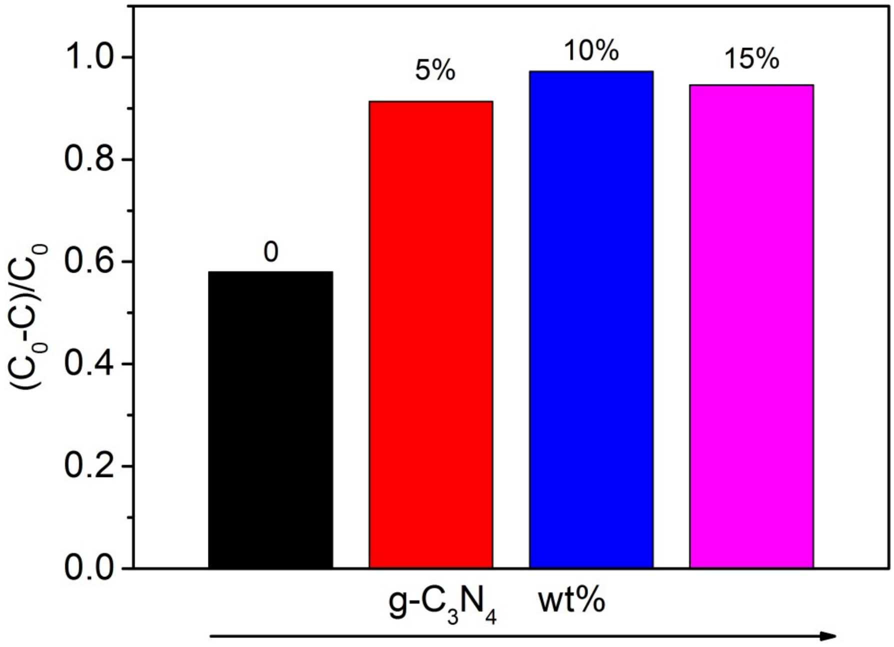


Fig.S5. The removal rate for the photodegradation of RhB in the presence of pure PAN and g-C_3N_4/PAN nanofibers with the different amount of g-C_3N_4 after 120 min under visible light irradiation (λ>400 nm). (RhB: 2×10^{-5} M, pH 5.6, PAN: 1 g/L, g-C_3N_4 /PAN: 1 g/L.)

Fig. S6. The photograph of photocatalytic degradation of RhB by g-C_3N_4 powder and g-C_3N_4/PAN nanofibers under solar irradiation. (Average sunlight intensity: 46760 lux, RhB: 2×10^{-5} M, g-C_3N_4:0.03 g/L, 10% g-C_3N_4/PAN: 0.3 g/L)

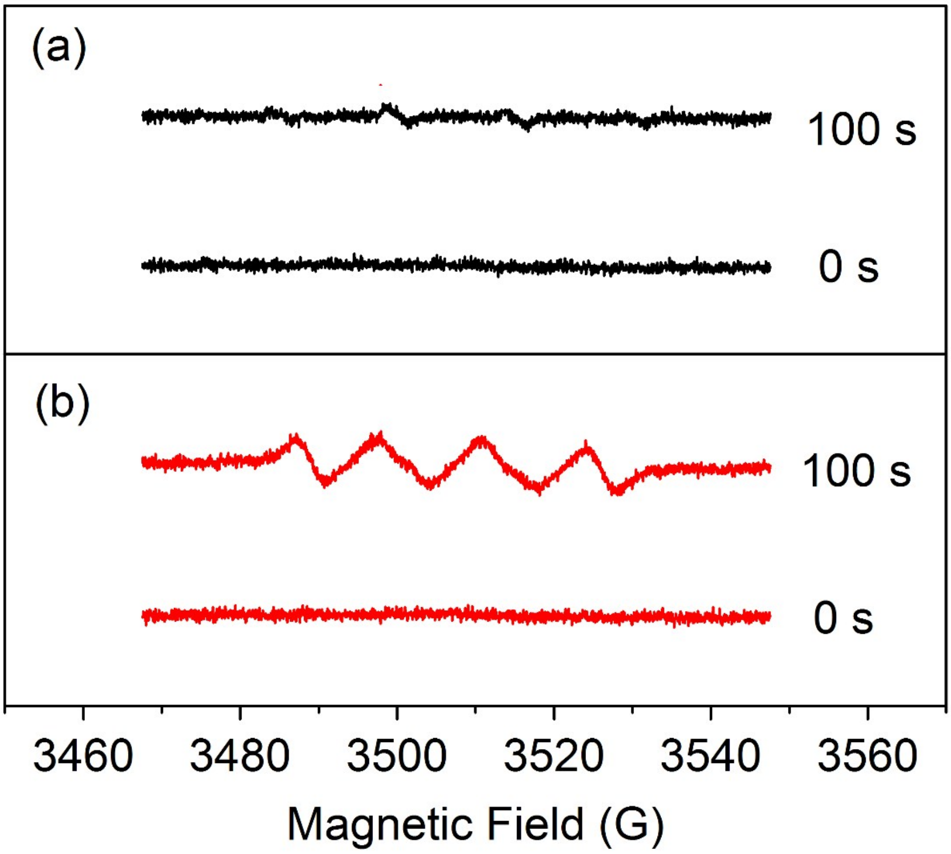
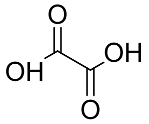
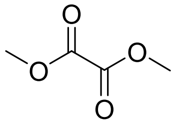
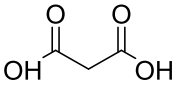
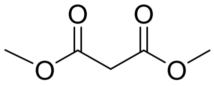
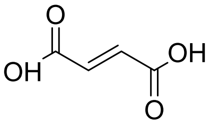
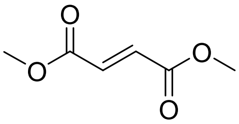
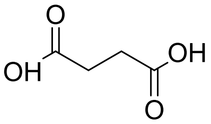
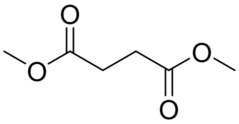


Fig. S7. DMPO spin-trapping EPR spectra in aqueous or methanol solutions in the presence of g-C_3N_4/PAN nanofiber under visible light irradiation (λ>400 nm), (a) aqueous solution; (b) methanol solution. (DMPO: 10 mM, pH 9).

Table S2. Degradation intermediates of RhB in the presence of g-C_3N_4/PAN nanofiber under visible light irradiation (λ>400 nm), examined by UPLC Synapt G2-S HDMS in the positive ion mode (A-F) and the negative ion mode (G and H).

Intermediate Products	Retention Time (min)	Theoretical Mass (m/z)	Molecular Formula	Measured Accurate Mass (m/z)	mDa
A	3.56	443.2335	$C_{28}H_{31}N_2O_3$	443.2332	-0.3
B	2.54	415.2022	$C_{26}H_{27}N_2O_3$	415.2010	-1.2
C	1.51	387.1709	$C_{24}H_{23}N_2O_3$	387.1696	-1.3
D	1.40	387.1709	$C_{24}H_{23}N_2O_3$	387.1700	-0.9
E	0.89	359.1396	$C_{22}H_{19}N_2O_3$	359.1392	-0.4
F	0.68	331.1083	$C_{20}H_{15}N_2O_3$	331.1078	-0.5
G	1.12	121.0290	$C_7H_6O_2$	121.0284	-0.6
H	1.09	165.0188	$C_8H_6O_4$	165.0187	-0.1

Table S3. Identification of the intermediates of RhB under visible light irradiation (λ>400 nm) for 180 min by GC-MS.

Intermediate Products	Retention Time (min)	Structures (defore derivation)	Structures (after derivation)
P_1	6.565		
P_2	17.829		
P_3	14.378		
P_4	12.024		
P_5	11.421