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Supporting Information

**Sustainable and reusable electrospun g-C<sub>3</sub>N<sub>5</sub>/MIL-101(Fe)/poly(acrylonitrile-co-maleic acid) nanofiber for photocatalytic degradation of carbamazepine**

Dongying Zhu,<sup>a,b,‡</sup> Zhujun Huang,<sup>a,b,‡</sup> Haiyan Wang,<sup>b</sup> Qiujuan Lu,<sup>b</sup> Guihua Ruan,<sup>a</sup>

Chenxi Zhao,<sup>b</sup> Fuyou Du<sup>a, b\*</sup>

<sup>a</sup>Guangxi Key Laboratory of Electrochemical and Magnetochemical Functional Materials, College of Chemistry and Bioengineering, Guilin University of Technology, Guilin 541004, China

<sup>b</sup>College of Biological and Environmental Engineering, Changsha University, Changsha 410022, China

<sup>‡</sup>Dongying Zhu and Zhujun Huang contributed equally to this work.

## 20 **Text S1**

### 21 **Characteristics of materials**

22       The microstructure and surface morphology of materials were tested by scanning  
23 electron microscope (SEM, Hitachi SU8000, Japan) and transmission electron  
24 microscope (TEM, Tecnai G2 F30, PEI, USA), respectively. The elemental mappings  
25 and chemical compositions of the composites were performed by an energy-dispersive  
26 X-ray spectrometer (EDX) equipped on SEM. The surface area, pore volume and size  
27 of the photocatalysts were estimated by Micromeritics TriStar II 3020 analyzer  
28 (TriStar II 3020, Micromeritics Instrument Corporation, USA). The crystal phases of  
29 the composites were measured by X-ray diffraction (XRD, Bruker D8 Advance,  
30 Germany) with a Cu  $K_{\alpha}$  radiation. The patterns of X-ray photoelectron spectroscopy  
31 were measured on a K-Alpha spectrometer (XPS, Thermo escalab 250Xi, Thermo  
32 Scientific, USA). The chemical bonds analysis was investigated by the Nicolet  
33 Nexus 470 FT-IR spectrometer (Thermo Electron Corporation, USA). The active  
34 species to degrade carbamazepine were detected through trapping by BQ, t-BuOH,  
35 and EDTA-2Na (see Text S2). The characteristic signals of hydroxyl radicals ( $\bullet\text{OH}$ )  
36 and superoxide anion radicals ( $\bullet\text{O}_2^-$ ) were identified by electron spin resonance (ESR,  
37 Bruker A300-10/12, Germany), respectively. Photocatalytic degradation tests were  
38 carried out at room temperature under visible light irradiation (300W Xe lamp, CEL-  
39 HXF300, Aulight Co. Ltd., China) that equipped with a 380 nm cut-off filter ( $\lambda > 380$   
40 nm).

## 42 **Text S2**

### 43 **Trapping experiments of active species**

44 To probe the active species in degradation process, different scavengers (0.4  
45 mmol) were added in sample solution besides E-spun g-C<sub>3</sub>N<sub>5</sub>/MIL-  
46 101(Fe)/PANCMA (20 mg), deionized water (20 mL) and carbamazepine solution (20  
47 mL, 40 mg/L). Here benzoquinone (BQ, 0.5 mmol/L), tertiary butanol (t-BuOH, 5.0  
48 mmol/L), and ethylene diamine tetraacetic acid disodium salt (EDTA-2Na, 1.0  
49 mmol/L) were chosen as radical scavengers to capture superoxide radicals ( $\bullet\text{O}_2^-$ ),  
50 hydroxyl radicals ( $\bullet\text{OH}$ ), and holes ( $\text{h}^+$ ), respectively.

## 51 **Text S3**

### 52 **LC-UV-MS analysis**

53 UltiMate 3000 UHPLC (Thermo Fisher Scientific, USA), equipped with the  
54 Zorbax Eclipse Plus C18 column (4.6×250 mm, 5  $\mu\text{m}$ ), was used to detect the residual  
55 carbamazepine in sample solution. The possible intermediates of carbamazepine  
56 during the degradation process were identified by LC-MS (Q Exactive Focus MS,  
57 Thermo Scientific, USA).

58 For LC analysis, the mobile phase was composed of water (A, 60%) and  
59 acetonitrile (B, 40%) at a flow rate of 0.2 mL/min. The volume was 5  $\mu\text{L}$ . The  
60 detection wavelength was 285 nm, and the column temperature was 30 °C. The  
61 typical LC chromatograms are shown in Fig. S2, and the obtained calibration curve  
62 was presented in Table S2.

63 For MS analysis, the MS analysis parameters were operated in the range of 50-  
64 500 m/z with a positive ion mode using ESI ion source. The sheath gas, aux gas,

65 spray voltage, capillary temperature, and aux gas heater temperature was 35  
66 arbitrary units, 10 arbitrary units, 3500 V, 350 °C, and 300 °C, respectively. The  
67 obtained total ion chromatograms and MS fragments (m/z) were shown in Figs. S3  
68 and S4, respectively.

## 70 Tables

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72 Table S1 Calibration curve, linearity, linear regression coefficient ( $R^2$ ), and limit of  
73 detection (LOD) for determination of carbamazepine, ciprofloxacin, and tetracycline  
74 by HPLC-UV method

Compound	Detection wavelength	Calibration curve	Linearity (ng/mL)	$R^2$	LOD (ng/mL)	RSD (%)
Carbamazepine	285 nm	$y=18.287x+2292.7$	5-1000	0.9992	2.34	1.89
Ciprofloxacin	275 nm	$y=11.279x+2457.0$	5-1000	0.9999	3.27	2.15
Tetracycline	355 nm	$y=5.986x+1511.6$	5-1000	0.9998	4.09	3.37

75 *Note:* The limit of detection (LOD) was evaluated on the basis of a signal-to-noise ratio of 3.

77 Table S2 Results of N<sub>2</sub> adsorption-desorption characteristics on the E-spun PANCMA  
 78 and g-C<sub>3</sub>N<sub>5</sub>/MIL-101(Fe)/PANCMA NFs

Property	Parameter	PANCM	g-C <sub>3</sub> N <sub>5</sub> /	MIL-
		A	101(Fe)/PANCMA	
Surface Area	Single point surface area at P/Po (m <sup>2</sup> /g)	24.9760	21.6322	
	BET Surface Area (m <sup>2</sup> /g)	25.8848	22.3689	
	t-Plot External Surface Area (m <sup>2</sup> /g)	26.1887	22.2204	
Area	BJH Adsorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter (m <sup>2</sup> /g)	25.036	21.673	
	BJH Desorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter (m <sup>2</sup> /g)	46.0199	45.4090	
Pore Volume	Single point adsorption total pore volume of pores less than 130.9985 nm diameter at P/Po=0.985000000 (cm <sup>3</sup> /g)	0.134838	0.091247	
	t-Plot micropore volume (cm <sup>3</sup> /g)	-0.000190	0.000056	
Volume	BJH Adsorption cumulative volume of pores between 1.7000 nm and 300.0000 nm width (cm <sup>3</sup> /g)	0.138315	0.095571	
	BJH Desorption cumulative volume of pores between 1.7000 nm and 300.0000 nm diameter (cm <sup>3</sup> /g)	0.140227	0.097206	
Pore Size	Adsorption average pore width (4V/A by BET) (nm)	20.83660	16.31683	
	BJH Adsorption average pore width (4V/A) (nm)	22.0990	17.6388	
	BJH Desorption average pore width (nm)	12.1884	8.5672	

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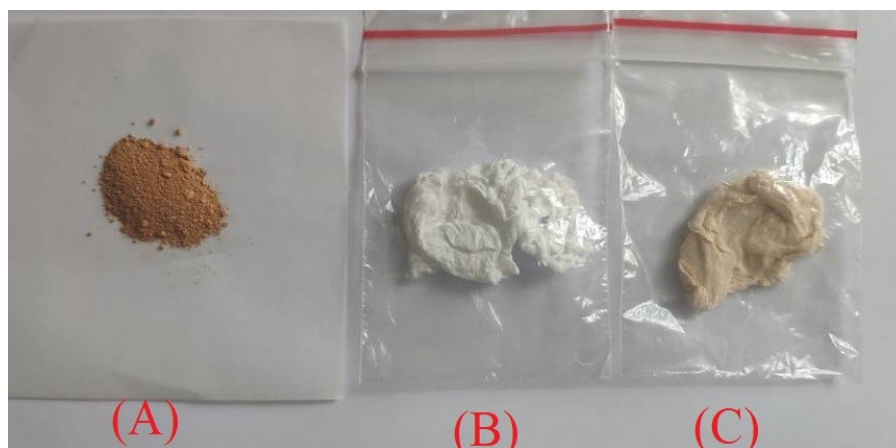
82 TableS 3 Comparison of the developed g-C<sub>3</sub>N<sub>5</sub>/MIL-101(Fe)/PANCMA nanofibers

83 with the other reported photocatalytical materials for removal of carbamazepine

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Materials	Removal mode	Time (min)	Removal efficiency	Cycle times	Referen cev
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> /MIL-101(Cr)	Photocatalytic degradation	180	100%	4	[42]
Bis-PDI-T@TiO <sub>2</sub>	Photocatalytic degradation	30	95%	4	[43]
TiO <sub>2</sub> /carbon dots/polyaniline	Photocatalytic degradation	60	77.63%-83.29%	5	[45]
Co <sub>3</sub> O <sub>4</sub> /CuBi <sub>2</sub> O <sub>4</sub> /SmVO <sub>4</sub>	Photocatalytic degradation	300	76.1%	4	[46]
AgIO <sub>3</sub> /BiVO <sub>4</sub>	Photocatalytic degradation	60	97.86%	1	[47]
E-spun g-C <sub>3</sub> N <sub>5</sub> /MIL-101(Fe)/PANCMA nanofibers	Photocatalytic degradation	40	94.2%	15	This work

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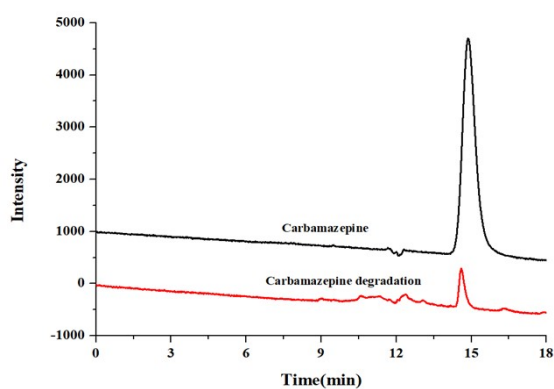
89 **Figure S1** The appearance of g-C<sub>3</sub>N<sub>5</sub>/MIL-101(Fe) composites (A), E-spun90 PANCMA (B), and E-spun g-C<sub>3</sub>N<sub>5</sub>/MIL-101(Fe)/PANCMA (C) nanofibers

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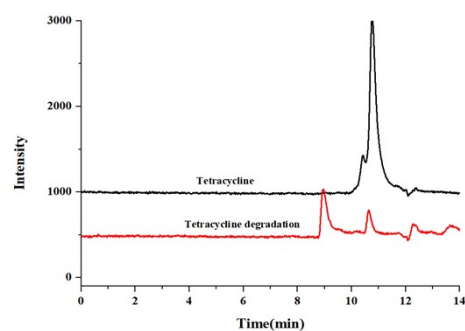
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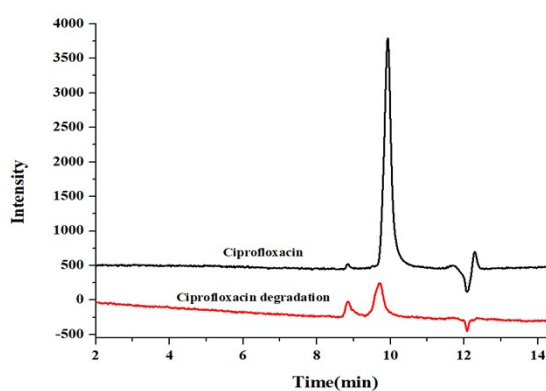




(A)



(B)



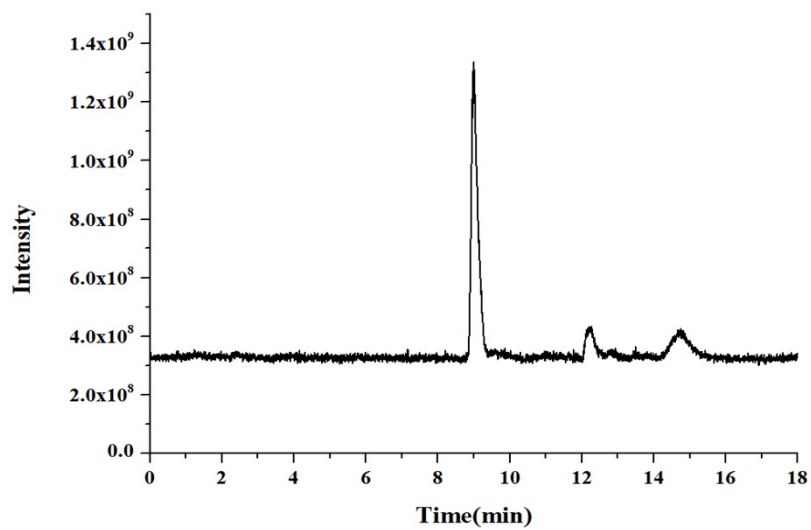
(C)

96 **Figure S2** Typical HPLC-UV chromatograms of carbamazepine (A), tetracycline (B),  
97 and ciprofloxacin (C) before and after photocatalytical degradation with E-spun g-  
98  $C_3N_5/MIL-101(Fe)/PANCMA$  NFs. The original concentration spiked in sample  
99 solution was 200 ng/mL.

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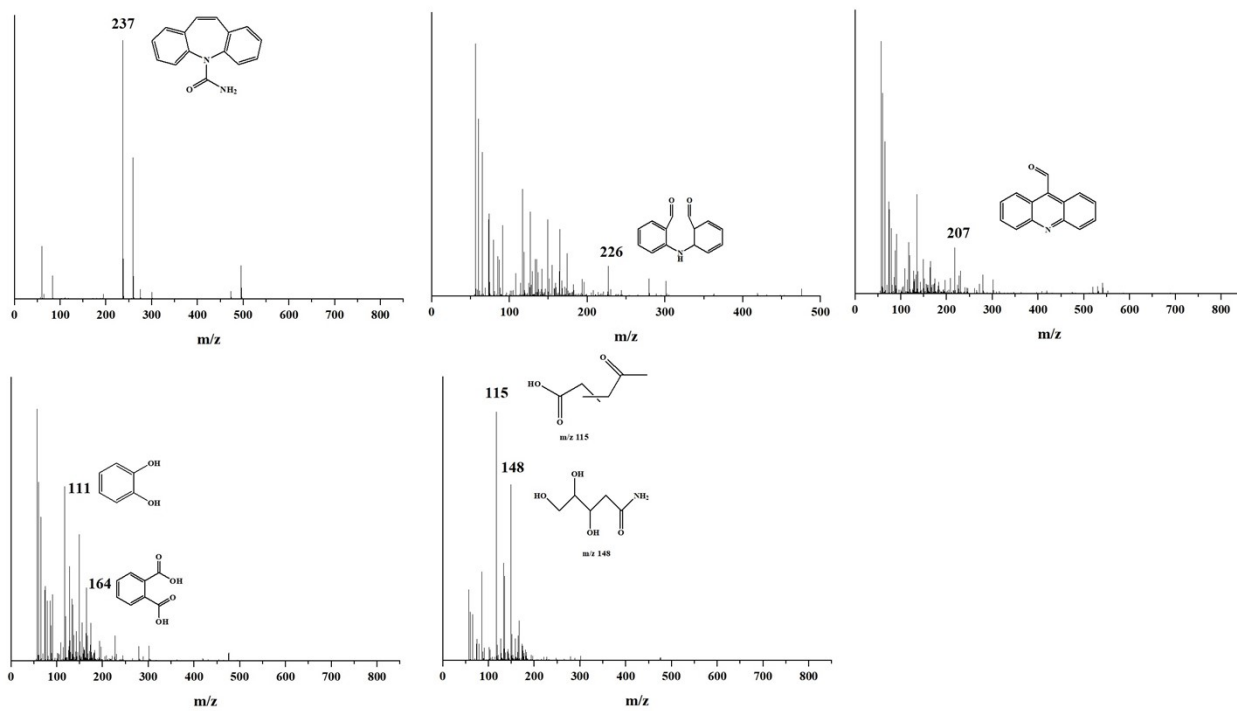
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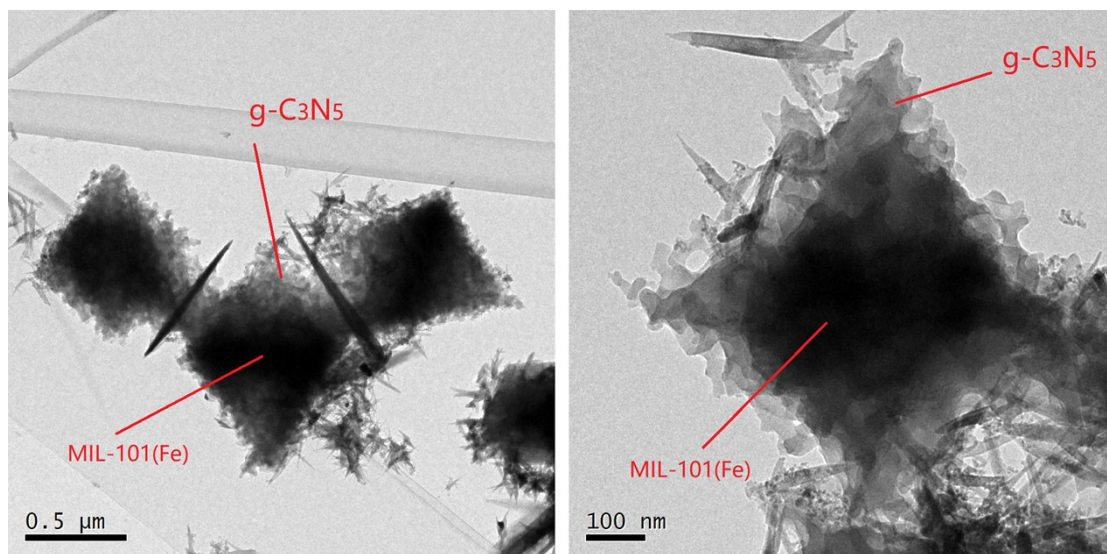
105 **Figure S3** Typical total ion chromatograms of degradation products by photocatalytic  
106 degradation of carbamazepine with E-spun g-C<sub>3</sub>N<sub>5</sub>/MIL-101(Fe)/PANCMA  
107 nanofibers.



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111 **Figure S4** Typical MS of photodegradation products obtained by LC-MS method.

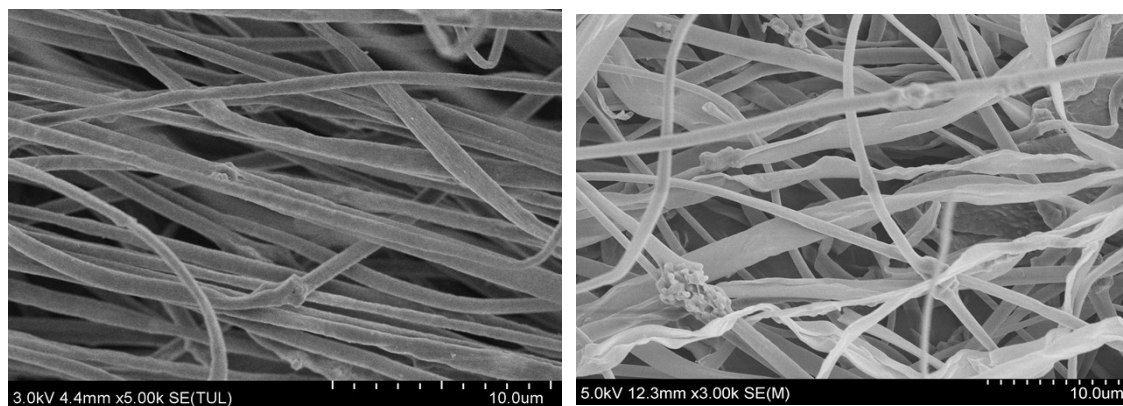
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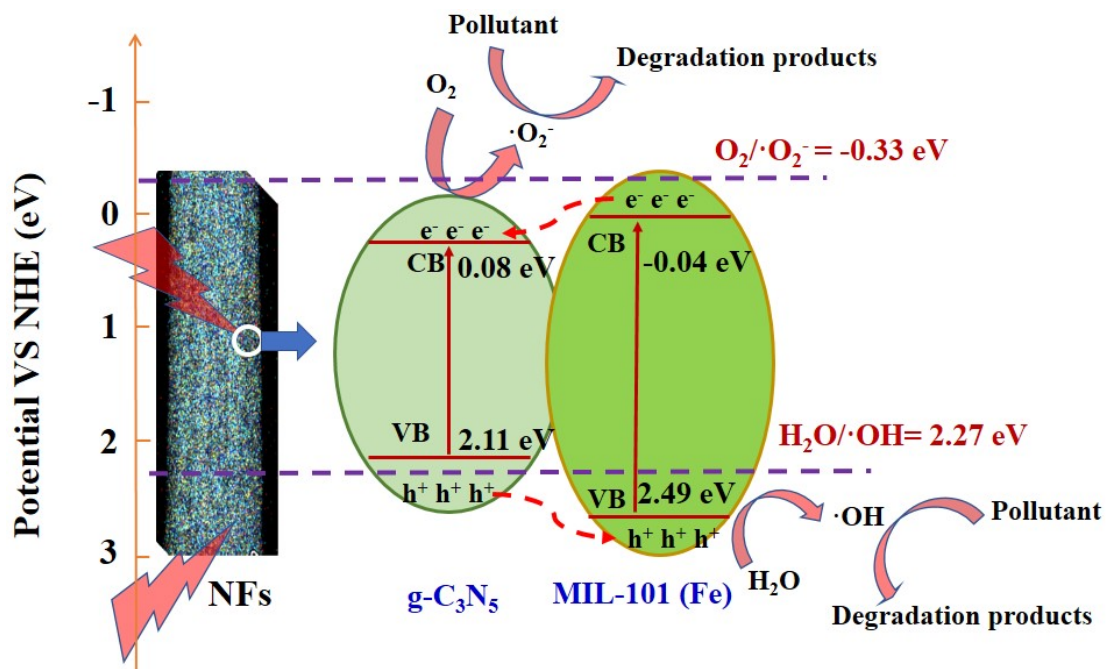
Figure S5 TEM images of g-C<sub>3</sub>N<sub>5</sub>/MIL-101(Fe) composites.



(A)

(B)

118 **Figure S6** SEM images of E-spun  $g\text{-C}_3\text{N}_5/\text{MIL-101}(\text{Fe})/\text{PANCMA}$  NFs before and  
119 after consecutive 20 cycle times of photocatalytic degradation process.



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122 Figure S7 The possible degradation mechanism of  $g-C_3N_5/MIL-101(Fe)/PANCMA$

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towards carbamazepine pollutant.

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