Supplementary Materials

In-situ growing of heterostructure g-C_3N_4/MIL-101(Fe) on iron mesh for activated persulfate to removal degradation of methyl orange: Mechanism and pathway

Zheng Zhou[1], Xuemei Wang[1,2]*, Pengfei Huang[3], Yuan Ma[1], Wei Luo[1], Xinzhen Du[1,2], Xiaoquan Lu[1,2]

[1]*Key Laboratory of Water Security and Water Environment Protection in Plateau Intersection, Ministry of Education, College of Chemistry and Chemical Engineering, Northwest Normal University, Lanzhou 730070, China.*

[2]*Key Laboratory of Bioelectrochemistry and Environmental Analysis of Gansu Province, Lanzhou 730070, China.*

[3]*College of Judicial Police, Gansu University of Political Science and Law, Lanzhou, 730070, China.*

*Corresponding author, Tel: +86-931-7973035

E-mail address: wangxuemei@nwnu.edu.cn (X.M. Wang)

2.1. Chemicals and materials

1,4-benzenedicarboxylic (1, 4-H_2BDC) was purchased from Tianjin Guangfu Fine Chemical Research Institute; N, N-dimethylformamide (DMF; $(CH_3)_2NCHO$) was purchased from Guangdong Guanghua Sci-Tech Co., Ltd.; Potassium Persulfate ($K_2S_2O_8$) was provided by Tianjin Kaitong Chemical Reagent Co. Ltd.; Hydrochloric acid (HCl) was purchased from Shanghai Wokai Biotechnology Co., Ltd.; Ethylenediamine tetra-acetic acid disodium salt (EDTA-2Na), methyl alcohol (CH_3OH), Methyl Orange (MO), *p*-benzoquinone (BQ), *t*-butanol (TBA), and methanol (MeOH) were purchased from Sinopharm Chemical Reagent Co. Ltd; Iron mesh (30 mm × 30 mm network, diameter of single iron wire = 0.14 mm), ultrapure water was used throughout the experiments.

2.2. Characterization

Fluorescence stereo microscopes (FSM, Leica M205 FA) and scanning electron microscopy (SEM, JSM-6701E) were used to determine the morphologies of materials. The structural was characterized by X-ray diffraction (XRD, Rigaku D/max-2400/PC), X-ray photoelectron spectroscopy (XPS, Esclab-250), and Fourier-transform infrared spectroscopy (FTIR, Digilab FTS3000). Optical properties were determined by TU-1901 ultraviolet-visible spectrophotometer and photoluminescence spectrometer (PL, Fluoro Sens 9003). UV–vis curves were obtained by UV-1750 spectrometer. Brunauer–Emmett–Teller (BET) specific surface area and pore size distribution of the samples was performed on a surface analyzer (BELSORP-max II). In order to measure transient photocurrent and electrochemical impedance (EIS), CHI-660 electrochemical workstation (Shanghai Chenhua Instrument, China) was employed. In the three-electrode detection system, Pt was used as counter electrode; Ag/AgCl (saturated KCl) was the reference electrode; 0.5 mol/L Na_2SO_4 was used as electrolyte; 300 W model CEL-HXF300 xenon lamp was the light source. The

ESR (electron spin resonance) spectrum were acquired using a Bruker a300 spectrometer. MO degradation products were analyzed by the high resolutions mass spectrum (HRMS, Q Exactive).

Figure S1. a) UV-vis absorption spectra of different concertation MO. b) The standard curve of MO.

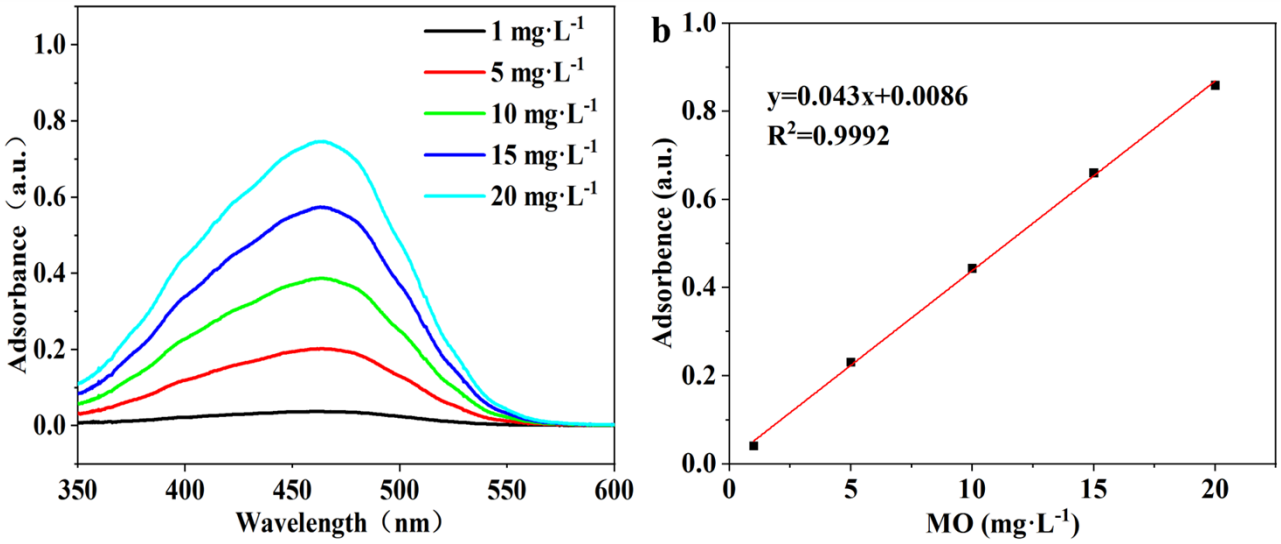


Figure S2.PL spectra of g-C_3N_4, MIL-101(Fe), and g-C_3N_4/MIL-101(Fe)

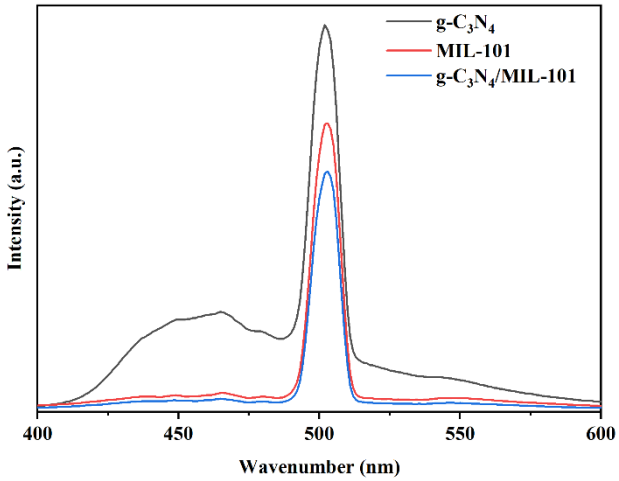


Figure S3. N_2 adsorption/desorption isotherms and pore size distribution plot of g-C_3N_4 (a), MIL-101(Fe) (b).

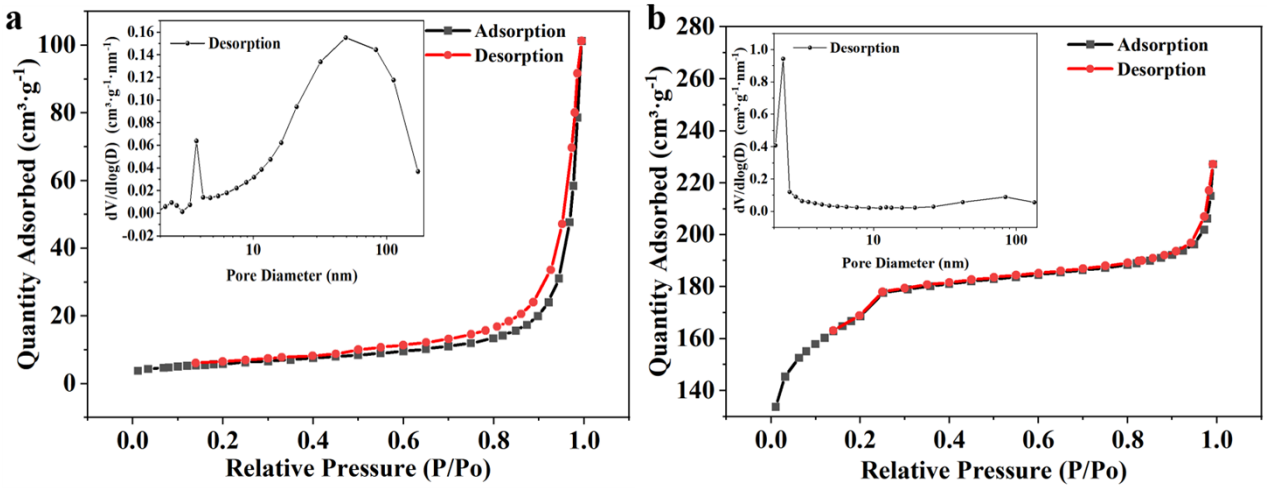


Figure S4. The influence of visible light on the degradation of MO.

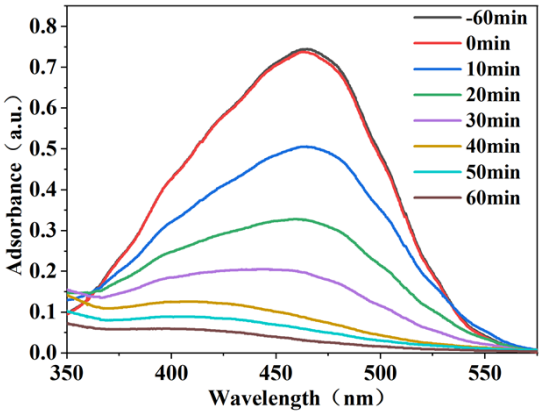


Figure S5. MS of MO solution at different reaction time

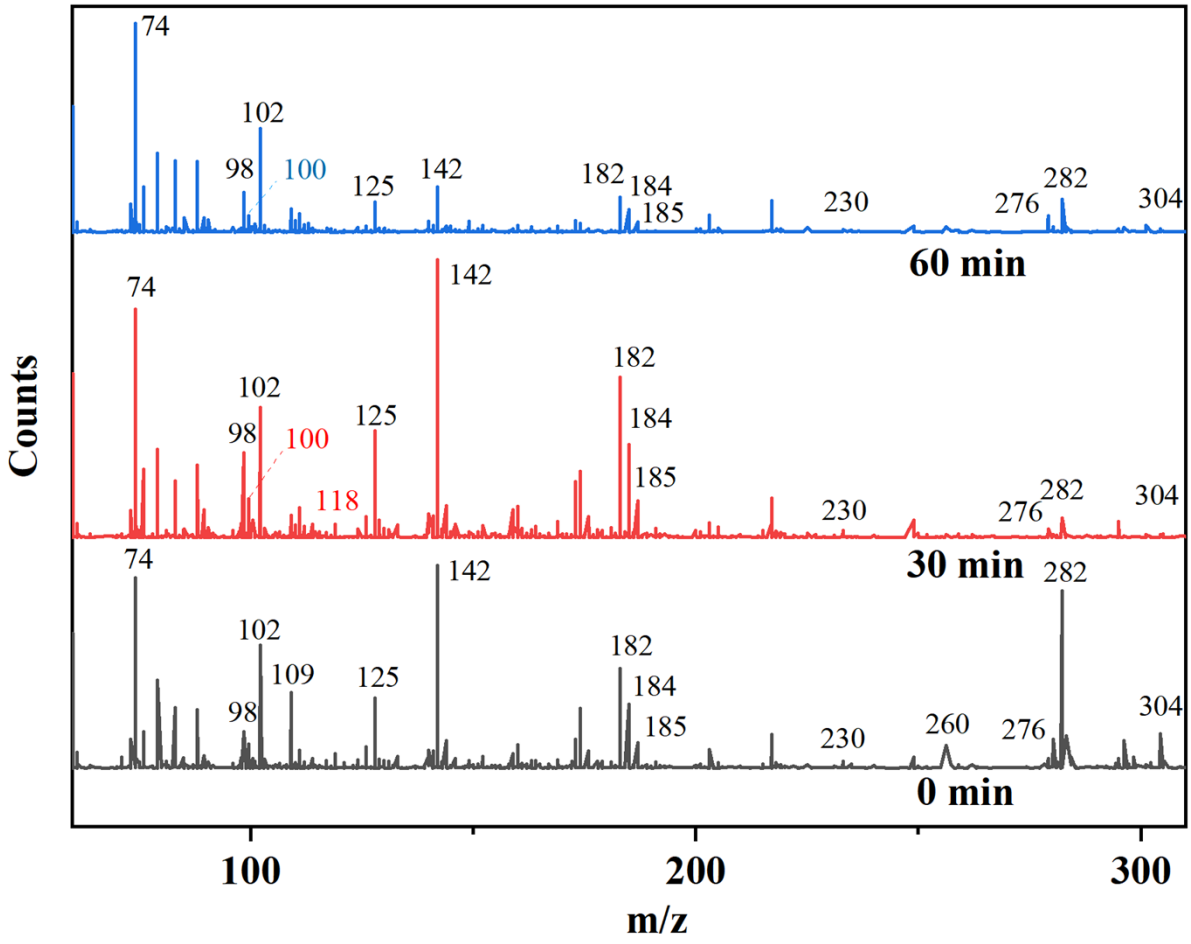
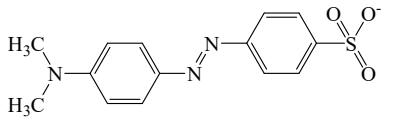
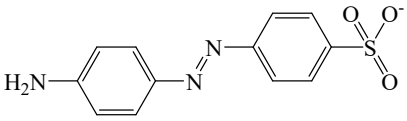
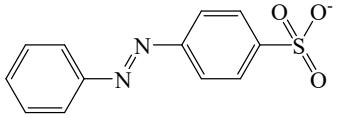
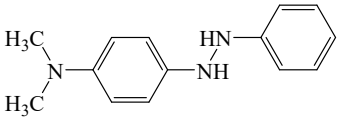
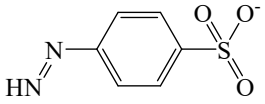
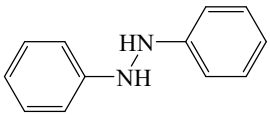
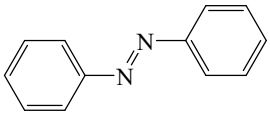
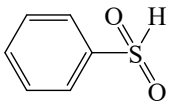
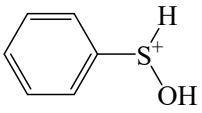
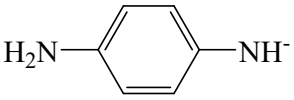
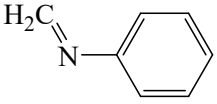
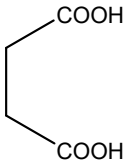


Table S1 The BET specific surface (S_{BET}), pore volume (V_p), and pore size (D) of MIL-101(Fe) and g-C_3N_4.

Samples	S_{BET} (m^2 /g)	Vp (cm^3 /g)	D (nm)[a]
g-C_3N_4	21.03	0.157	24.8/28.9
MIL-101(Fe)	600.57	0.170	3.8/4.1

[a] The pore diameters were calculated from the adsorption/desorption branch of the isotherm using the BJH method.

Table S2. HRMS analysis results of MO photocatalytic degradation products

Abbreviation	m/z	Possible structure
MO^-	304	
A	276	
B	260	
C	230	
D	185	
E	184	
F	182	
G	142	
H	125	
I	109	
J	102	
K	118	
L	100