MoS_2/MoO_3 nano-heterojunction towards enhanced photocatalytic activity under LED light irradiation via in-situ oxidation auxiliary

Jianfeng Qiu [a,b], Yanping Liu[b] , Minxian Cao[b],Luqi Xie [b], Yongkun Liu [b] ,Hongwen li[b], Junqiang Lu[b,] Qifeng Liang [b] , jiaqi Pan [a, c],Chaorong Li *[a, c]

a. College of Textile Science and Engineering, Zhejiang Sci-Tech University, Hangzhou, 310018, PR China. E-mail address: crli@zstu.edu.cn (C. Li).

b. Department of Physics, Shaoxing University, Shaoxing 312000, PR China

c. Key Laboratory of Optical Field Manipulation of Zhejiang Province, Department of Physics, Zhejiang Sci-Tech University, Hangzhou 310018, P. R. China

Photoelectrochemical measurements

Photoelectrochemical test were performed on an electrochemical workstation (CHI 660, CH Instruments Inc., Shanghai) in a conventional three-electrode cell system. 10 mg as prepared sample and 10 μl 5% nifion was added into 30 uL ethyl alcohol to form a uniform suspension after 30 min ultrasonic treatment. The suspension was coated on a piece of FTO glass (1×2 cm^2) with 0.28 cm^2 restricted by a sticky tape, then dried at room temperature for 24 h. The FTO glass, Pt electrode and saturated calomel electrode (SCE) were used as working, counter and reference electrodes, respectively. The working electrode was irradiated by a high-pressure xenon short arc lamp (XHA 350 W Xe lamp). The incident light intensity was 75.5 mW cm^{-2}, which was measured with a radiometer (Photoelectric Instrument Factory Beijing Normal University, model FZ-A). The electrochemical impedance spectroscopy (EIS) was carried out in the frequency range of 10^{-2} to 10^5 Hz with anac voltage amplitude of 10 mV at a AC bias of 0.3 V(vs the SCE, in 0.5 M Na_2SO_4 electrolyte).

Photocatalytic activity test

The photocatalytic performance was evaluated using a PCX-50C Discover multichannel photocatalytic reaction system equipped with 10 W LED lamps. In detailed, 10 mg photocatalyst was dispersed in 50 mL Rhodamine B aqueous solution (30 mg/L). The mixed solution was magnetically stirred in dark for 30 min to reach adsorption-desorption equilibrium. Then at 30 min irradiation intervals, 4 mL reaction solution was taken out and centrifuged. The concentration of rhodamine B was analyzed by a Shimadzu UV-3600 plus spectrophotometer at 550 nm. The value of C/C_0 was calculated according to the calibration curve of concentration and absorption, illumination intensity is 37.2 mW· cm^{-2} .

Characterization

The crystallographic phases were analyzed using an X-ray powder diffractometer (XRD, Empyrean). The surface morphology of the samples was examined via field emission scanning electron microscopy (FESEM, SIGMA 300) and high-resolution transmission electron microscopy (HRTEM, JEM-2100 F). The chemical states of the elements within the samples were determined through X-ray photoelectron spectroscopy (XPS, Thermo ESCALAB 250). Ultraviolet-visible diffuse reflectance spectra (UV–vis DRS) were acquired using a UV-3600 plus spectrophotometer (Shimadzu). Photoluminescence (PL) spectra were recorded on a fluorescence spectrophotometer (Hitachi F-7000), with an excitation wavelength of 350 nm. Electrochemical impedance spectroscopy (EIS) measurements were conducted using a CHI 660 electrochemical workstation in a standard three-electrode configuration.

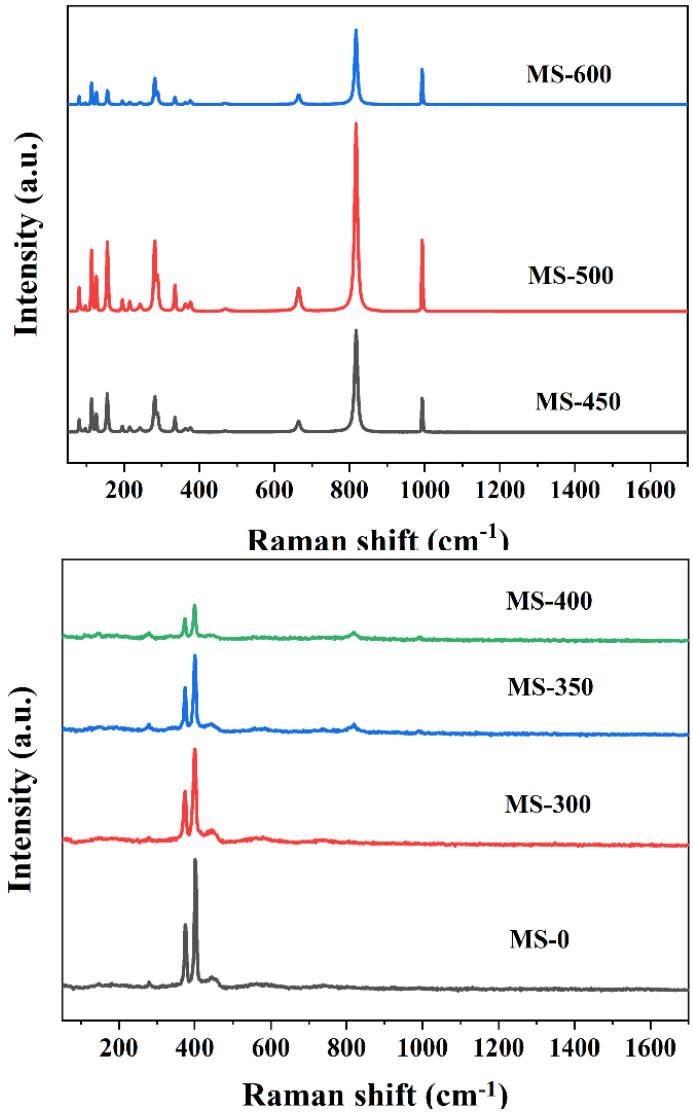

Fig. S1 Raman spectra of MS-0, MS-300, MS-350, MS-400, MS-450 ,MS-500 ,MS-600 samples

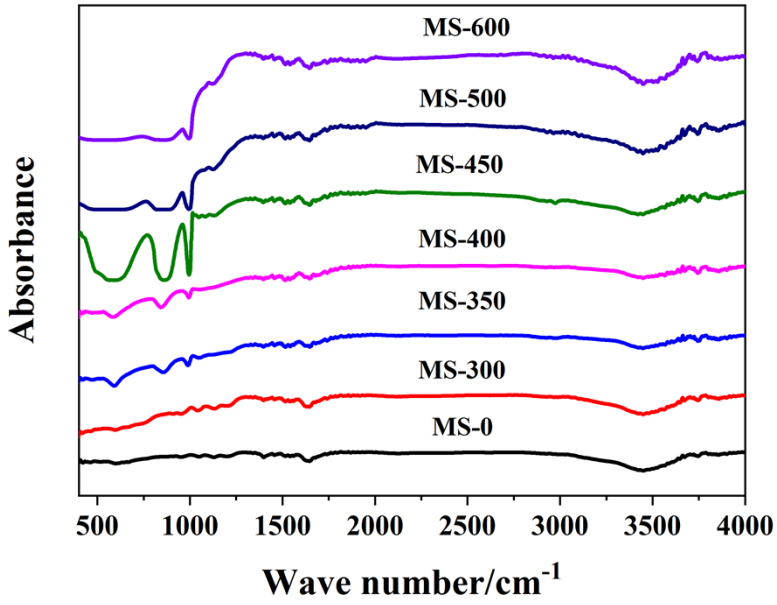

Fig.S2 The FTIR spectroscopy of samples at different sintering temperatures

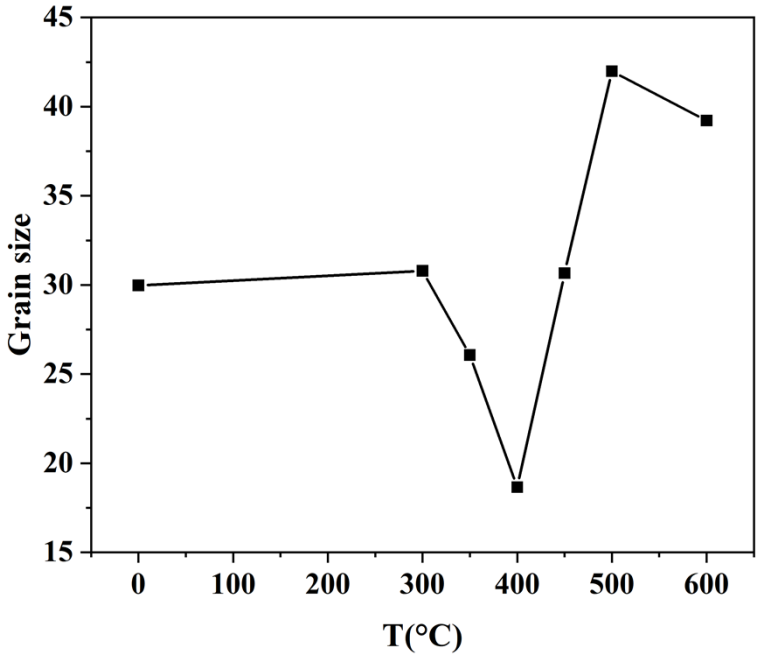

Fig.S3 The grain size of samples at different sintering temperatures

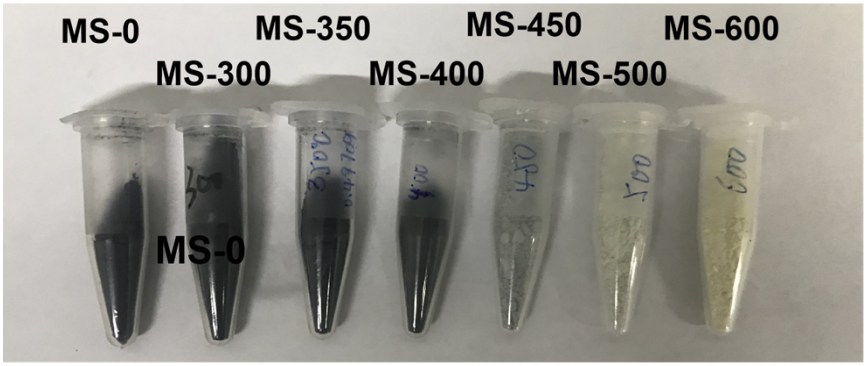

Fig.S4 The digital photos of samples at different sintering temperatures

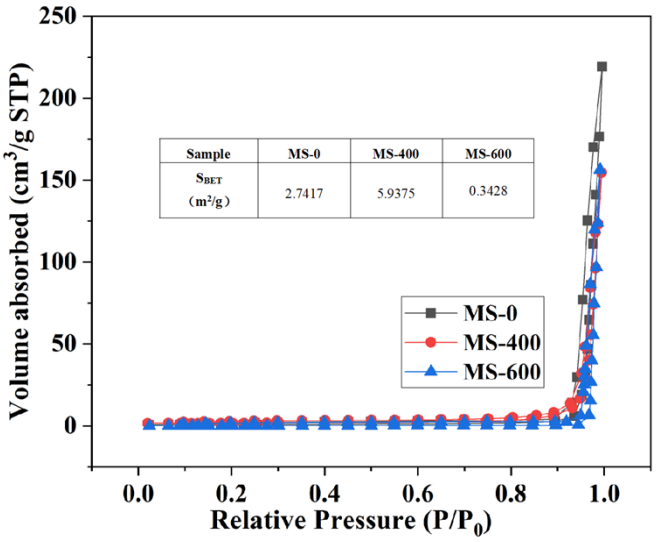

Fig.S5 The Nitrogen adsorption-desorption isotherms and The Specific Surface Area of samples

T/℃	0	300	350	400	450	500	600
before sintered /g	0.5						
after sintered /g	0.5	0.5	0.4543	0.4394	0.4113	0.4065	0.4017

Table S1 The sample quality changes at different sintering temperatures

Photocatalyst	**Methods**	**Photodegradation performance (x-flods (catalyst))**	**Reference**
MoS_2 / $CaTiO_3$	Hydrothermal	~5.17(CaTiO3)	1
MoS_2/BN	Hydrothermal	~1.5(MoS_2)	2
MoS_2/g-C_3N_4	impregnation and calcination method	~ 7.7(g-C3N4)	3
MoS_2/$ZnIn_2S_4$	hydrothermal	~5 ($ZnIn_2S_4$)	4
BiOBr/Bi2WO6	hydrothermal	~8.5(Bi2WO6)	5

N-doped MoS_2 nanoflowers	hydrothermal	~ 26.4(MoS2 nanosheets) ~ 7(P25)	6
MoS_2/NiFe LDH	electrostatic self-assembled and hydrothermal	~3 (NiFe LDH) ~10.9 (MoS2)	7
DR-MoS_2/PMS/US	hydrothermal	~13.3 (MoS2/US) ~3 (MoS2/US/PMS)	8
C_3N_4/rGO/MoS_2	hydrothermal	~ 5 C3N4/rGO/MoS2	9
MoS_2/MoO_3	sintering	~8.7-folds MoS_2 ~31.5-folds MoO_3	**This work**

Table S2 the comparative table at different work

Reference

1. M. H. Luo, J. X. Xu, W. J. Xu, Y. Zheng, G. Wu and T. Jeong, *Nanomaterials*, 2023, **13**, 14.
2. L. J. Chen, B. S. Navya and C. D. Dong, *J. Phys. Chem. Solids*, 2024, **193**, 11.
3. H. Gao, Y. Liu, L. J. Wang, J. C. Zhu, S. W. Gao and X. F. Xia, *Res. Chem. Intermed.*, 2019, **45**, 3687-3703.
4. Y. Chen, L. Z. Zhu, J. Z. Li, L. F. Qiu, C. H. Zhou, X. Xu, Y. L. Shen, J. B. Xi, D. Men, P. Li and S. Duo, *Inorg. Chem.*, 2023, **62**, 7111-7122.
5. F. Qin, Y. Luo, Q. Yu, J. Cheng, Q. Qin, X. Zhu and W. Feng, *Journal of Molecular Structure*, 2024, **1304**.
6. P. T. Liu, Y. G. Liu, W. C. Ye, J. Ma and D. Q. Gao, *Nanotechnology*, 2016, **27**, 8.
7. S. Nayak, G. Swain and K. Parida, *ACS Appl. Mater. Interfaces*, 2019, **11**, 20923-20942.
8. S. H. Li, X. E. Ning, P. Y. Hao, Y. L. Cao, J. Xie, J. D. Hu, Z. J. Lu and A. Z. Hao, *Dyes Pigment.*, 2022, **206**, 11.
9. B. Wu, C. W. Wang, Z. Y. Wang, K. Shen, K. Y. Wang and G. Li, *Langmuir*, 2024, **40**, 1931-1940.