

## **Solar Light Motivated Photoelectrocatalytic and Photocatalytic Applications Based on Flower-like NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> Heterojunction**

Bicheng Hu<sup>a</sup>, **Yuhui Zhang**<sup>a</sup>, Jincheng Zhang<sup>a</sup>, Jiazan Liu<sup>a</sup>, Meng Lei<sup>a</sup>, Chenxi Zhao<sup>a</sup>,

QiuJun Lu<sup>a</sup>, Haiyan Wang<sup>a\*</sup>, Fuyou Du<sup>a\*</sup> and Shiyong Zhang<sup>b\*</sup>

<sup>a</sup> *College of Biological and Chemical Engineering, Changsha University, Changsha,  
410022, Hunan Province, China*

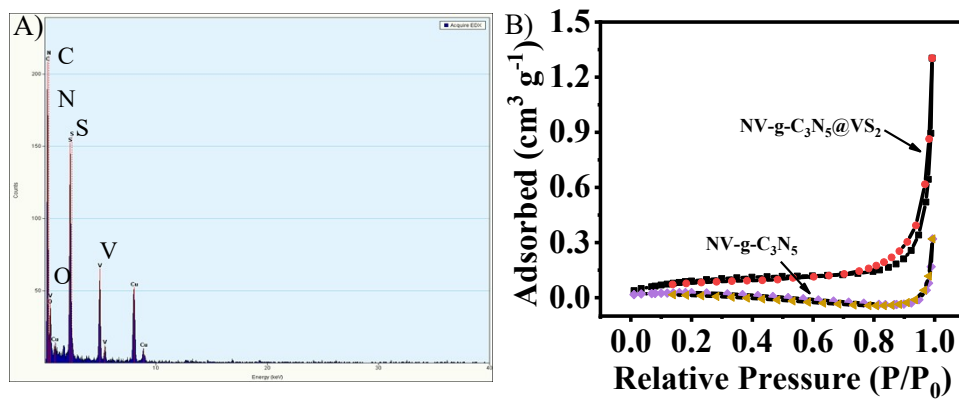
<sup>b</sup> *Hunan Key Laboratory of Applied Environmental Photocatalysis, Changsha  
University, Changsha, 410022, Hunan Province, China*

<sup>c</sup> *Hunan Engineering Research Center of Degradable Materials and Molding  
Technology, Changsha University, Changsha, 410022, Hunan Province, China*

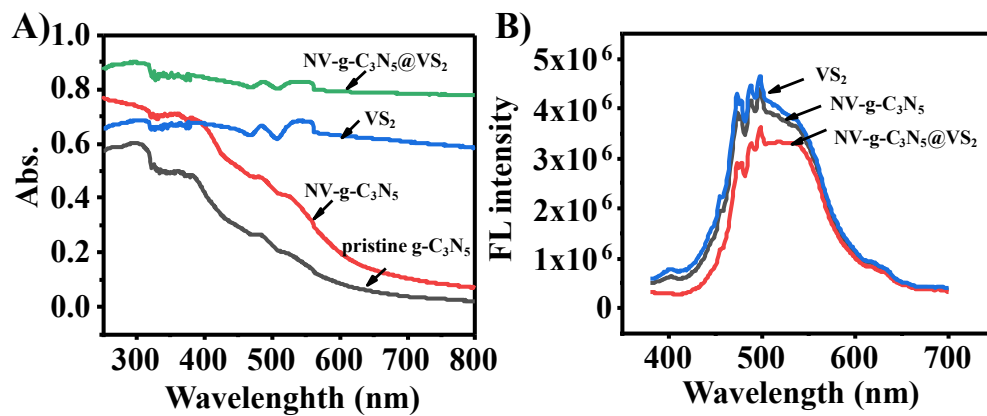
\* Corresponding author; Tel: +86-84261506; Fax: +86-731-84261382;

E-mail address: wanghaiyan@ccsu.edu.cn (Haiyan Wang), dufu2005@126.com (Fuyou Du),

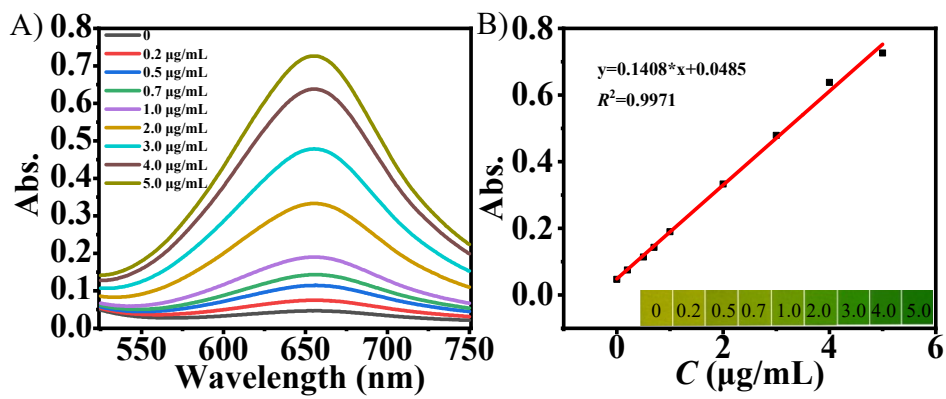
cdzhangshiyong@163.com (Shiyong Zhang)



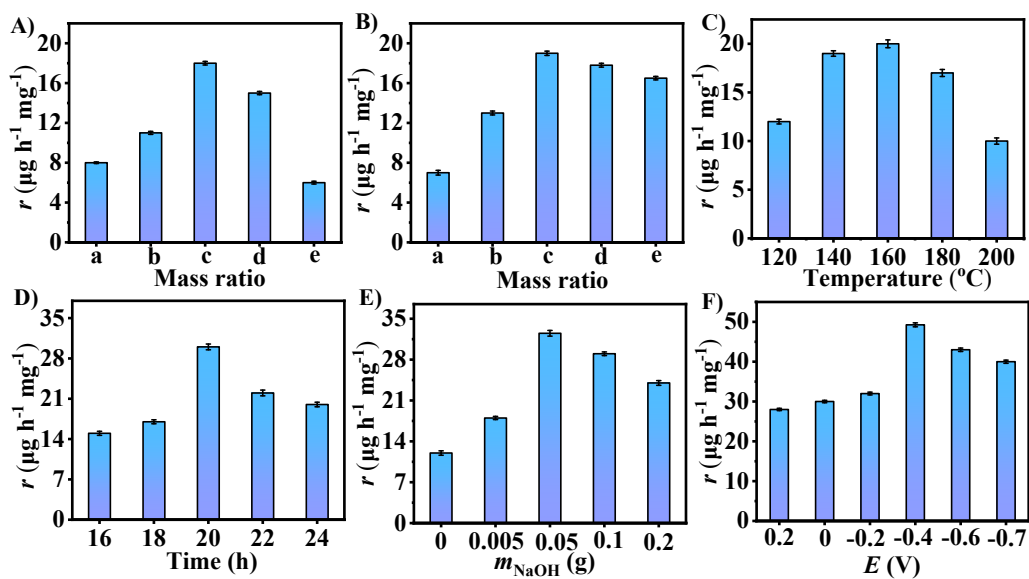
**Fig. S1** (A) EDX image of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction; (B) N<sub>2</sub> adsorption and desorption isotherm curves of NV-g-C<sub>3</sub>N<sub>5</sub> and NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction.



**Fig. S2** (A) UV-vis DRS spectra of VS<sub>2</sub>, NV-g-C<sub>3</sub>N<sub>5</sub>, g-C<sub>3</sub>N<sub>5</sub> and NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction. (B) Solid-state fluorescence spectra of VS<sub>2</sub>, NV-g-C<sub>3</sub>N<sub>5</sub>, NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub>.



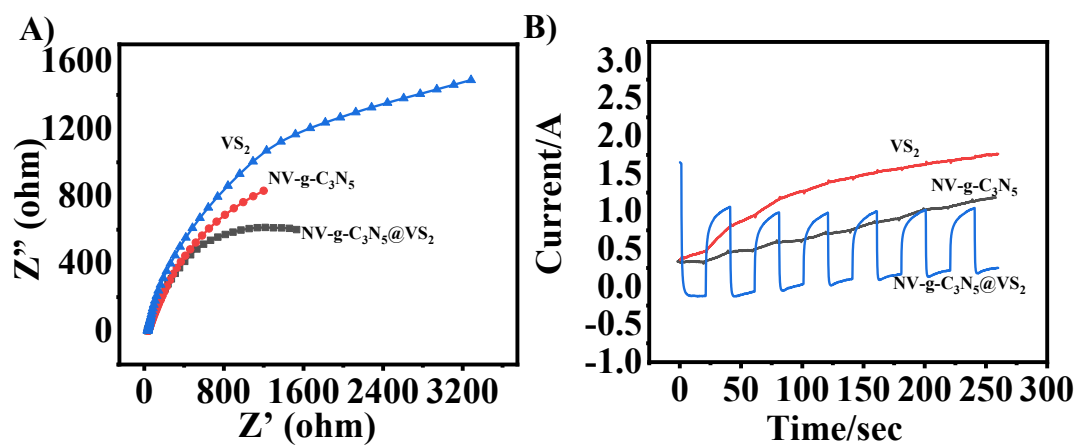
**Fig. S3** (A) UV-Vis absorption spectra of  $\text{NH}_4^+$  treated with indoxyl blue at room temperature for 2 h. (B) The linear relationship between the standard concentration of  $\text{NH}_4^+$  and the absorbance at 650 nm.



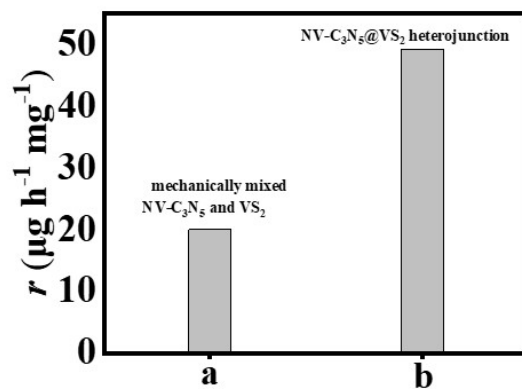
**Fig. S4** (A) Ammonia yields of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunctions synthesized by different mass ratio of NV-g-C<sub>3</sub>N<sub>5</sub>, ammonium metavanadate and thioacetamide. a, b, c, d and e represent 0.4:0.2:0.8, 0.2:0.2:0.8, 0.1:0.2:0.8, 0.05:0.2:0.8 and 0.04:0.2:0.8, respectively. (B) Ammonia yields of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunctions synthesized by different mass ratio of NV-g-C<sub>3</sub>N<sub>5</sub>, ammonium metavanadate and thioacetamide. a, b, c, d and e represent 0.1:0.2:0.2, 0.1:0.2:0.4, 0.1:0.2:0.6, 0.1:0.2:0.8 and 0.1:0.2:1.0, respectively. (C) Ammonia yields of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunctions synthesized by different hydrothermal temperature. (D) Ammonia yields of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunctions synthesized by different hydrothermal time. (E) Ammonia yields of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunctions synthesized by NV-g-C<sub>3</sub>N<sub>5</sub> treated with different NaOH (0, 0.005, 0.05, 0.1, 0.2, 0.3 g). (F) Ammonia production rates of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunctions catalyzed at different applied voltages for 1 h.

**Tab. S1** Comparison of photoelectric catalytic nitrogen reduction with different materials.

Material	electrolyte solution	Ammonia production rate ( $\mu\text{g h}^{-1} \text{mg}^{-1}$ )	References
NV-g-C <sub>3</sub> N <sub>5</sub> /BiOBr	0.05 M HCl+0.05 M Na <sub>2</sub> SO <sub>4</sub>	29.4	1
N-NiO/CC	0.1 M LiClO <sub>4</sub> at -0.5 V	22.7	2
Au NPs-PTFE	0.05 M H <sub>2</sub> SO <sub>4</sub> +0.05 M Na <sub>2</sub> SO <sub>3</sub>	18.9	3
g-C <sub>3</sub> N <sub>4</sub> /ZnMoCdS	0.1 M KCl	2.5	4
Au/TiO <sub>2</sub>	0.1 M HCl at -0.40 V	34.1	5
NV-g-C <sub>3</sub> N <sub>5</sub> /VS <sub>2</sub>	0.1 M Na <sub>2</sub> SO <sub>4</sub>	49.26	This work

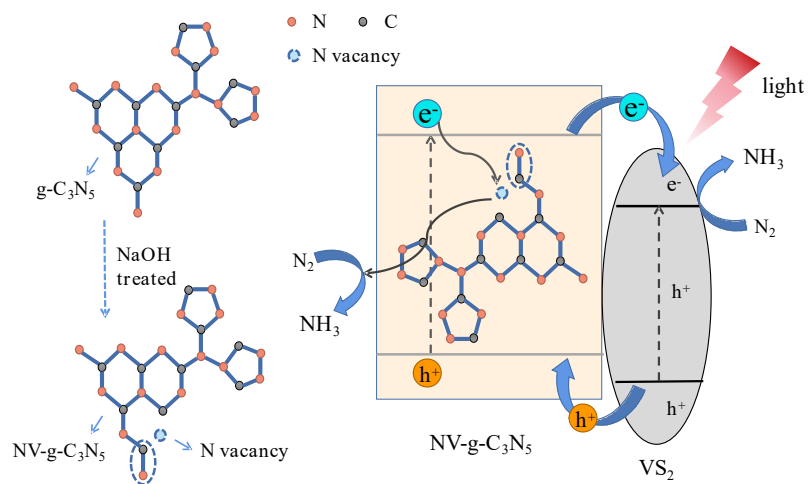


**Fig. S5** (A) The electrochemical impedance spectroscopy (EIS) results and (B) photocurrent responses of  $NV-C_3N_5$ ,  $VS_2$  and  $NV-g-C_3N_5@VS_2$  heterojunction under visible light irradiation, respectively.

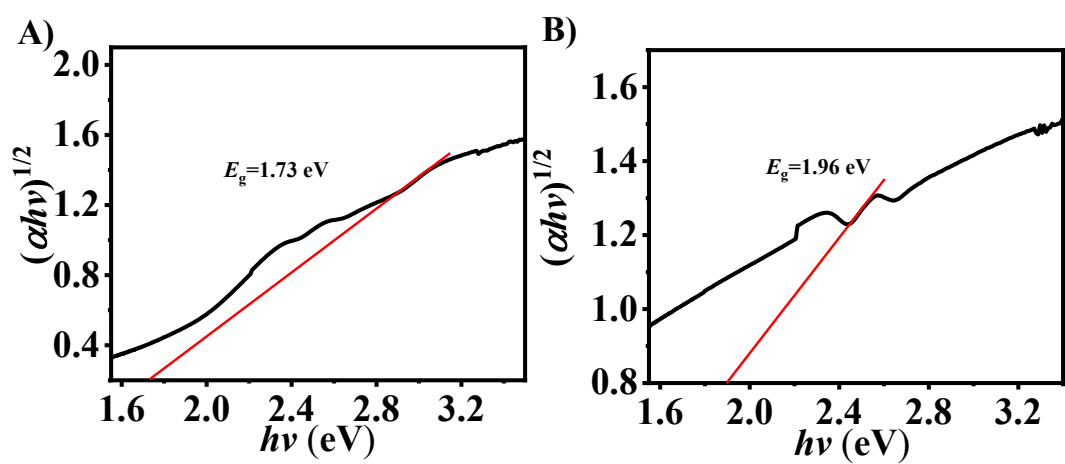


**Fig. S6** The productive rate of ammonia of the mechanically mixed NV-C<sub>3</sub>N<sub>5</sub> and VS<sub>2</sub> (a), and NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction (b) in 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution filled N<sub>2</sub> with the bias voltage of -0.4 V under visible light.

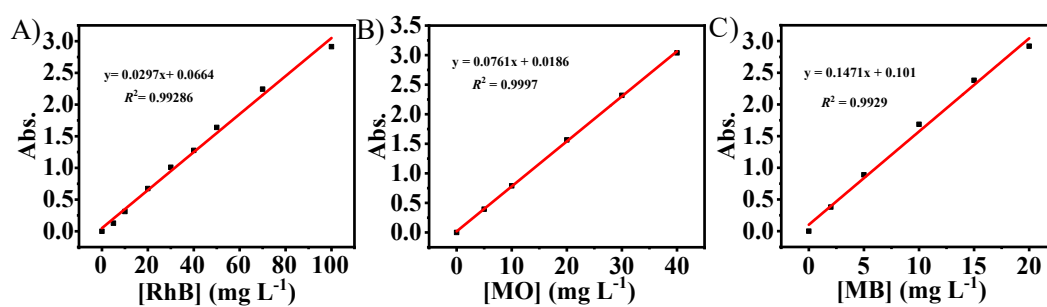




**Fig. S7** Schematic illustration of the preparation of NV-g-C<sub>3</sub>N<sub>5</sub> and the photoelectric catalytic nitrogen reduction reaction of NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction.

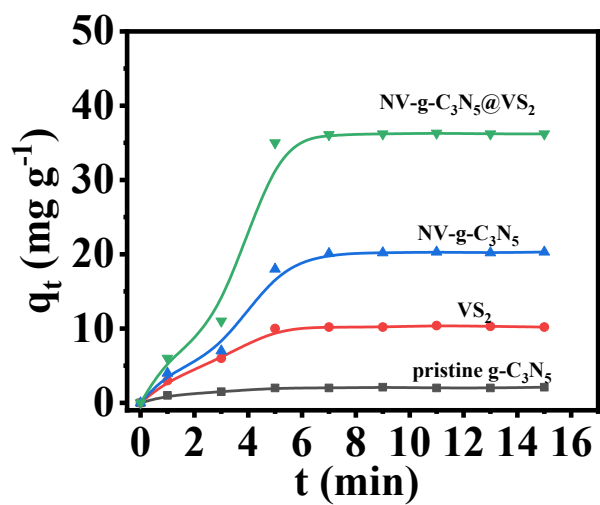


**Fig. S8** Plots of transformed Kubelka–Munk function versus photon energy of (A) NV-g-C<sub>3</sub>N<sub>5</sub> and (B) VS<sub>2</sub>.

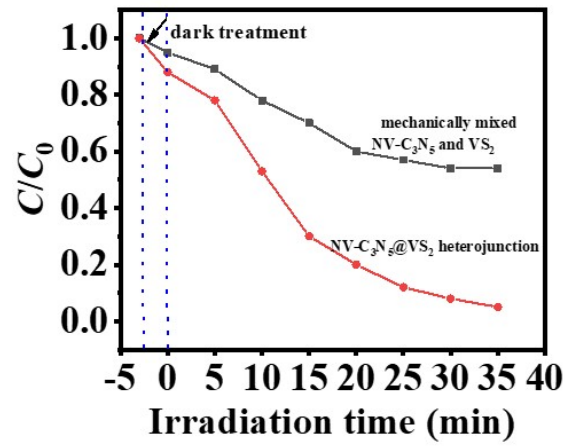


**Fig. S9** The linear relationships between the absorbance and the standard concentration of RhB

MB and MO, respectively.

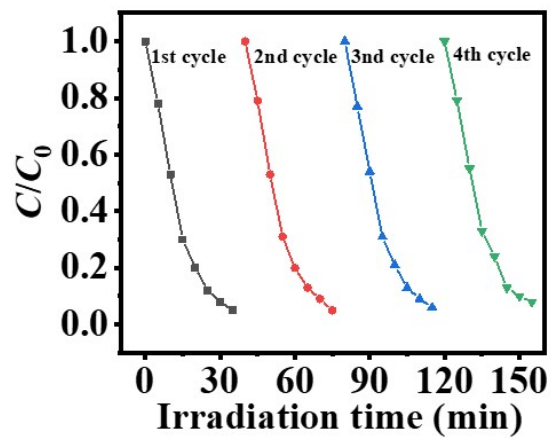


**Fig. S10** The maximum adsorption capacity of pristine g-C<sub>3</sub>N<sub>5</sub>, NV-g-C<sub>3</sub>N<sub>5</sub>, VS<sub>2</sub> and NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction.



**Fig. S11** The changes ratio Rhodamine concentration with irradiation time over the mechanically mixed NV-C<sub>3</sub>N<sub>5</sub> and VS<sub>2</sub> (a), and NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction under visible light irradiation.

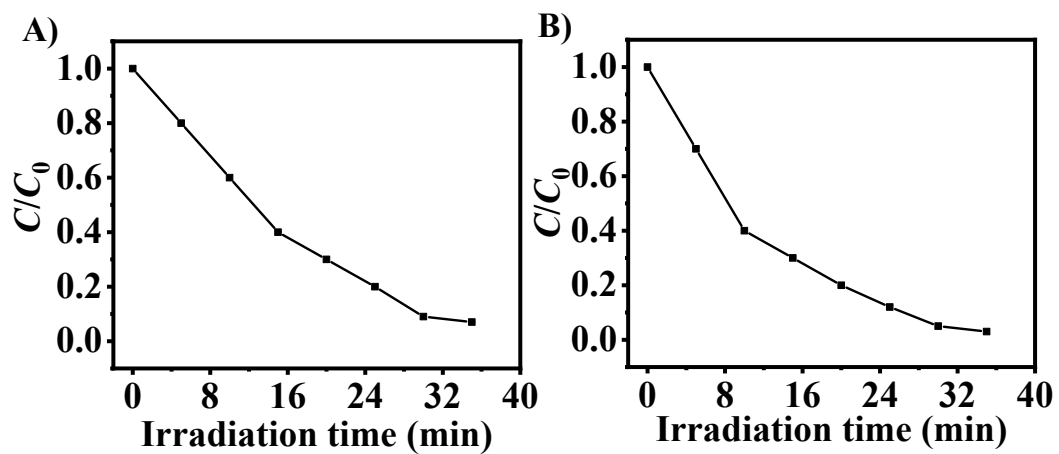
$C_0=40 \text{ mg L}^{-1}$ .



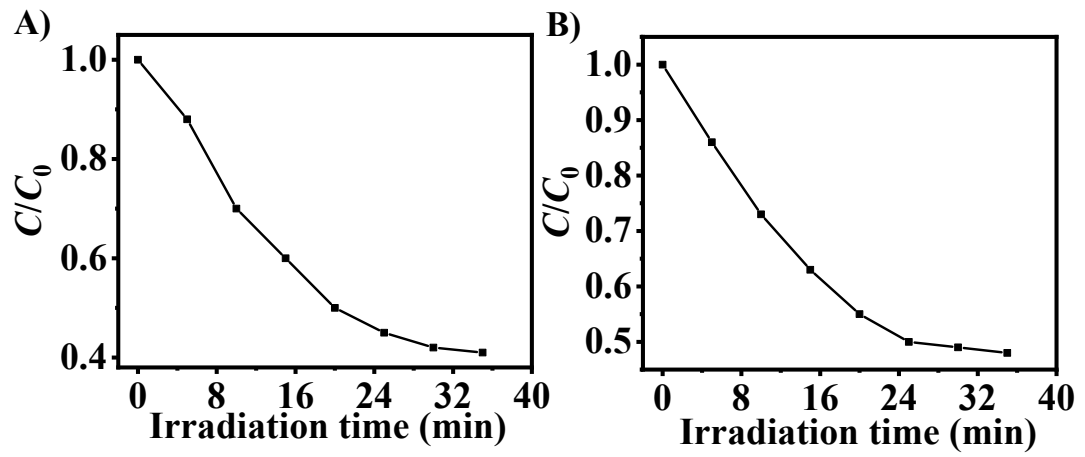
**Fig. S12** Recyclability of the visible photocatalytic decomposition of MB by NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub>

heterojunction.  $C_0$  is the initial concentration of RB.  $C$  is the remaining concentration of RB.

$C_0=40 \text{ mg L}^{-1}$ .



**Fig. S13** The change ratio (A) MO and (B) MB concentration with irradiation time over NV-g- $C_3N_5@VS_2$  heterojunction under visible light irradiation.  $C_0=40 \text{ mg L}^{-1}$ .  $C_0$  and  $C$  are the original and the remaining concentration of pollution (MO and MB), respectively.



**Fig. S14** The changes ratio RB concentration with (A) p-benzoquinone and (B) dimethyl sulfoxide over NV-g-C<sub>3</sub>N<sub>5</sub>@VS<sub>2</sub> heterojunction under visible light irradiation.



## Reference

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