Electronic Supplementary Material (ESI) for Catalysis Science & Technology.

Supporting Information

Orientation of 1T Phase Mediated 1T/2H-MoS_2 Nanoflowers Embellished BCN Nanosheets Z-Scheme Heterojunction towards Antibiotic Degradation and H_2 Evolution

Sarmistha Das, Lopamudra Acharya, Lijarani Biswal, Bhagyashree Priyadarshini Mishra and Kulamani Parida*

Centre for Nano Science and Nano Technology, Institute of Technical Education and Research, Siksha 'O' Anusandhan University, Bhubaneswar-751030

**Corresponding author*

***Corresponding Author's Email:**
kulamaniparida@soa.ac.in
paridakulamani@yahoo.com

Tel. No.: +91-674-2351777 and Fax. +91-674-2350642

Characterization Techniques:

To analyze the phase purity and crystalline nature of the prepared material Powder X-ray Diffraction (XRD) were conducted with a Rigaku Miniflex X-ray diffractometer having a monochromator outfitted with Cu K$\propto$ radiation ($\lambda = 0.154\,nm,$ 30 KV/50 mA). X-ray photoelectron spectroscopy (XPS) analyses were carried out with a VG Microtech Multilab ESCA-3000 spectrometer using Mg-Kα as an X-ray sources, to identify the chemical state of the produced samples. The morphological analysis such as FESEM was conducted using FEIN Quanta-400 FEG-SEM, whereas the internal structure of the as–synthesized samples were analyzed by JEOL-JEM-2100 transmission electron microscope (HRTEM). The bandgap and absorbance results of the obtained sample i.e. UV-Vis DRS spectra were determined by JASCO-V-750 UV-Vis spectrometer taking $BaSO_4$ as reference with a range of 200-800 nm. The excitation and emission spectra of photoluminescence (PL) were studied by using a JASCO FP-8300 fluorescence spectrometer having an excitation wavelength 325 nm along with a Xenon (Xe) lamp as light source.

Photoelectrochemical Measurements:

Considering three electrode configuration including reference (Ag/AgCl), counter (Pt foil) and working electrode (fluorine doped Tin Oxide (FTO)) respectively, a multi-channel-Ivium potentiostat-galvanostat (IVIUM-n-STAT) electrochemical workstation was used to perform all photo electrochemical measurements. A visible light source of 300 W Xenon lamp with 400 nm cut-off was supplied to the whole workstation. 0.1 M Na_2SO_4 aqueous solution was utilized as electrolyte. Furthermore, working electrode was papered with 10 mg of the sample, 1% Nafion (20 μL) and 0.7 ml of ethanol and the resulting slurry was then dropped-cast onto the conducting surface of the FTO substrate. The FTO was kept for oven dry at 90

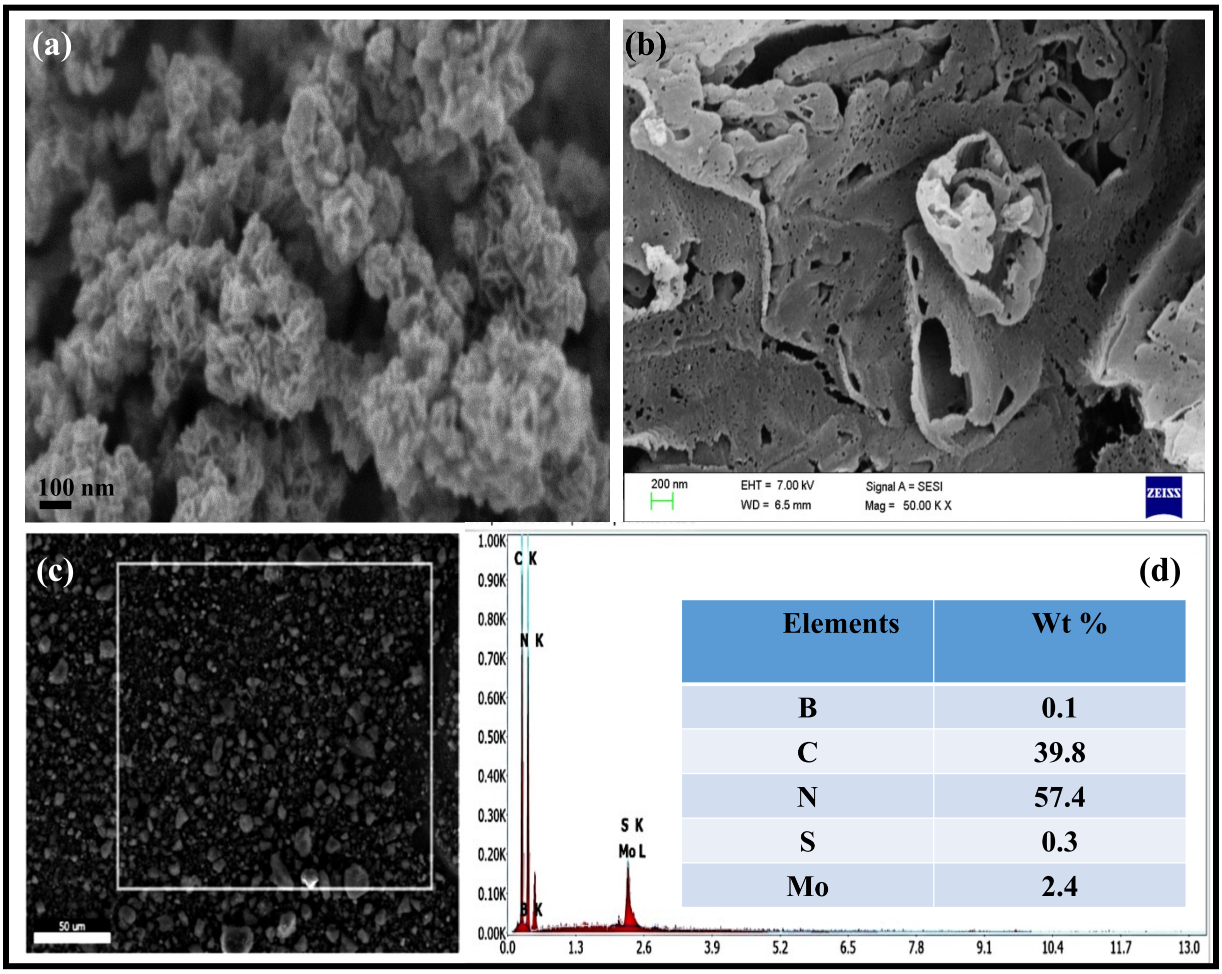
°C for 12 hours to make the working electrode. Light source can be performed by using 300 W Xenon lamps equipped with a 400 nm cut-off filter.

Photocatalytic TCH Degradation:

The photocatalytic behavior of 1T/2H-MoS_2, BCN, and 1T/2H-MoS_2@BCN-*x* (i.e. MBCN-5, MBCN-10 and MBCN-15) composites were investigated by means of the degradation of 10 ppm TCH (by dissolving required amount of TCH in deionized water) under illumination of solar light for 1 h. For this purpose, prepared photocatalysts of 0.02 g each (1T/2H-MoS_2, BCN and MBCN-*x*) were ultrasonically dispersed in 20 mL of TCH solution taken in 5 different conical flasks (100 mL). Then the samples were stirred for 30 min under dark condition in order to achieve the adsorption-desorption equilibrium, followed by irradiation for 1 h under solar light. Thereafter, the samples were centrifuged at 6000 rpm for 7 min to extract the photocatalyst material from the solution. Subsequently, the diminished concentration of TCH in samples was analyzed with the help of an UV-Vis spectrometer (JASCO V-750). To figure out the intermediates of TCH degradation, Liquid Chromatography (LC) technique using the TSQ Quantum Access MAX Triple Quadrupole Mass Chromatography (MS) of Thermo-Fisher Scientific analysis of the supernatant is also carried out.

Photocatalytic H_2 Evolution:

The photocatalytic water splitting reaction was implemented in a Pyrex quartz glass photo reactor connected to a 150 W Xenon lamp of wavelength higher than 420 nm. The experiment was carried out by taking 20 mg of catalysts (1T/2H-MoS_2, BCN, and 1T/2H-MoS_2@BCN-*x* (i.e. MBCN-5, MBCN-10 and MBCN-15)) dispersed in to 20 mL of 10 % Methanol solution (sacrificial agent). Then the suspension was stirred to prevent the catalysts from settling down at the bottom of the reactor. Before the light illumination, the atmosphere of reactor as maintained inert by purging N_2 gas for several times. After 1 h of light irradiation, the evolved hydrogen gas was collected via water displacement technique. Then the amount of H_2 evolution was measured by GC-7890B (Agilent technology) supplied with thermal conductivity detector (TCD) and a 5 Å molecular sieve packed column.



Elements	Wt %
B	0.1
C	39.8
N	57.4
S	0.3
Mo	2.4

Figure S1. FESEM images of (a) 1T/2H-MoS_2 (b) BCN (c) SEM image for EDX spectra of MBCN-10 and (d) EDX spectra of MBCN-10 composite.

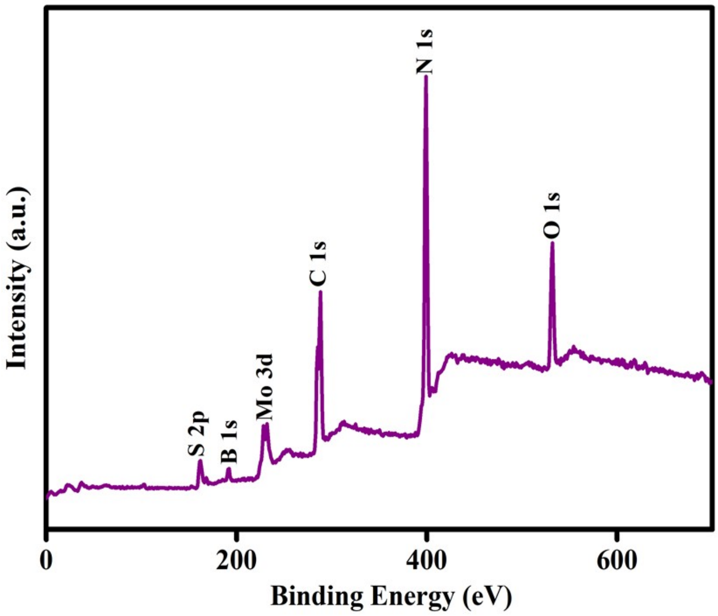


Figure S2. XPS survey scan of the MBCN-10 composite.

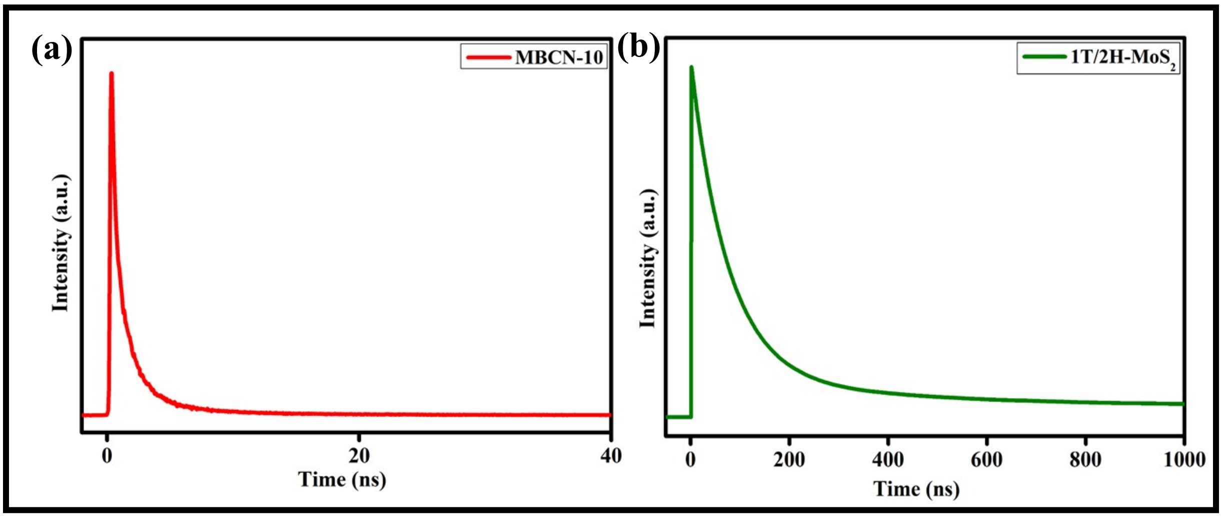


Figure S3. TRPL spectra of (a) MBCN-10 (b) 1T/2H-MoS$_2$.

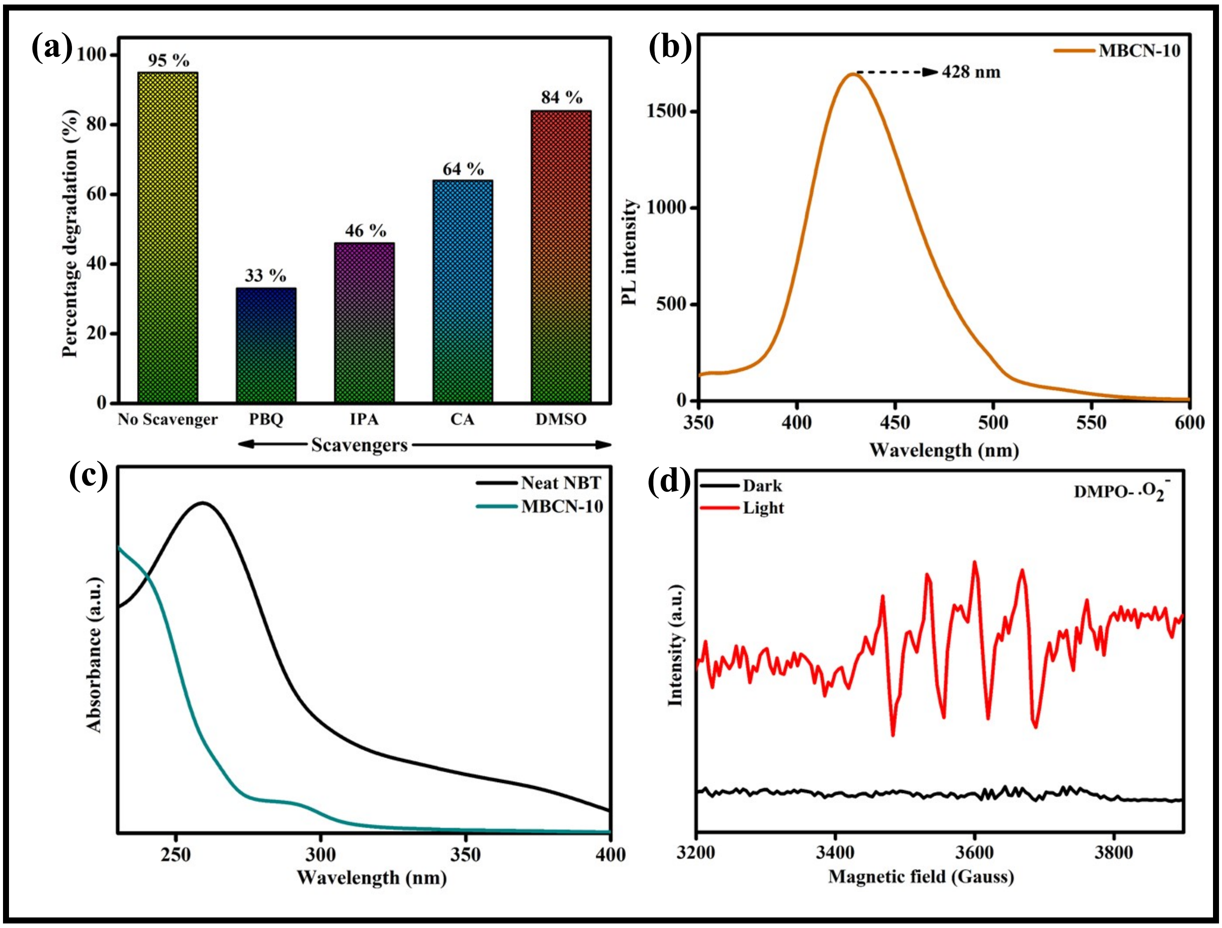


Figure S4. (a) Scavenger (b) TA test for •OH (c) NBT test for $•O_2^-$ and (d) EPR spectra of DMPO- $•O_2^-$

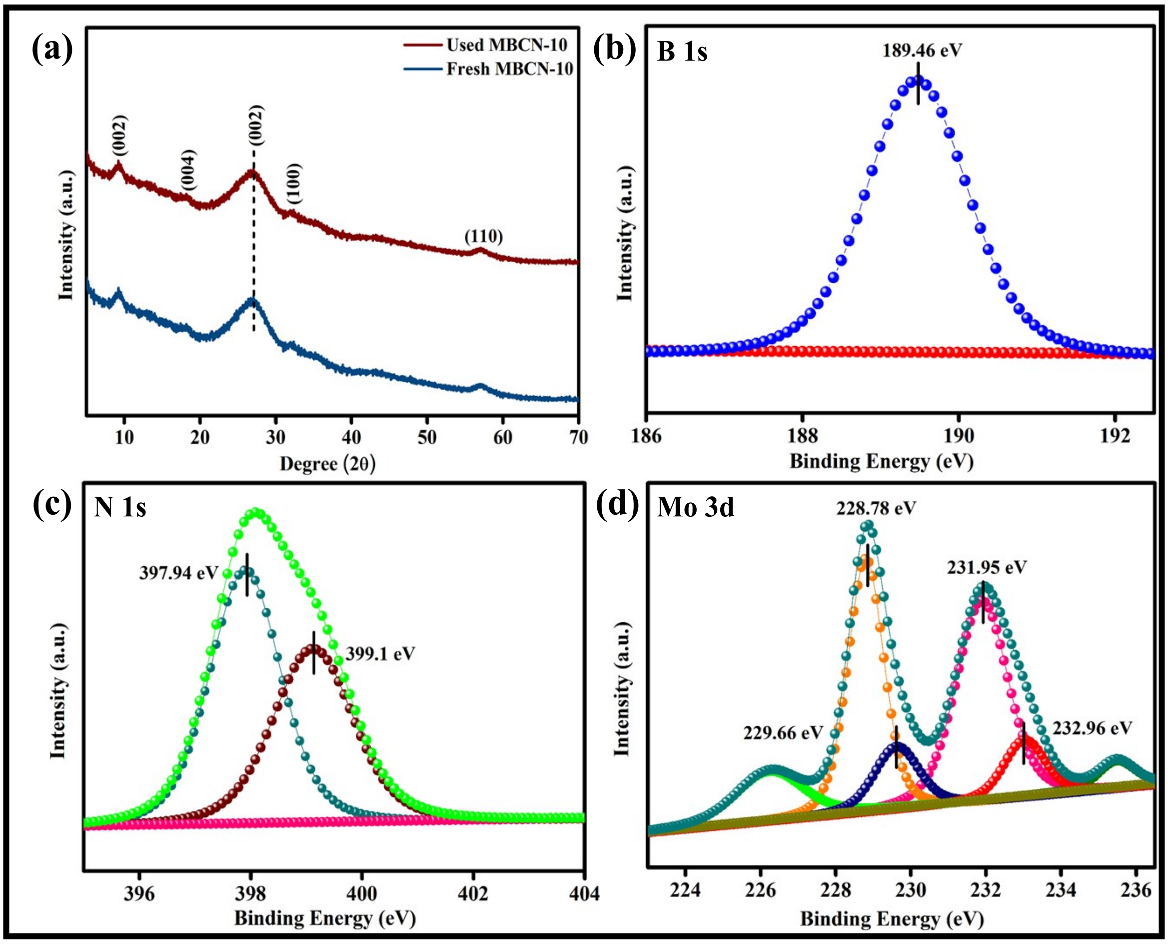


Figure S5: (a) XRD pattern for fresh and used MBCN-10 photocatalyst. XPS of (b) B 1s (c) N 1s and (d) Mo 3d of MBCN-10 composite after photocatalytic activity.

Table S1. Pseudo-first order kinetic data obtained for TCH degradation by BCN, 1T/2H-MoS_2 and MBCN-*x* composites.

Sl. No.	**Photocatalysts**	**k (min^{-1})**	**R^2**	**$t_{1/2}$**
1	TCH	0	0	0
2	BCN	9×10^{-3}	0.97363	77.2
3	1T/2H-MoS_2	14×10^{-3}	0.97228	49.64
4	MBCN-5	34×10^{-3}	0.98709	20.44
5	MBCN-10	50×10^{-3}	0.98921	13.90
6	MBCN-15	27×10^{-3}	0.98441	25.74

Table S2. Comparison table representing photocatalytic H_2 evolution of various photocatalysts

Sl. No.	Material	Amount of catalyst (mg)	Sacrificial agent	Light source	H_2 production rate	Reference
1	1T/2H-MC	50	0.5 M Na_2SO_3/Na_2S	300 W Xe lamp	73.1 µmol/h	1
2	1T-MoS_2/CNB	10	Triethanolamine (TEOA)	300 W Xe lamp	5334 µmol $h^{-1}g^{-1}$	2
3	$MoS_2@TiO_2$	-	0.35 M Na_2S and 0.25 M Na_2SO_3	300 W Xe lamp	8.43 µmol cm^{-2} h^{-1}	3
4	1T-MoS_2/O-g-C_3N_4	10	Triethanolamine (TEOA)	300 W Xe lamp	1841.72 µmol/g/h	4
5	e-BN/e-CN	20	Methanol	125 W Xe lamp	7359 µmol/g/h	5
6	30-1T/2H-MoS_2	20	0.35 M Na_2S and 0.25 M Na_2SO_3	150 W Xe lamp	142.7 µmol/h	6
7	MBCN-10	20	Methanol	150 W Xe lamp	290 µmol/h	This work

Table S3. Comparison table representing photocatalytic TCH degradation of various photocatalysts

Sl. No.	Material	Source	Pollutant	Illumination time (min)	Degradation %	References
1	VTM-5%	1000 W Xe lamp	OTTCH	80	92.4	7
2	$ZrO_2@MoS_2$/g-C_3N_4	300 W Xe lamp	TCH	90	94.8	8
3	MS/BEuCN	400 W	TCH	50	99	9

		halogen lamp				
4	MoS_2/B-rGO	300 W Xe lamp	TCH	90	85.2	10
5	g-C_3N_4/MoS_2	300 W Xe lamp	TCH	40	98.6	11
6	BN/BCN	Solar light	TCH	60	88.1	12
7	MBCN-10	Solar light	TCH	60	95	This work

References:

1. C. Zhu, Q. Xian, Q. He, C. Chen, W. Zou, C. Sun, S. Wang and X. Duan, *ACS Appl. Mater. Interfaces*, 2021, **13**, 35818-35827.
2. L. Ding, F. Qi, Y. Li, J. Lin, Y. Su, Y. Song, L. Wang, H. Sun and C. Tong, *J. colloid interface sci.*, 2022, **614**, 92-101.
3. Y. Liu, Y. Li, F. Peng, Y. Lin, S. Yang, S. Zhang, H. Wang, Y. Cao and H. Yu, *Appl. Catal. B: Environ.,* 2019, **241***,* 236-245.
4. H. Xu, J. Yi, X. She, Q. Liu, L. Song, S. Chen, Y. Yang, Y. Song, R. Vajtai, J. Lou and H. Li, *Appl. Catal. B: Environ.,* 2018, **220***,* 379-385.
5. L. Acharya, S. P. Pattnaik, A. Behera, R. Acharya and K. Parida, *Inorg. Chem.*, 2021, **60**, 5021-5033.
6. S. Das, G. Swain, B. P. Mishra and K. Parida, *New J. Chem.*, 2022, **46**, 14922-14932.
7. C. Zhu, Q. Xian, H. Yao, C. Chen, W. Zou, X. Duan, Z. Jiang and P. K. Wong, *Mater. Today Energy*, 2022, **23**, 100901.
8. E. Vijayakumar, M. G. Raj, M. G. Narendran, R. Preetha, R. Mohankumar, B. Neppolian, A. J. Bosco, *ACS omega*, 2022, **7**, 5079-5095.
9. P. Guo, F. Zhao and X. Hu, *J. Alloys Compd.*, 2021, **867**, 159044.
10. E. Vijayakumar, B. Neppolian, S. K. Lakhera and A. J. Bosco, *Mater. Lett.,* 2021, **296***,* 129891.

11. Y. Cao, S. Alsharif and A. S. El-Shafay, *Mater. Sci. Semicond. Process.,* 2022, **144***,* 106569.
12. L. Acharya, S. Nayak, S. P. Pattnaik, R. Acharya and K. Parida, *J. colloid interface sci.*, 2020, **566**, 211-223.