

Supporting Information

Tuning of Surface Oxygen Vacancies for enhancing Photocatalytic Performance under Visible Light Irradiation in Sb_2WO_6 Nanostructures

Manisha Sharma, Rahul Singh, Anitya Sharma and Venkata Krishnan*

School of Chemical Sciences and Advanced Materials Research Center,

Indian Institute of Technology Mandi, Mandi 175075, Himachal Pradesh, India.

Email: vkn@iitmandi.ac.in

Sl. No.	Content	Pg. No.
1	Material characterization	S-2
2	Calculated crystallite size, lattice strain and lattice parameters	S-3
3	EDAX spectra and elemental mapping of SWO	S-4
4	High resolution XPS survey spectra of SWO and DSWO2	S-5
5	Tauc plots for SWO, DSWO1, DSWO2, DSWO3	S-6
6	BET plots for SWO, DSWO1, DSWO2, DSWO3	S-7
7	UV-vis absorption spectra of CRV	S-8
8	UV-vis absorption spectra of RhB	S-9
9	UV-vis absorption spectra of LFX	S-10
10	UV-vis absorption spectra for mixture of dyes	S-11
11	Zeta potential values for SWO, DSWO1, DSWO2, DSWO3	S-12
12	Langmuir and Freundlich adsorption isotherms of RhB on DSWO2	S-13
13	XPS spectra of recycled DSWO2 photocatalyst	S-14
14	PXRD of recycled DSWO2 photocatalyst	S-15
15	PL spectra of SWO, DSWO1, DSWO2, DSWO3	S-16
16	Comparison table	S-17

Materials characterization

Powder X-ray diffraction (PXRD) measurements were done in 2θ ranging from 5° to 80° by using Rigaku SmartLab rotating anode x-ray diffractometer (9 kV) with Ni-filtered $\text{Cu K}\alpha$ irradiation having wavelength 0.1542 nm with a scanning rate of $2^\circ \text{C min}^{-1}$ with 45 kV and 100 mA. Raman spectroscopic studies were carried out with high-resolution Horiba LabRAM using a 633 nm laser for excitation. FTIR measurements were done on PerkinElmer Spectrum 2 spectrometer in the range 500 to 4000 cm^{-1} using KBr as reference. Photoluminescence (PL) studies were done on Agilent Technologies Cary Eclipse fluorescence spectrophotometer. The absorbance of as-prepared samples was recorded by using diffuse reflectance spectroscopy (DRS) employing polytetrafluoroethylene (PTFE) polymer as a standard on Perkin Elmer UV-visible-NIR lambda 750 spectrophotometer. The morphological studies were done by scanning electron microscope (FEI Nova Nano SEM-450 instrument), and high-resolution images were taken on transmission electron microscopy (TEM) Technai G 20 (FEI) S-twin microscope operating at 200 kV. Energy dispersive x-ray spectroscopy (EDAX) and elemental mapping were done by using the same SEM instrument. X-ray photoelectron spectroscopy (XPS) studies were performed on the samples using ThermoFisher Scientific NEXSA photoemission spectrometer working at 12 kV anode voltage and 6.50 mA filament current using $\text{Al K}\alpha$ (1486.6 eV) anode as a source. The obtained data was analyzed by using Avantage software. The thermogravimetric analysis (TGA) analysis was done on the NETZSCH STA 449 F1 Jupiter, wherein about 2 mg of sample was heated under nitrogen atmosphere from room temperature to 900°C at a heating rate of $10^\circ \text{C min}^{-1}$. The BEL/CAT2 instrument was used for temperature programmed desorption studies (TPD). The Brunauer-Emmett-Teller (BET) surface area studies were carried out at 77 K on Quantachrome Autosorb-iQ-MP-XR system. The photocatalytic experiments were done in a home-built photoreactor setup consisting of two 45 W CFL lamps emitting visible light, and UV-vis spectra of samples collected at various intervals were recorded on Shimadzu UV-2450 spectrophotometer. Further, the degradation products were detected by using Bruker HD compact high resolution mass spectrometer instrument.

Table S1 Calculated crystallite size and lattice strain of SWO and oxygen-vacancy rich DSWO1, DSWO2 and DSWO3 samples.

Sl. No.	Sample Name	Crystallite Size corresponding to $(20\bar{1})$ plane (nm)	Lattice Strain (ϵ)	Lattice Parameters (\AA)
1.	SWO	13.36	0.1657	a = 5.94; b = 4.87; c = 9.12
2.	DSWO1	15.48	0.1396	a = 5.91; b = 4.90; c = 9.13
3.	DSWO2	13.09	0.1687	a = 5.92; b = 4.91; c = 9.11
4.	DSWO3	11.49	0.2286	a = 5.92; b = 4.91; c = 9.14

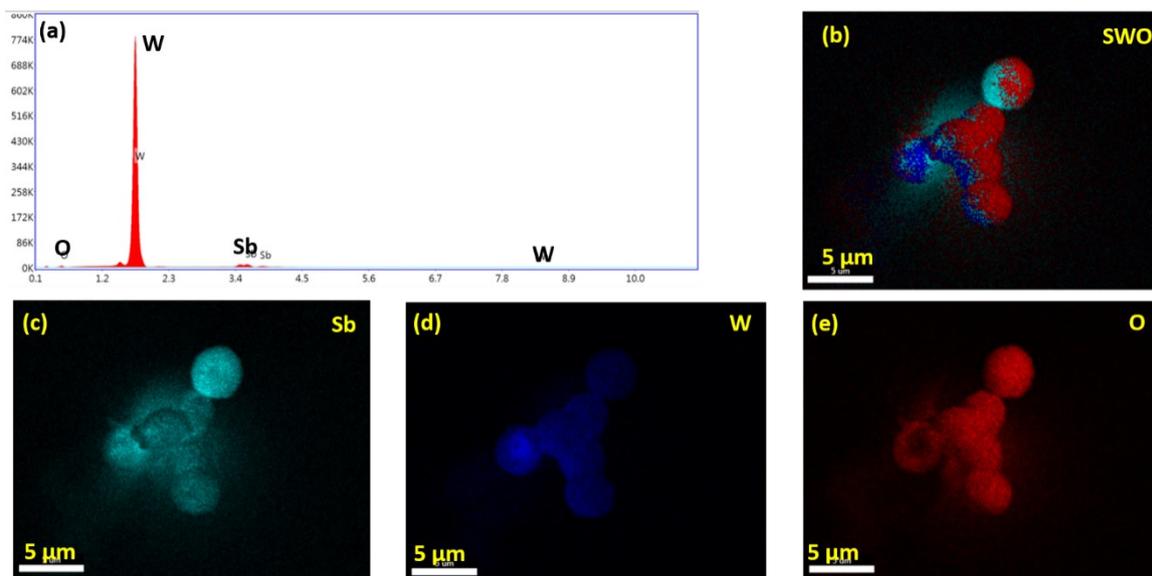


Fig. S1 (a) EDAX spectra and (b-e) elemental spectra of SWO.

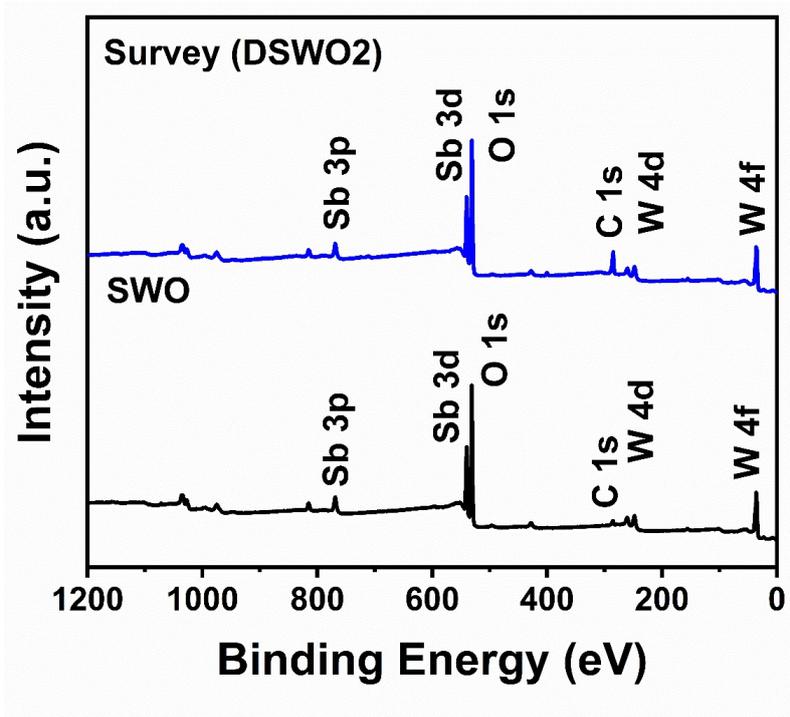


Fig. S2 XPS survey spectra of SWO and DSWO2 samples.

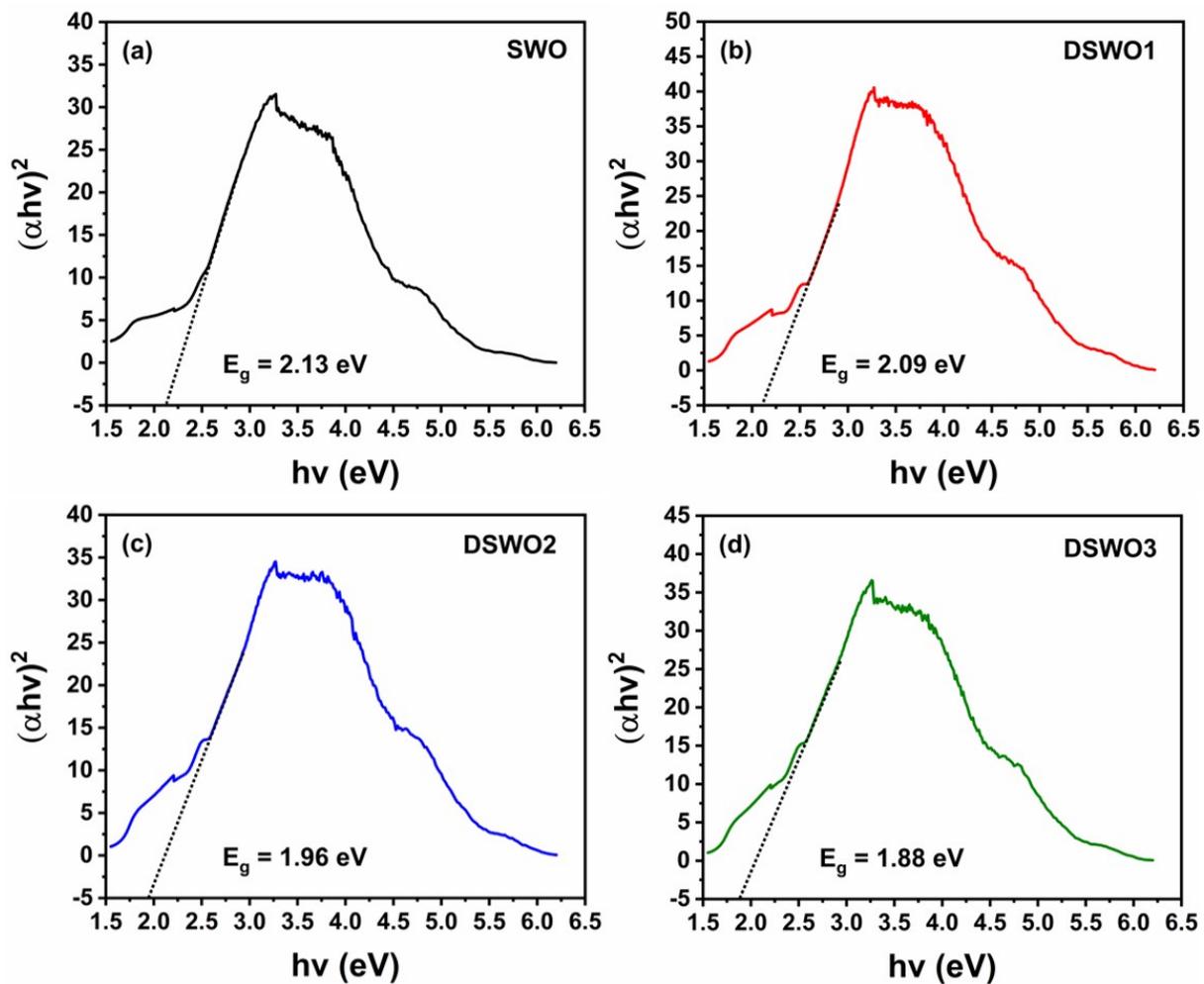


Fig. S3 (a- d) Tauc plots of SWO, DSWO1, DSWO2 and DSWO3 samples.

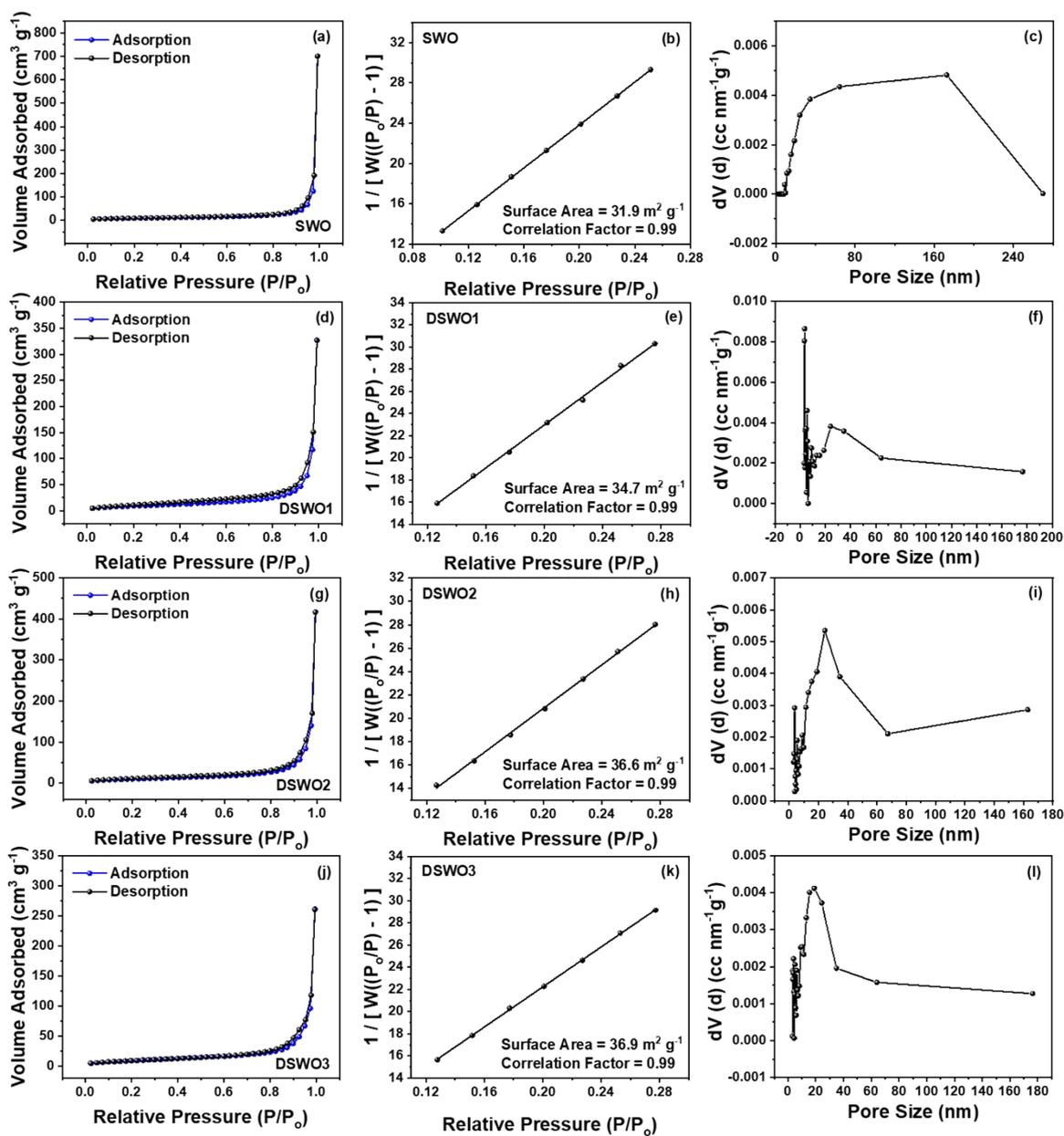


Fig. S4 N_2 adsorption-desorption isotherm, multipoint BET and pore size distribution curves for (a, c) SWO, (d, f) DSWO1, (g, i) DSWO2 and (j, l) DSWO3.

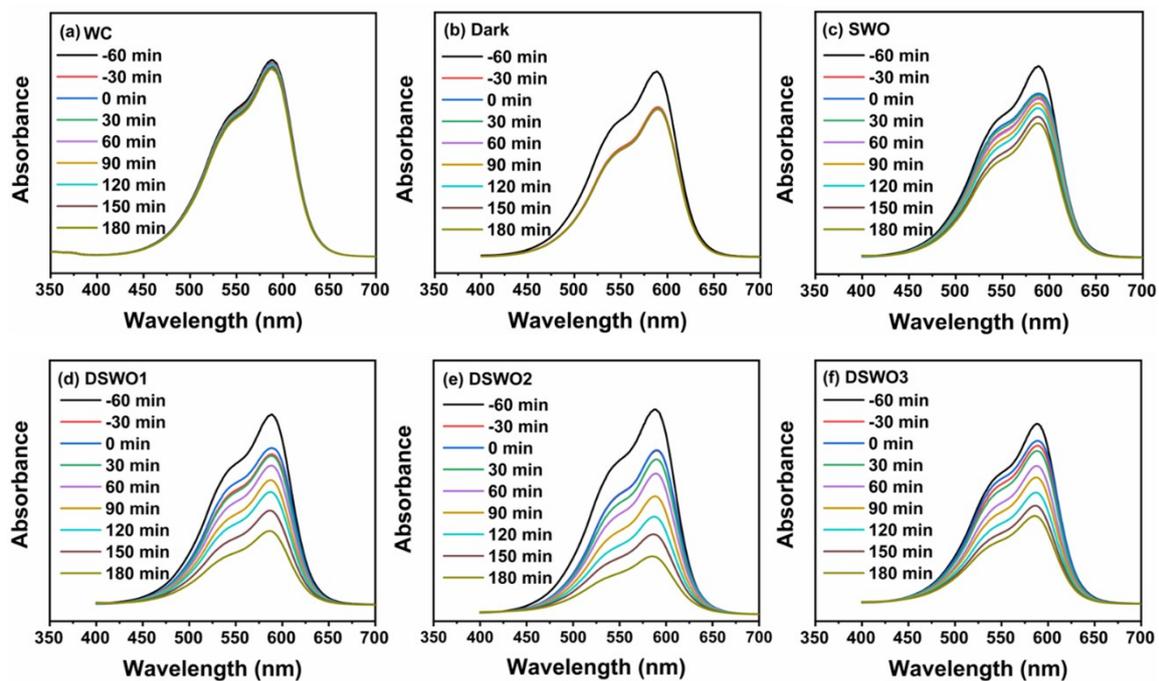


Fig. S5 UV-vis absorption spectra for photocatalytic degradation of CRV dye using (a) WC, (b) dark, (c) SWO, (d) DSWO1, (e) DSWO2 and (f) DSWO3 under visible light irradiation.

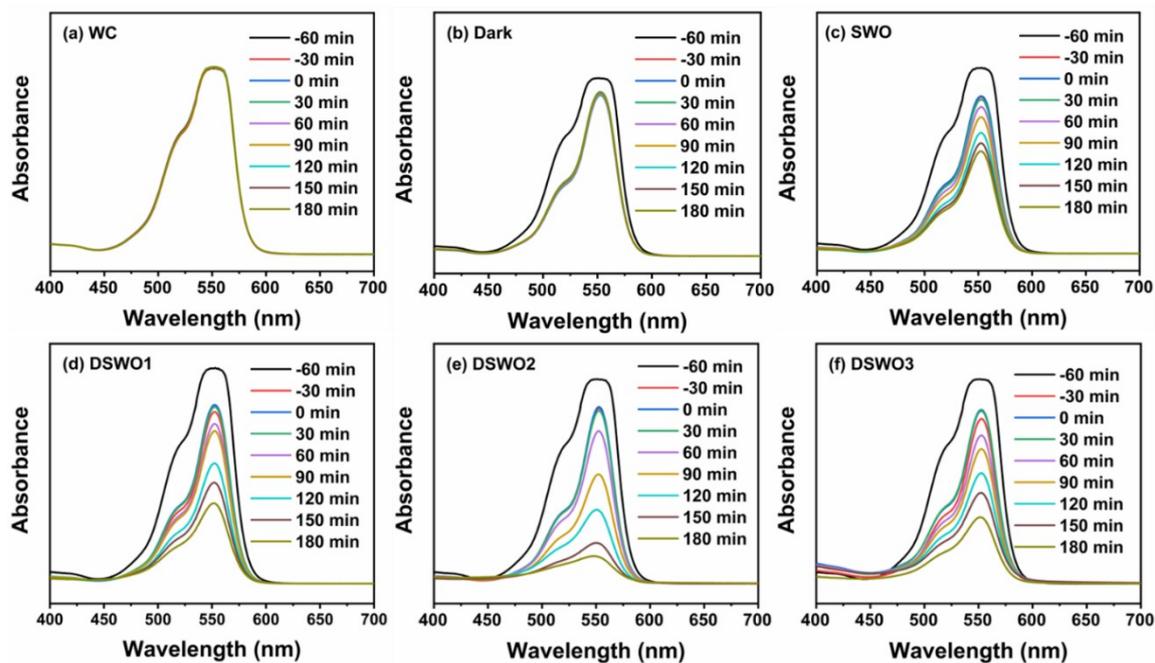


Fig. S6 UV-vis absorption spectra for photocatalytic degradation of RhB dye using (a) WC, (b) dark, (c) SWO, (d) DSWO1, (e) DSWO2 and (f) DSWO3 under visible light irradiation.

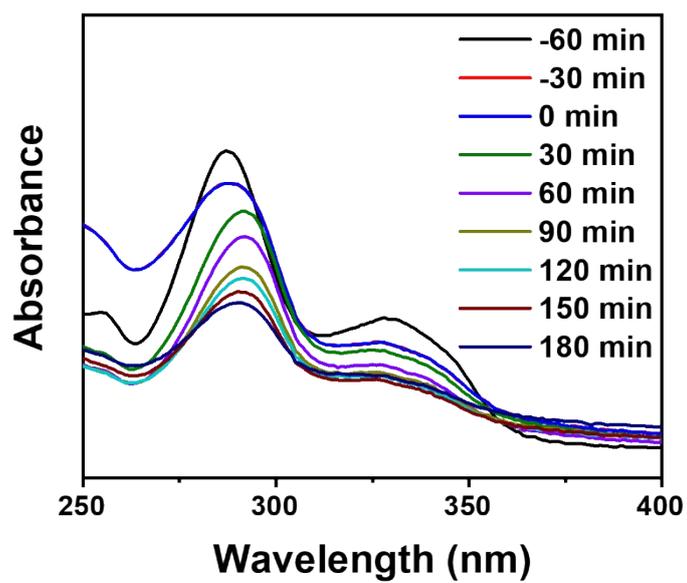


Fig. S7 UV-vis absorption spectra for photocatalytic degradation of LFX over the surface of DSWO2 under visible light irradiation.

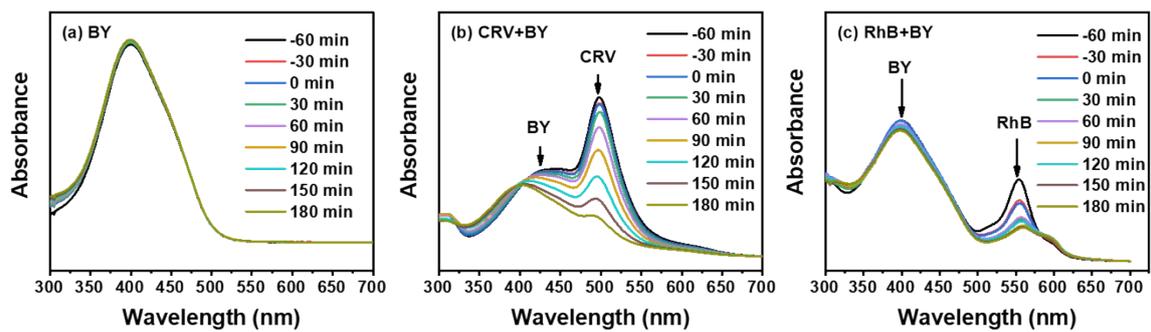


Fig. S8 UV-vis absorption spectra for photocatalytic degradation of (a) BY, (b) CRV+BY and (c) RhB + BY over the surface of DSWO₂ under visible light irradiation.

Table S2 Zeta potential values for SWO, DSWO1, DSWO2 and DSWO3.

Sl. No.	Sample Name	Zeta Potential (mV)	Mean Zeta Potential (mV)
1.	SWO	-38.8 -43.0 -42.2	-41.3
2.	DSWO1	-23.9 -22.4 -23.0	-23.1
3.	DSWO2	-19.0 -20.2 -16.8	-18.6
4.	DSWO3	-19.8 -17.2 -17.7	-18.2

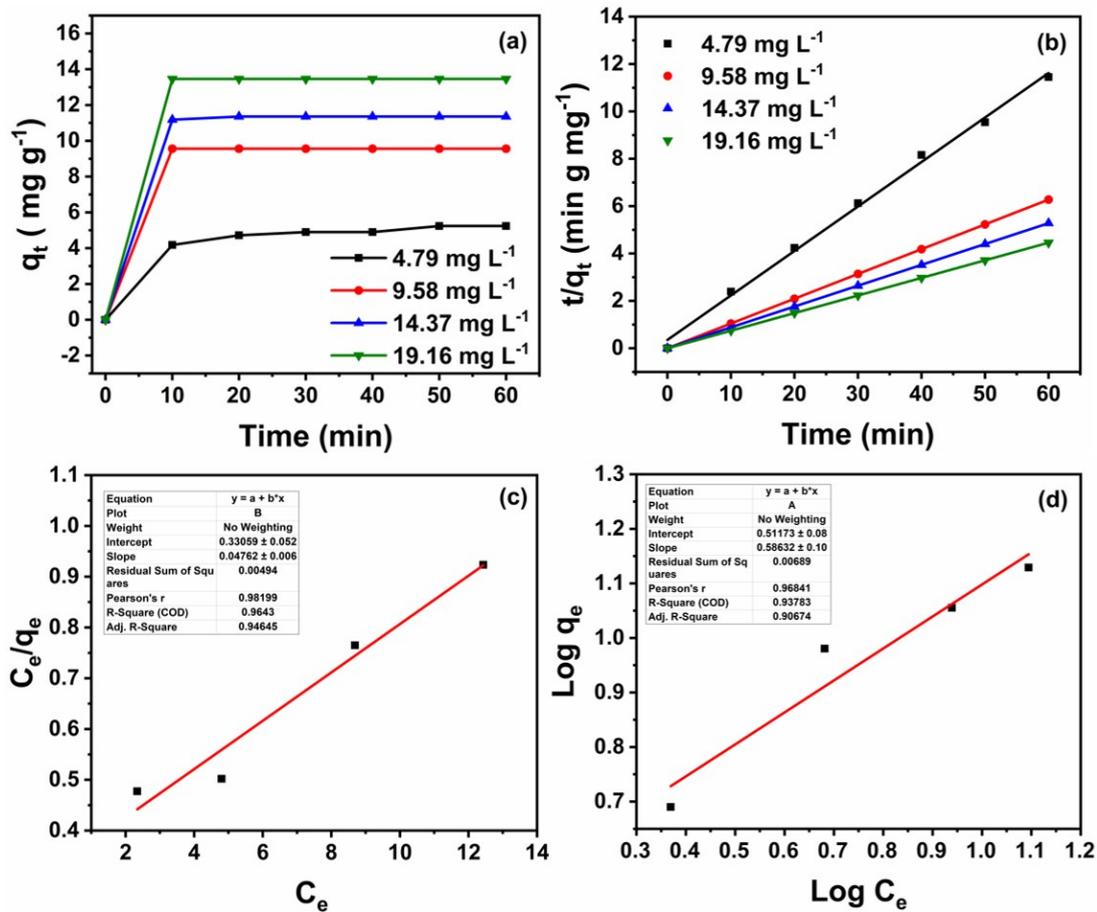


Fig. S9 (a) Time-dependent adsorption study; (b) Pseudo-second-order model for adsorption of RhB on the surface of DSWO2; (c) Langmuir adsorption isotherm and (d) Freundlich adsorption isotherm of RhB on DSWO2.

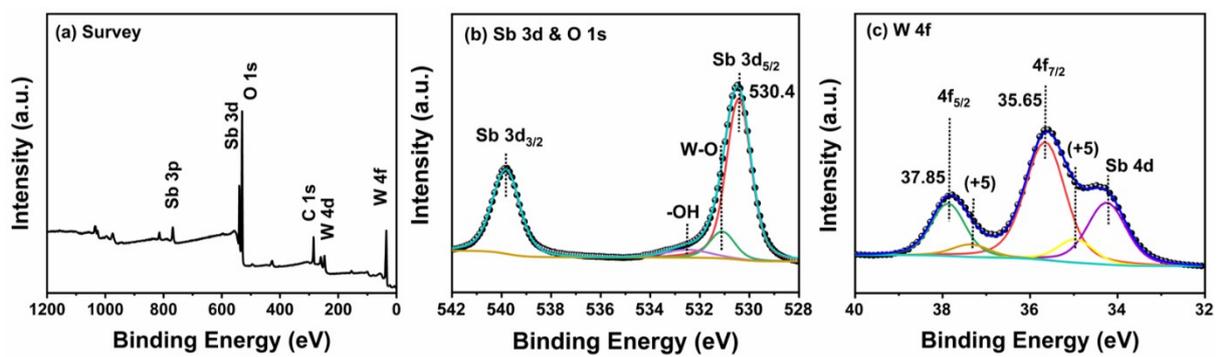


Fig. S10 XPS of recycled DSWO₂ photocatalyst.

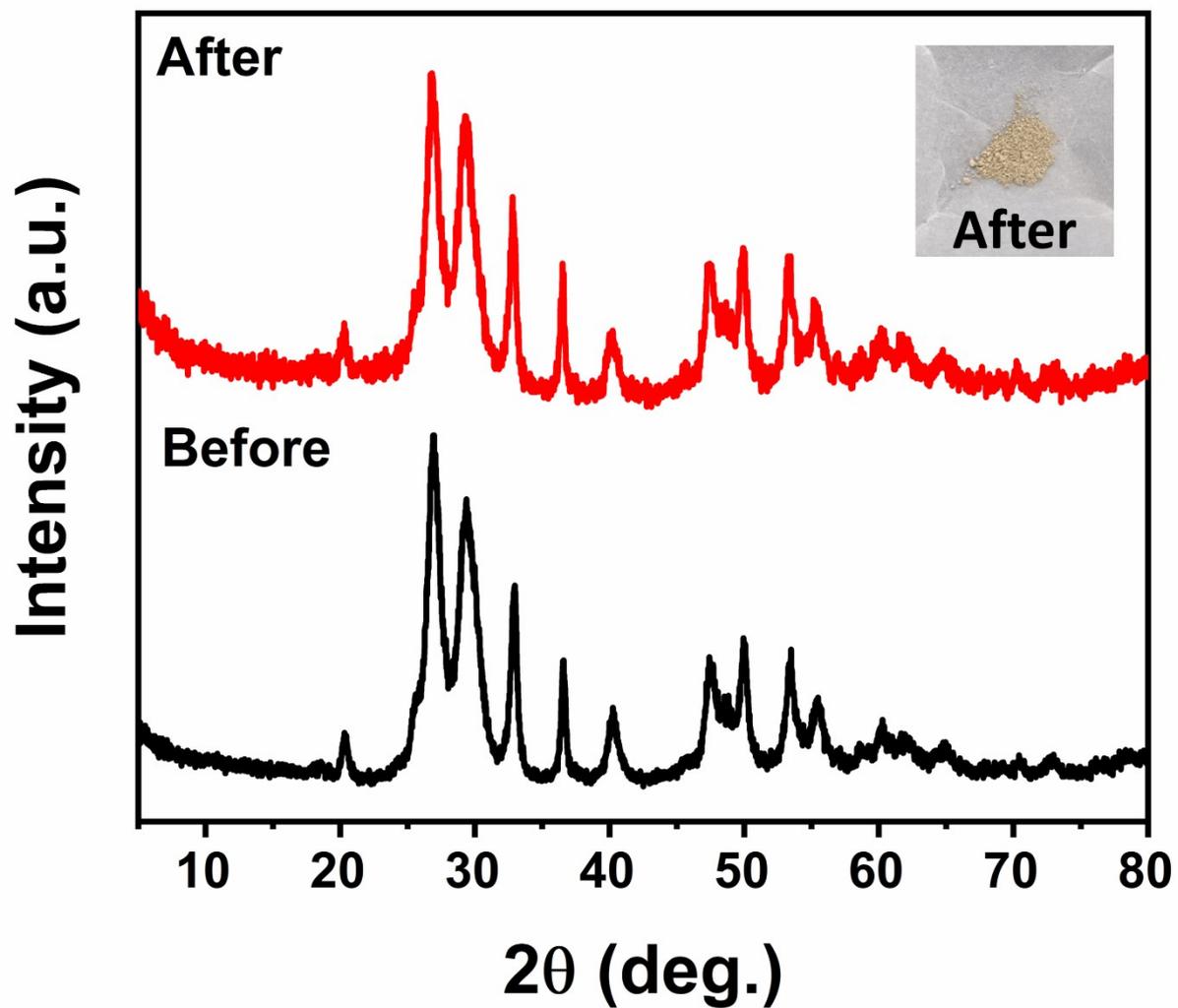


Fig. S11 XRD of DSWO₂ photocatalyst before and after photocatalysis along with the inset of photograph of recycled DSWO₂.

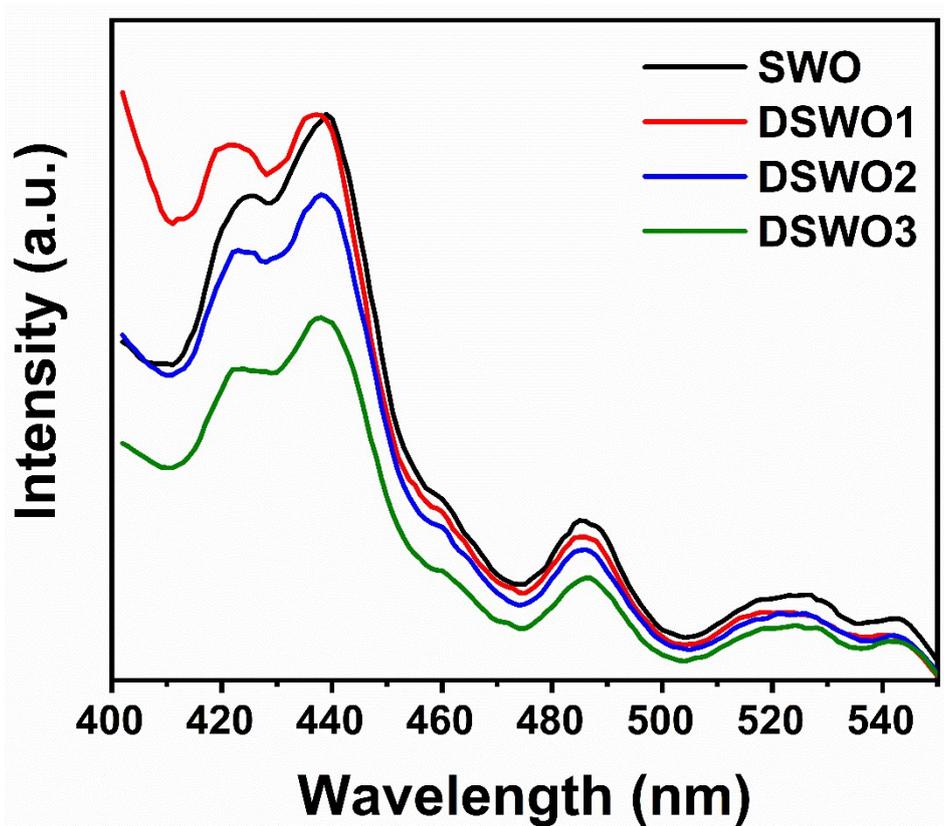


Fig. S12 PL spectra of SWO, DSWO1, DSWO2 and DSWO3 samples.

Table S3 Comparison table of different type of materials towards pollutant degradation.

Sl. No	Photocatalyst	Pollutant Degraded	Concentration of Pollutant	% Degradation and Time	Rate Constant	Year (Ref.)
1.	La-Mn co-doped Fe ₂ O ₃	Rhodamine B	50 ppm = ≈ (1 X 10 ⁻⁴) M	91.78 % (240 min)	0.0092 min ⁻¹	2023 ¹
2.	Cd-doped Bi ₂ MoO ₆	Rhodamine B	10 ppm = ≈ (2 X 10 ⁻⁵) M	97.80 % (70 min)	0.0125 min ⁻¹	2023 ²
3.	Fe-doped TiO ₂	Crystal Violet	10 ppm = ≈ (2.5 X 10 ⁻⁵) M	96 % (180 min)	-----	2023 ³
4.	Tea Leaf Powder/ ZnIn ₂ S ₄	Crystal Violet	10 ppm = ≈ (2.5 X 10 ⁻⁵) M	85.36 % (120 min)	-----	2023 ⁴
5.	Ag ₃ BiO ₃ /ZnO/B C	Levofloxacin	10 ppm = ≈ (2.8 X 10 ⁻⁵) M	95.8 % (120 min)	0.0118 min ⁻¹	2023 ⁵
6.	Sc ₂ VO _{5-δ} /g- C ₃ N ₄	Levofloxacin	12 ppm = ≈ (3.3 X 10 ⁻⁵) M	89.1 % (80 min)	0.0234 min ⁻¹	2022 ⁶
7.	Oxygen vacancy rich Sb ₂ WO ₆	Crystal Violet	6 X 10 ⁻⁵ M	71 % (180 min)	0.0045 min ⁻¹	This Work
		Rhodamine B	4 X 10 ⁻⁵ M	86 % (180 min)	0.0080 min ⁻¹	
		Levofloxacin	5 X 10 ⁻⁵ M	52 % (180 min)	---	

References

1. S. Maqbool, A. Ahmed, A. Mukhtar, M. Jamshaid, A. U. Rehman and S. Anjum, *Environmental Science and Pollution Research*, 2023, **30**, 7121-7137.
2. B. Zhang, C. Fang, J. Ning, R. Dai, Y. Liu, Q. Wu, F. Zhang, W. Zhang, S. Dou and X. Liu, *Carbon Neutralization*, 2023, **2**, 646-660.
3. A. Mancuso, N. Blangetti, O. Sacco, F. S. Freyria, B. Bonelli, S. Esposito, D. Sannino and V. Vaiano, *Nanomaterials*, 2023, **13**, 270.
4. M. Chand, S. Barthwal, A. S. Rawat, M. Khanuja and S. Rawat, *ACS omega*, 2023, **20**, 17880–17890.
5. G. Dong, W. Chi, D.-f. Chai, Z. Zhang, J. Li, M. Zhao, W. Zhang, J. Lv and S. Chen, *Applied Surface Science*, 2023, **619**, 156732.
6. J. Feng, L. Zu, H. Yang, Y. Zheng, Z. Chen, W. Song, R. Zhao, L. Wang, X. Ran and B. Xiao, *RSC advances*, 2023, **13**, 688-700.