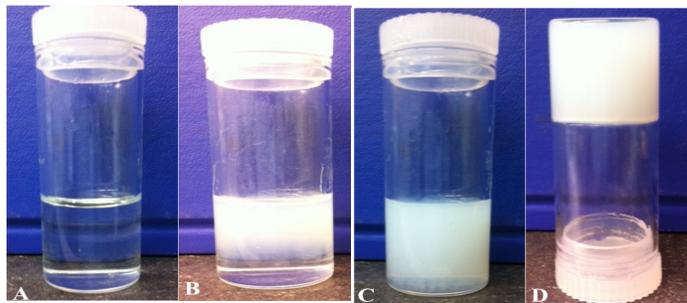


Electronic Supporting Material

1. Experimental section

The synthetic procedure for the preparation of the ZnO-SnO₂ nanoparticles is very straightforward. For example, in a typical procedure, (0.8 mmol, 0.2380 g) of Zn(NO₃)₂•6H₂O (J.T. Baker) and (0.8 mmol, 0.2805 g) SnCl₄•5H₂O was dissolved in (3.0 mL) 2-propanol (Fisher) with stirring approximately 20s to yield a colorless solution. Subsequently, 10 equivalents of propylene oxide (ACROS) was quickly added, shaken and set aside to gel undisturbed. Gels begin to form with 20 s and complete gelation was complete after ~60s.



ESI Fig S1: Gel formation: a) $t=0s$, b) at 30s, c) 50s, d) 60s (*inverted to illustrate stability*)

Upon gelation, the resulting wet gels were washed with acetone 3-5 days with fresh acetone exchanged daily. Afterwards, the gels were placed into the critical point dryer where acetone was exchanged for liquid CO₂ for 3-5 days, after which time the temperature was increased from 10°C to 40°C to achieve a pressure around 1100 psi. At this time, the dryer was slowly depressurized over a 2-3 h period to obtain porous, monolithic aerogel networks.

For calcination studies, the as-synthesized aerogels were annealed to 500°C in a programmable furnace. The temperature was increased slowly from 25°C to 500°C at 2°C min⁻¹ and then permitted to dwell for 4h before returning to room temperature at 2°C min⁻¹.



ESI Fig. S2: Free standing aerogel after supercritical drying

2. Physical characterizations

Powder x-ray Diffraction

The powder x-ray diffraction patterns of both the as-synthesized and annealed samples were recorded with a Rigaku Ultima III diffractometer using Cu K α radiation. To prepare the samples, they were, first, finely ground before being placed on the sample holder. The collection was taken at a 2θ range of 10-80° at a step-width of 0.03° sec⁻¹. The diffraction patterns were then identified by comparison to the phases in the International Centre for Diffraction Data (ICDD) powder diffraction file (PDF) database.

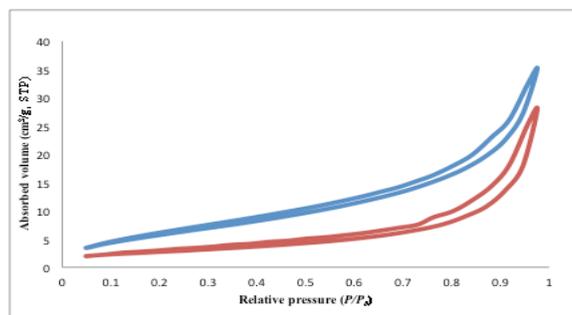
Electron Microscopy

The surface morphology of the ZnO-SnO₂ aerogels was studied using a high-resolution scanning electron microscopy (SEM) Hitachi S-4300. The aerogel powders were mounted on an aluminum stub with carbon tape. The microscope was operated between 2 and 10 kV to minimize charging effects.

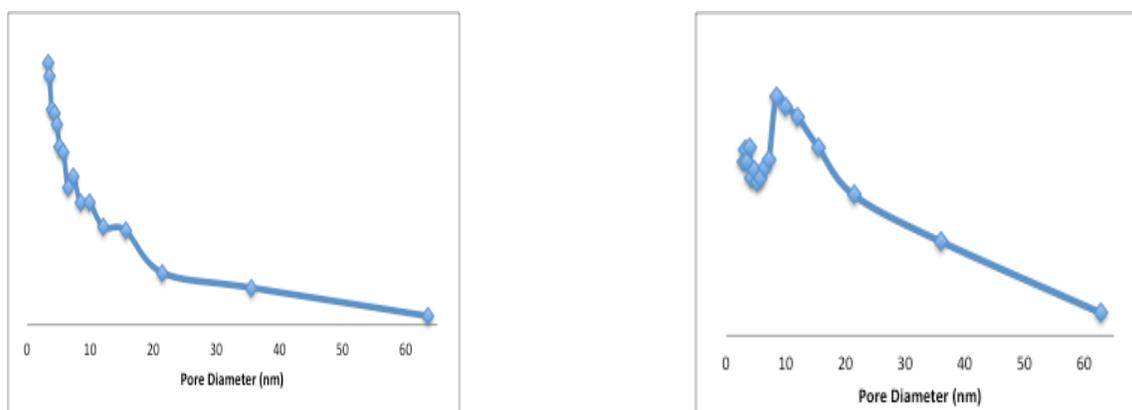
High-resolution transmission electron microscopy (HRTEM) analyses were conducted using a JEOL JEM-2100 at an accelerating voltage of 200kV. The aerogel samples were prepared by suspending a small amount of the sample into 2-propanol and sonicating for 5 minutes. Afterwards, one drop was placed onto an ultrathin carbon-400 mesh Cu grid and permitted to dry before imaging.

N₂ adsorption/desorption

The specific surface area, pore volume and average pore sizes were obtained from the N₂ adsorption/desorption analyses conducted at 77K on a NOVA 4200e model Surface area/pore size analyzer (Quantachrome Instrument Corp.) The Brunauer-Emmett-Teller (BET) specific surface areas were calculated from the adsorption branch of the isotherm. The pore volume, pore sizes and average size distribution was taken from the desorption branch of the isotherm. Prior to obtaining any results, the samples were placed on degas for 24h at 100°C. Each set of measurements was taken at 77 mmHg and an equilibrium time of 600s, which resulted in a total experimentation time of 6-9h.



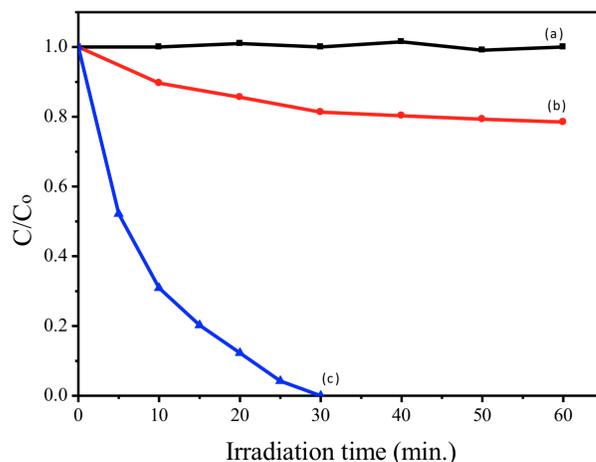
ESI Fig. S3: N₂-adsorption/desorption isotherms of as-synthesized (*upper*) and annealed, ZnO-SnO₂.



ESI Fig. S4: Pore size distribution plots of as-synthesized (*left*) and annealed (*right*).

Photocatalytic Activity

The photocatalytic activity experiments on ZnO-SnO₂ nanoparticles for the degradation of Rhodamine B (RhB) were conducted at room temperature. Typically, 5 mg of ZnO-SnO₂ nanocomposites were added to a 3 mL of 1.2×10^{-5} M of RhB aqueous solution in a quartz cuvette. The solution was magnetically stirred at all times to ensure dispersion of the nanoparticles. The nanocomposite containing solutions were then exposed to ultraviolet light (250 W, $\lambda=365$ nm) at room temperature. The samples were situated at 50 cm from the lamp filament to prevent temperature overheating. Absorbance spectra were recorded using a Lambda 1050 UV/Vis/NIR (Perkin-Elmer, UK) spectrometer at 0.5 nm resolutions equipped with in situ magnetic stirrer.



ESI Fig. S5: The normalized absorbance change of 1.2×10^{-5} M of 3 mL of RhB aqueous solution monitored at 550 nm: (a) In the dark with the presence of 5 mg of ZnO-SnO₂ photocatalyst, (b) In the absence of the catalyst under UV light, (c) Upon illumination with UV light in presence of 5 mg of ZnO-SnO₂.