

Online Supporting Information's for

Shape-selective Synthesis of Sn(MoO₄)₂ Nanomaterials for Catalysis and Supercapacitor Applications

K. Sakthikumar, Sivsankara Rao Ede, Soumyaranjan Mishra
and Subrata Kundu*

Electrochemical Materials Science (ECMS) Division, CSIR-Central Electrochemical Research Institute (CECRI), Karaikudi-630006, Tamil Nadu, INDIA.

* To whom correspondence should be addressed, *E-mail:* skundu@cecri.res.in; kundu.subrata@gmail.com, Phone: (+ 91) 4565-241487, FAX: +91-4565-227651.

Instruments.

The shape-selective Sn(MoO₄)₂ nanomaterials were characterized by using several spectroscopic techniques. The UV-Visible (UV-Vis) absorption spectra were recorded in a double beam spectrophotometer purchased from Unico (model 4802 UV-Vis spectrophotometer). The transmission electron microscopy (TEM) analysis was done with a Tecnai model TEM instrument (TecnaiTM G2 F20, FEI) with an accelerating voltage of 200 KV. The Field Emission Scanning Electron Microscopy (FE-SEM) analysis was recorded with Zeiss ultra FE-SEM instruments. The Energy Dispersive X-ray Spectroscopy (EDS) analysis was done with the same FE-SEM instrument with a separate EDS detector (INCA) connected to that instrument. The X-ray diffraction (XRD) analysis was done using a PAN analytical Advanced Bragg-Brentano X-ray powder diffractometer (XRD) with Cu K α radiation ($\lambda = 0.154$ nm) with a scanning rate of 0.020 s⁻¹ in the 2 θ range 10-80°. The X-ray photoelectron spectroscopic (XPS) analysis was done to check the chemical composition and the state of elements present in the outermost part of materials and analyzed by using Theta Probe AR-XPS System, Thermo Fisher Scientific (U.K.). The LASER Raman measurements were carried out with Renishaw inVia Raman Microscope using an excitation wavelength of 633 nm (He-Ne laser). The excitation light intensity in front of the objective was ~10 mW with a spectral collection time of 1 sec for Raman experiment. The integration time for our measurement was set to 10 sec. The thermal analysis study was recorded with a thermal

analyser-simultaneous TGA/DTA instrument with model name SDT Q600 and the analysis was performed in air. The Fourier Transform Infrared (FT-IR) spectroscopy analysis was done with the model Nexus 670 (FT-IR), Centaurms 10X (Microscope) having spectral Range 4,000 to 400 cm^{-1} with a MCT-B detector. A hot air oven (temperature up to 1000 $^{\circ}\text{C}$) was used to anneal the samples at specific temperature. The UV photo-irradiation was done with a UV-lamp (UVP-Black Ray model, B-100A, AP High Intensity UV lamp, 365 nm maximum wavelength) having a lamp power of 100 watt and working in 230 V – 50 HZ to irradiate the nitroarenes for catalysis work. The distance of the sample stage from the light source was ~ 10-15 cm. The sample solution was kept over a wood piece such that the UV light falls directly onto the sample solution. The supercapacitor study were examined using an electrochemical work station CHI 6034C in 3(M) KOH aqueous solution.

FT-IR bands – cetyl trimethyl ammonium bromide (CTAB)-Experimental and Reported values		
FT-IR bands (cm^{-1}) (experimentally observed)	FT-IR frequency range (cm^{-1}) (reported value)	Absorbing bonds/vibration types
3339, 3649	3330, 3600	-N-H stretching vibration; -O-H stretching vibration
3015	2959, 2870	-CH ₃ - anti-symmetrical and symmetrical stretching
2921, 2849	2916, 2828	-(CH ₂)- anti-symmetrical and symmetrical stretching
1388	1376	-CH ₃ - symmetric deformation
1139, 1240, 1299	1300-1000 (1041,1151,1265)	-(CH ₂)- out-plane swinging
1046	1040	Symmetric stretching vibration C-N bonds
908, 959, 1472	908, 961, 1472	Tertiary Amine [RN(CH ₃) ₃ ⁺]
728	720, 728	-(CH ₂)- in-plane swinging
Reference: Huang, L.; Chen, X.; Li, Q. <i>J. Mater. Chem.</i> 2001 , 11, 610.		

Table S-1: The specific FT-IR band of CTAB reported and the bands observed with corresponding band assignments are given.

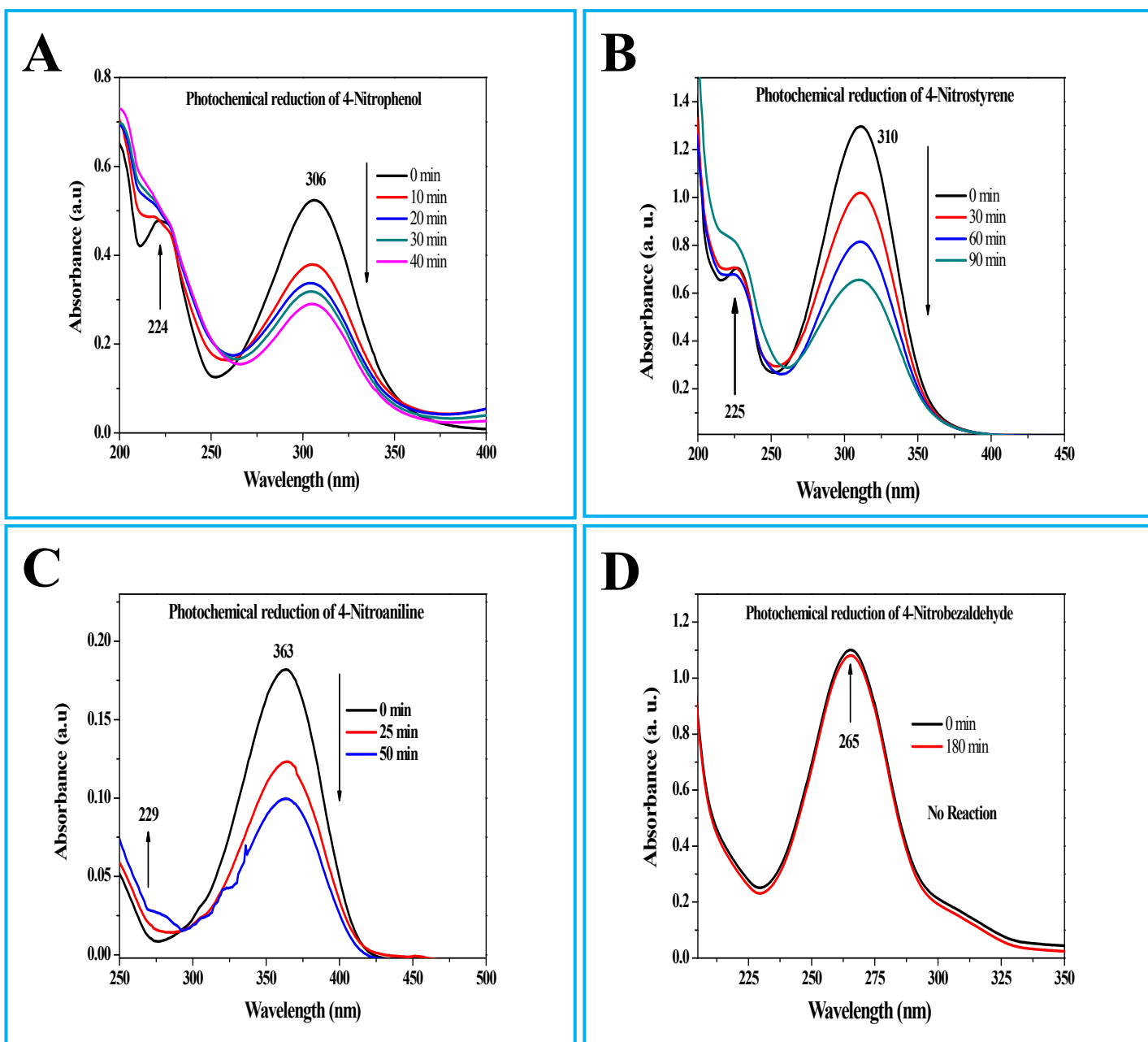


Figure S-1. The successive decrease in absorbance value for the reduction of nitroarenes using $\text{Sn}(\text{MoO}_4)_2$ flake-like nanomaterials as catalyst for 4-NP (Figure S-1A), 4-NS (Figure S-1B), 4-NA (Figure S-1C) and 4-nitro benzaldehyde (Figure S-1D) respectively.

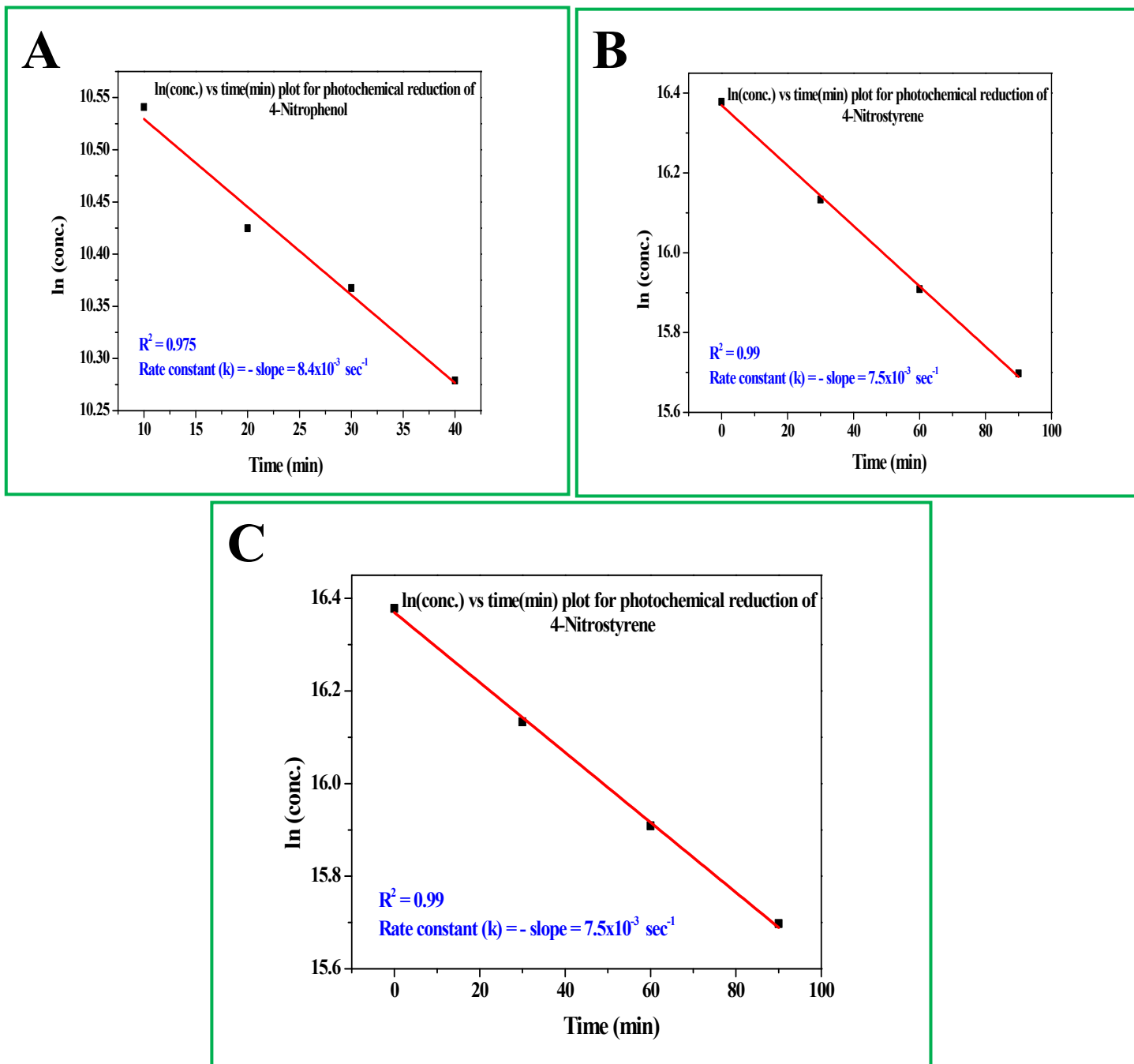


Figure S-2. ln (conc.) versus time (min) plot for the determination of rate constant for catalytic reduction of nitroarenes using flake-like $\text{Sn}(\text{MoO}_4)_2$ nanomaterials as catalyst for 4-NP (Figure S2-A), 4-NS (Figure S2-B), 4-NA (Figure S2-C) respectively.