Synthesis of binary $NiCo_2O_4$/ZnO composites as efficient photocatalysts for methylene blue degradation under visible light

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S1 Catalysts Characterization

The crystal structure of the prepared catalysts was analysed through the powder X-ray diffraction technique (PXRD, Bruker D8 Advance, Central X-ray Facility, IISc Bengaluru). The Fourier Transform Infrared Spectroscopy (Bruker-alpha FTIR spectrophotometer) measures the samples with the spectral range of 500-4000 cm^{-1} with a scan rate of 32 attenuated total reflectance. Morphological analysis was carried out by scanning electron microscopy technique (SEM) equipped with EDS. The optical performance of the composites was analysed through UV-Vis DRS with 200-800 nm (Perkin Elmer, CRF CeNS Bengaluru India). Photoluminescence (PL) spectra were examined by Agilent Cary Eclipse Fluorescence spectrometer using Xe lamp with an excitation wavelength of 350 nm. X-ray photoelectron spectroscopy (XPS) were carried out on an ICMS Lab X-ray photoelectron spectrometer (Thermofisher) using the Micro-focused Al K-a radiation. Zeta potential (ξ) was measured using the Malvern Nano ZS model at TSAMRC lab CRF CeNS, Bengaluru. LC/MS analysis was performed with an Acquity UPLC and Synapt G2 mass spectrometer system (Waters Corporation, MA, USA) equipped with an Acquity UPLC- BEH-C18 column and a SYNAPT G2 detector (1.7 µm, 2.1 ×50 mm, Waters Corporation, MA, USA).

S2 Photoelectrochemical analysis.

A standard three-electrode system was used to analyse the photoelectrochemical measurements of the prepared photocatalysts. The working electrode was a fluorine-doped tin oxide (FTO) substrate (sample dispersed in ethanol and coated onto the FTO plate). The counter electrode was a platinum wire, and the electrolyte was a 0.5 M Na_2SO_4 solution (pH 7) with Ag/AgCl in saturated KCl as the reference electrode in the electrochemical cell. A 300 W Xe lamp with an intensity of 100 W/cm² was utilized as the light source to provide illumination from the back side of the FTO. The electrochemical impedance spectra (EIS) studies were conducted in a dark environment.

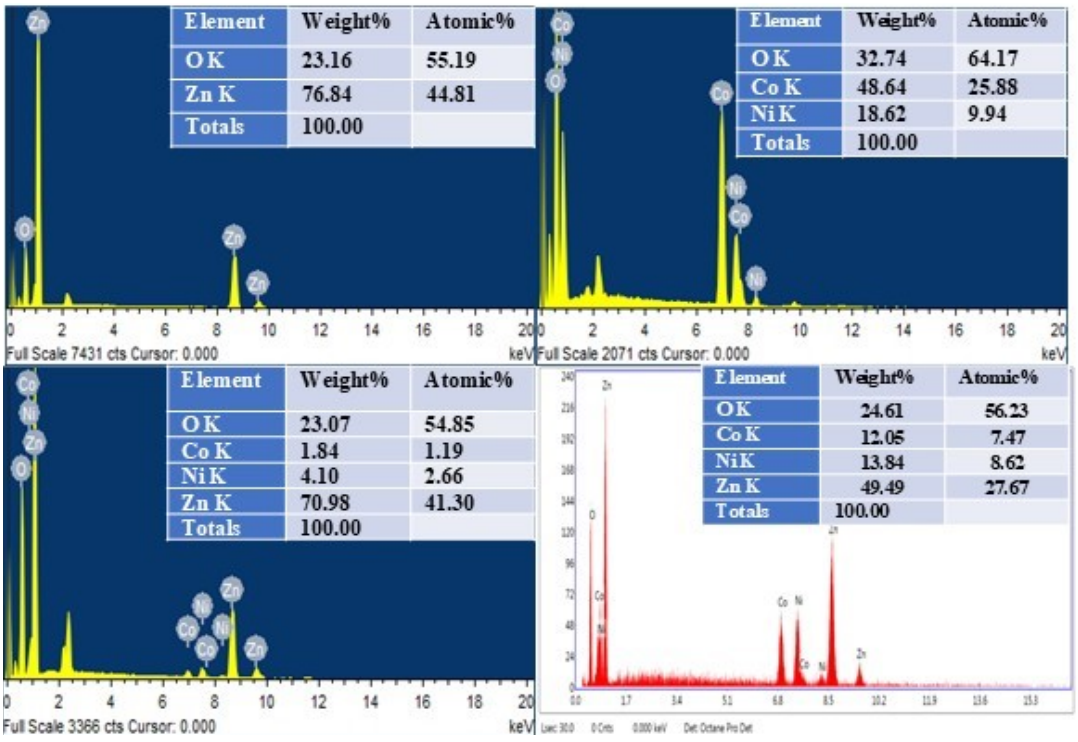
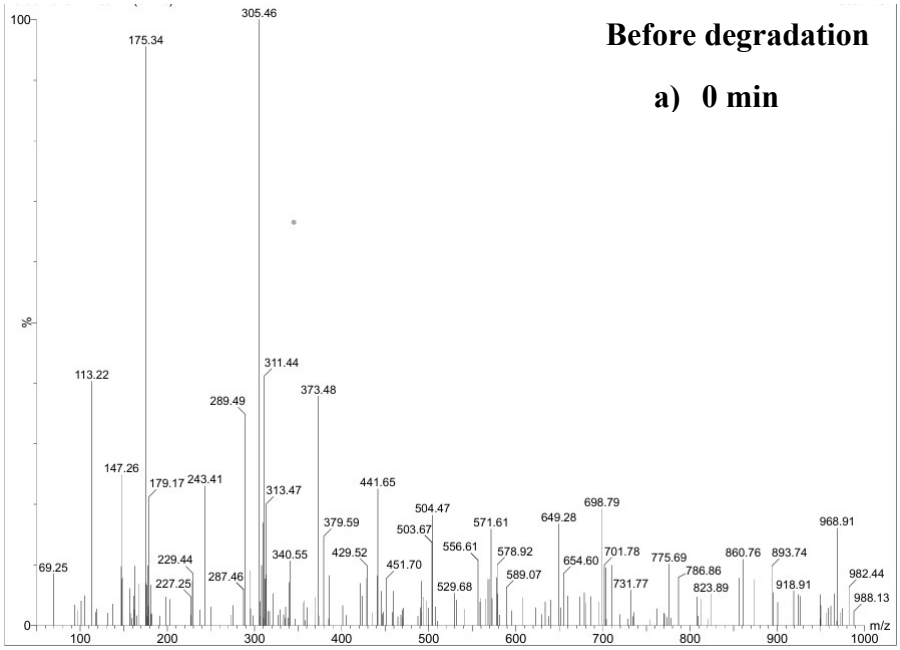
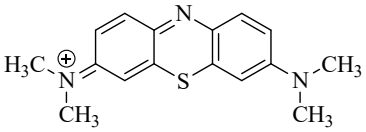


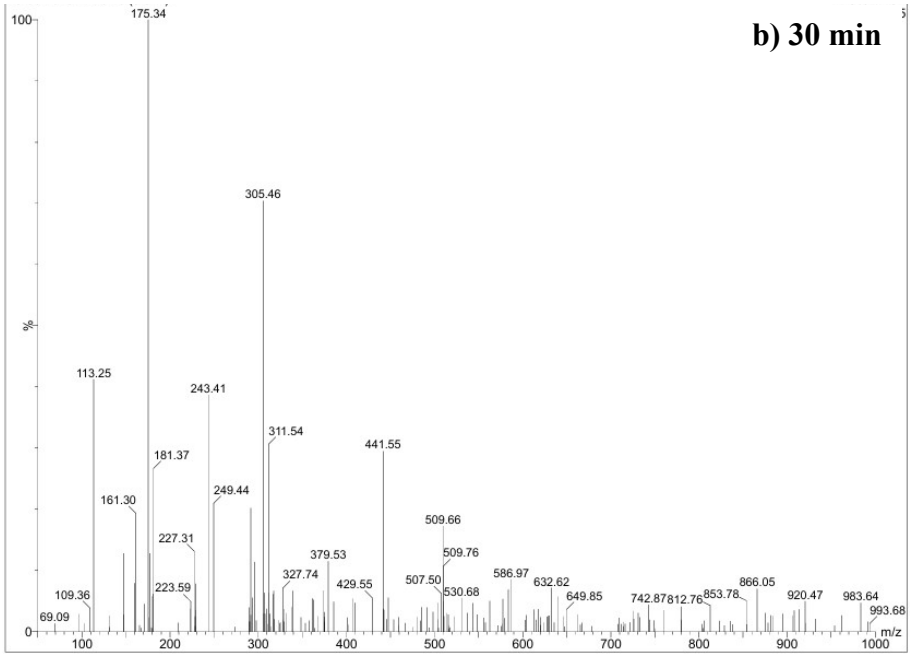
Figure S1: EDS spectra of ZnO (a), $NiCo_2O_4$ (b) $NiCo_2O_4$/ZnO@0.3 (c) and $NiCo_2O_4$/ZnO@0.05 (d).



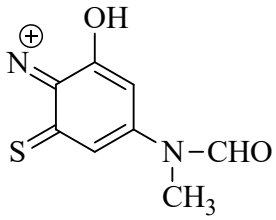
Before degradation

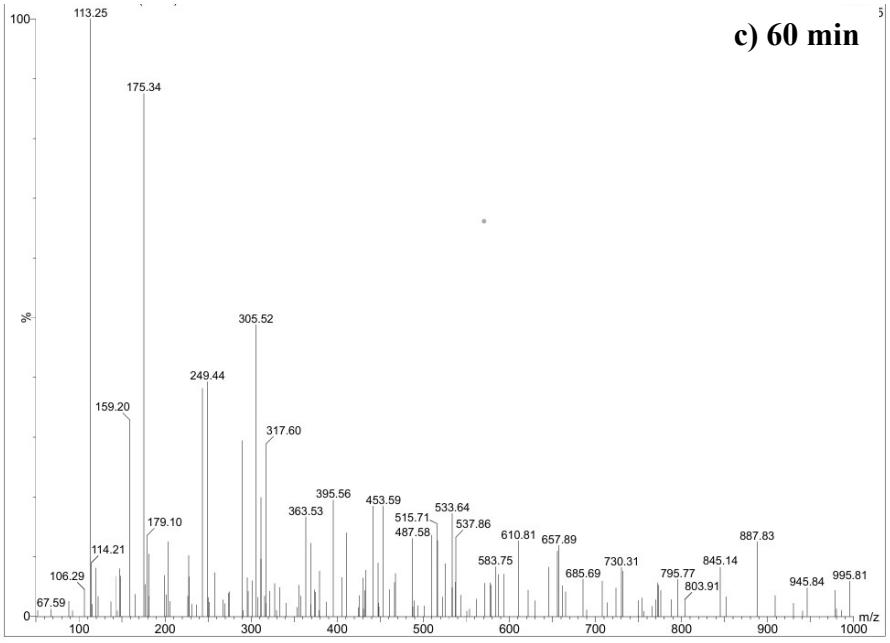
a) 0 min

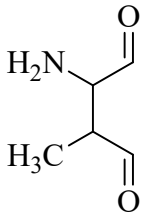


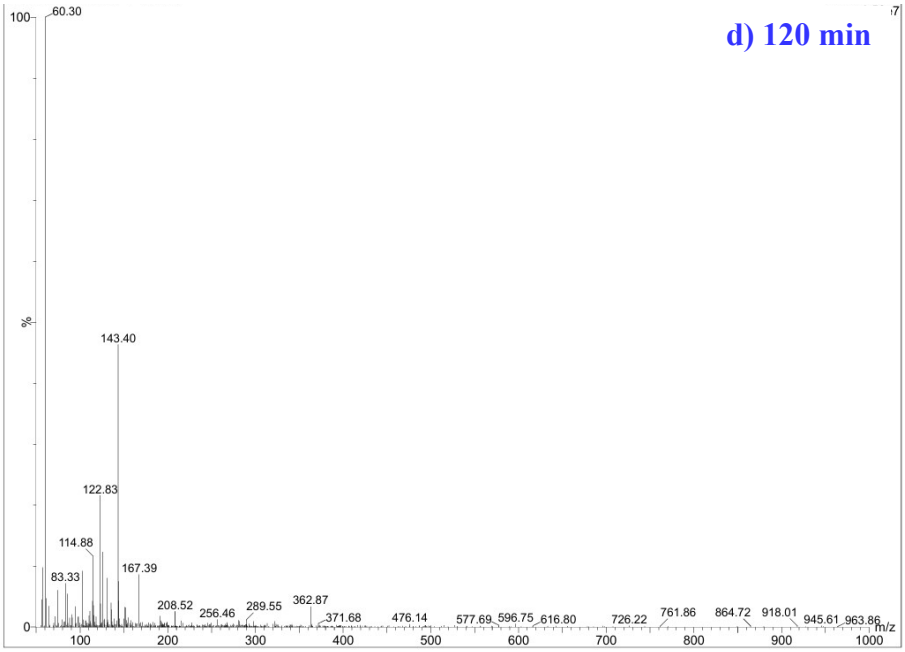


b) 30 min









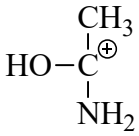


Figure S2. Intermediate compounds m/z values identified through LC-MS analysis.

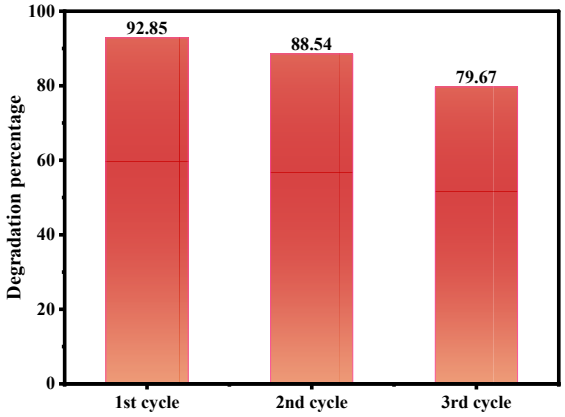


Figure S3. Recyclability test for the $NiCo_2O_4$/ZnO@0.05 composite for 3 successive cycles.