

Waste-to-Work Photocatalysis: Orange-Peel-Derived CQD@CdS/Biochar Hybrids Enabling Efficient Visible-Light-Driven Dye Mineralisation and Mechanistic Elucidation

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S1. Characterisation

An extensive analysis was carried out utilizing advanced technologies to study CdS, CQD, Biochar, and the composites derived from these materials (5CC, 10CC, 15CC). The crystal structures were investigated across a 2θ range of $5-90^\circ$ using X-ray diffraction (XRD) with a Pan Analytical X'Pert-Pro diffractometer, employing $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). The composites' size, elemental composition, and surface characteristics were examined through High-Resolution Transmission Electron Microscopy (HR-TEM, FEI Tecnai G2 F20) and Field Emission Scanning Electron Microscopy (FESEM-EDS, JEOL). The chemical states of the metal oxides were analyzed using X-ray Photoelectron Spectroscopy (XPS, Omicron ESCA), with the XPS spectra calibrated to the C 1s peak at 284.8 eV for accurate binding energy measurements. Peak deconvolution was performed with Gaussian fitting in OriginPro 2024 64Bit to resolve overlapping peaks. Fourier Transform Infrared (FTIR) spectroscopy (Shimadzu IRTracer-100) was employed to identify functional groups, calibrated with a polystyrene reference standard to ensure spectral precision. Baseline correction and spectral analysis were also conducted using OriginPro 2024 64Bit. The surface area, pore size, and volume were assessed using N_2 adsorption-desorption isotherms on a Microtrac BELSORP MINI II, with BET and BJH methods applied for analysis. The photoluminescence spectra were recorded using a spectrofluorometer (Shimadzu RF-6000). Mott-Schottky measurements were performed using a three-electrode electrochemical workstation to determine the semiconductor type and flat band potential of the photocatalyst. The working electrode was prepared by dispersing the photocatalyst in ethanol and DI water containing Nafion as a binder to obtain a uniform slurry, which was subsequently drop-cast onto a pre-cleaned fluorine-doped tin oxide (FTO) glass substrate and dried under room temperature. A platinum wire and Ag/AgCl electrode served as the counter and reference electrodes, respectively. The electrochemical

measurements were conducted in an electrolyte composed of 0.5 M Na₂SO₄ prepared in phosphate buffer solution (PBS) at neutral pH. To investigate the degradation products and possible intermediate byproducts of the degradation process, High Resolution Mass Spectrometry was employed.

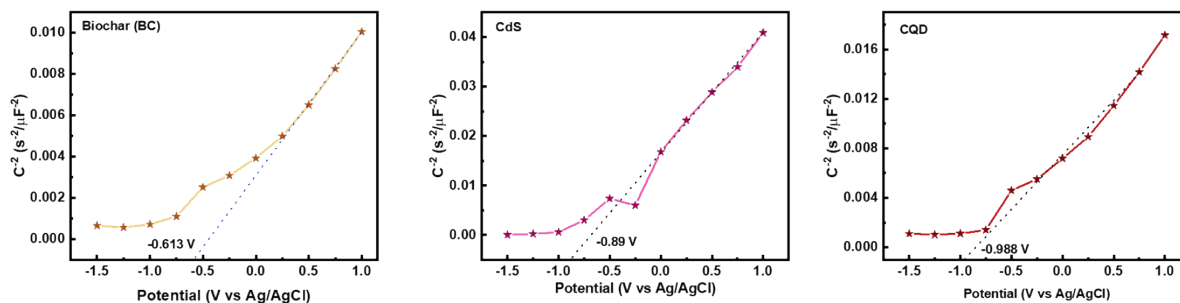


Figure S1: Mott-Schottky plot of the BC, CdS, and CQD recorded showing n-type semiconductor behavior.

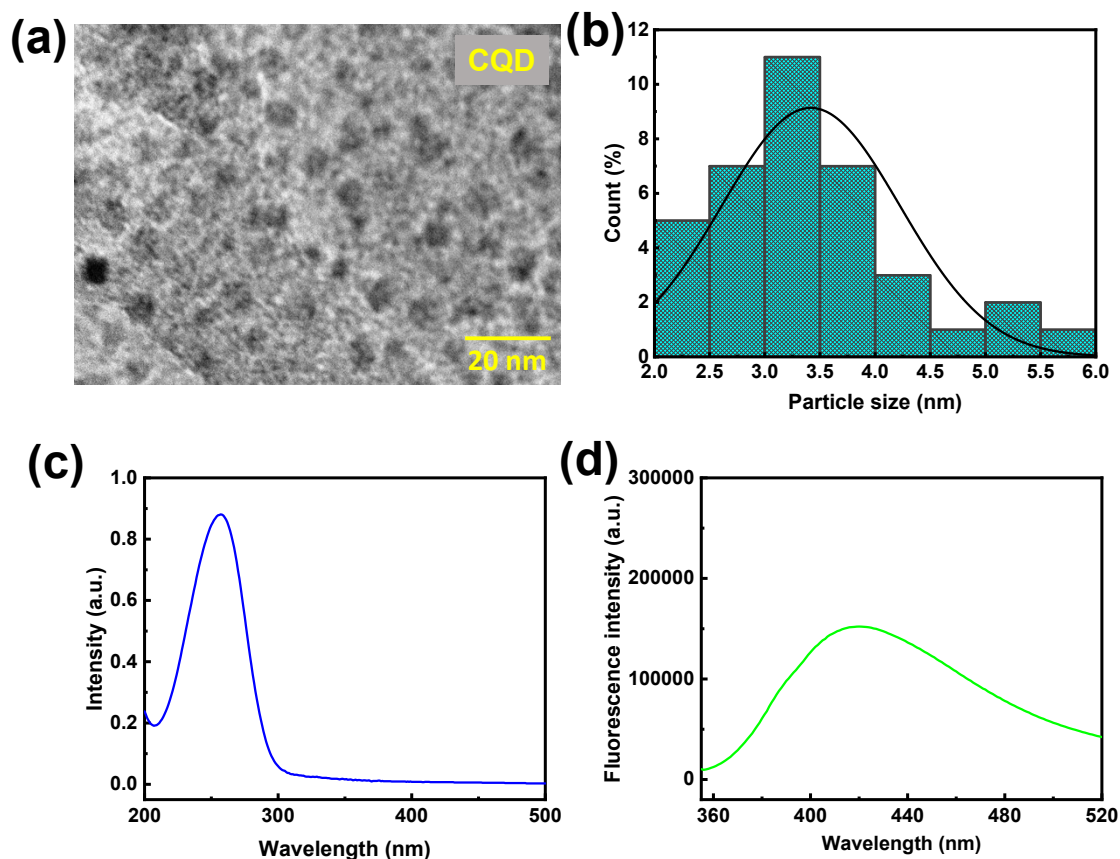


Figure S2: Characterization of the synthesized CQD: (a) HRTEM image showing the morphology and dispersion of CQD, (b) particle size distribution histogram calculated from the HRTEM images, (c) UV-Vis absorption spectrum of CQD, and (d) fluorescence (PL) emission spectrum confirming the characteristic photoluminescent behavior of CQD.

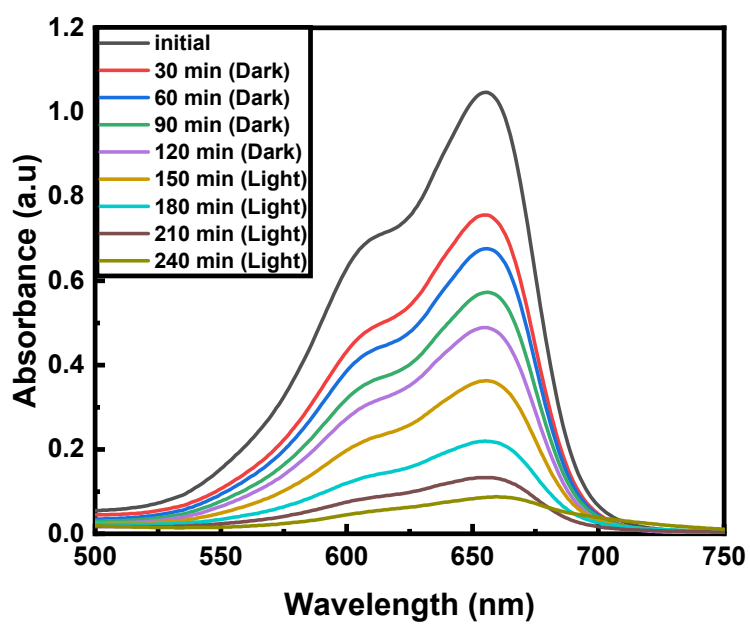


Figure S3: UV-Vis degradation profile of MB.

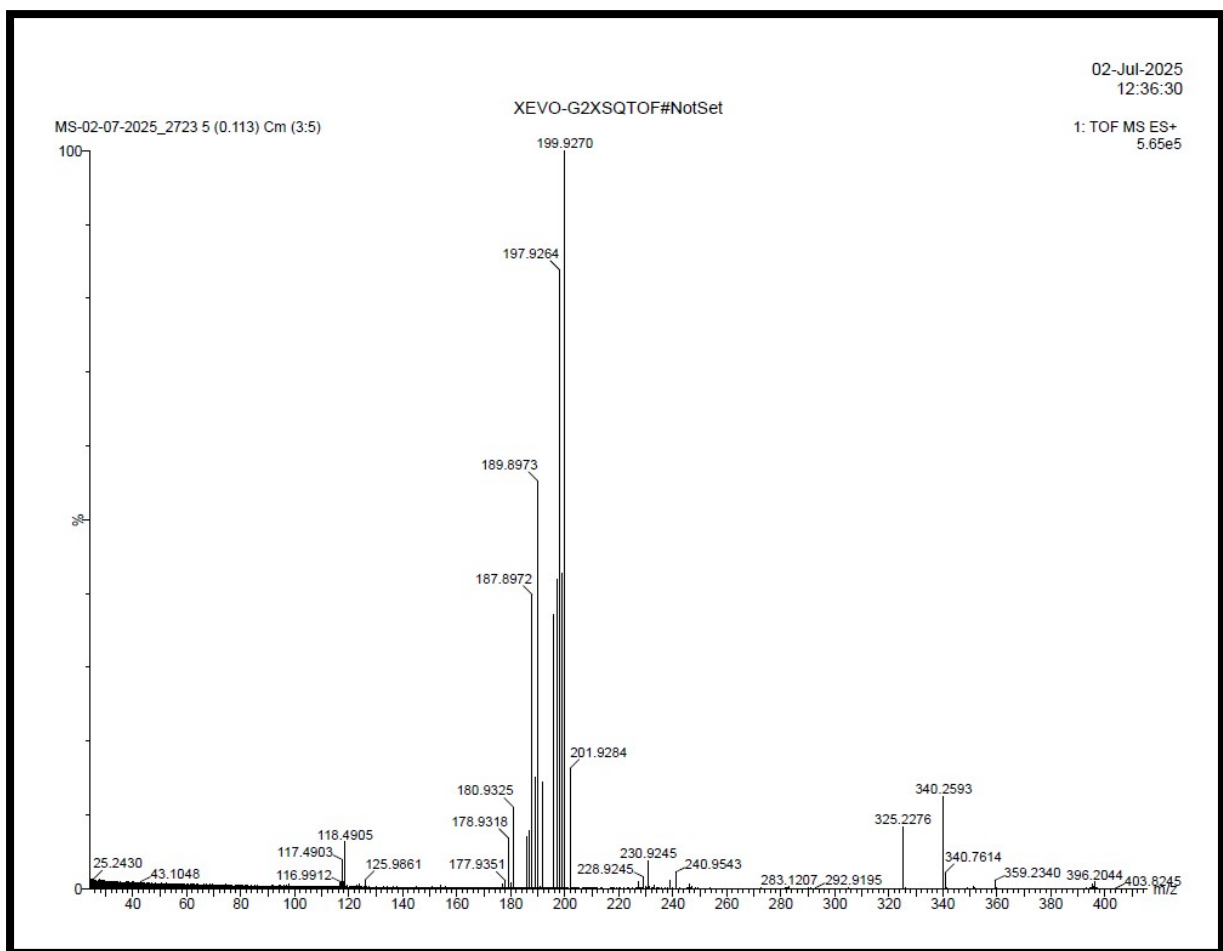


Figure S4: HRMS peaks for the degradation pathway of MB.

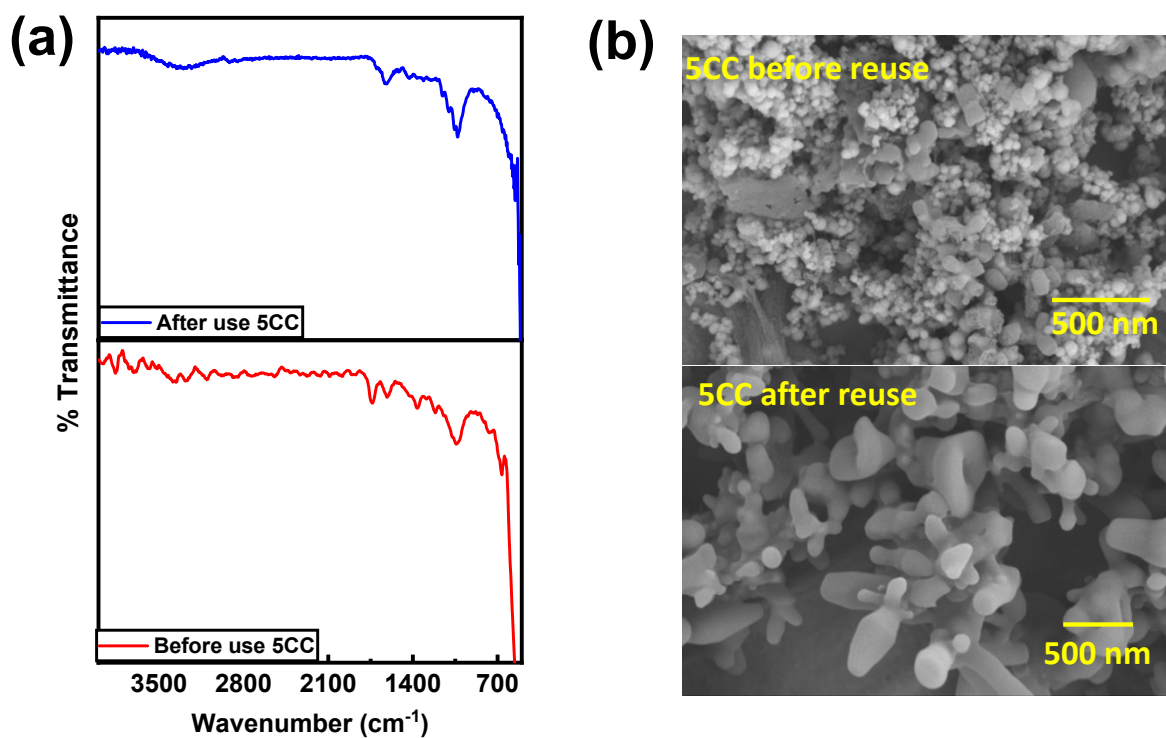


Figure S5: FT-IR spectrum and FESEM image of the recovered photocatalyst after photocatalytic degradation cycles demonstrating that the morphology remains largely unchanged.