

Electronic Supplementary material (ESI)

**NOVEL MOLYBDENUM OXIDE - STARBON CATALYST FOR WASTE-WATER
REMEDICATION**

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Characterization

FTIR data were obtained using PerkinElmer FTIR Spectrum 400 Analyser. High Resolution Scanning Electron Microscope (HR-SEM) images were taken using JEOL JSM-6500F with elemental mapping performed with hd-2700 instrument operated at an accelerating voltage of 200 kV and equipped with an Energy Dispersive X-ray Detector (EDX). High Resolution Transmission Electron Microscope (HR-TEM) images were taken using JEOL 1000 with an accelerating voltage of 200kV. JEOL 2010 TEM conventional (2Å) High Resolution Transmission Electron Microscope with CCD Camera. ICP-MS elemental analysis was conducted at Yara Laboratory in York (UK). Nitrogen adsorption-desorption isotherms were measured at -196°C using Micromeritics ASAP 2010 porosimeter, using N₂ as the probe adsorbate. Prior to analysis, samples were degassed under vacuum at 150°C for 6 h to vaporize physisorbed water. Specific surface areas and pore size distributions were determined by BET (Braunauer-Emmett-Teller) and BJH (Barrett-Joyner-Halenda) methods. Powder X-Ray diffraction (XRD) patterns were recorded using a PANalytical X'Pert Pro MPD diffractometer with CuK α radiation ($\lambda = 1.54056 \text{ \AA}$) in the 10°-45° 2 θ range. A Kratos Axis Ultra DLD system was used to collect XPS spectra using monochromatic Al K α X-ray source operating at 120 W (10 mA x 12 kV). Data was collected with pass energies of 160 eV for survey spectra, and 20 eV for the high-resolution scans with step sizes of 1 eV and 0.1 eV respectively. Data was analysed using CasaXPS (v2.3.20rev1.2H) after subtraction of a Shirley background and using modified Wagner sensitivity factors as supplied by the manufacturer. Simultaneous thermogravimetric analysis (STA) was conducted on NETZSCH STA 409. Samples were mounted in an alumina crucible (sample weight <15.0 mg) and heated at 10°C min⁻¹ to 600°C under flowing nitrogen gas (50 mL/min). EPR spectra were recorded on a Bruker EMX Micro EPR spectrometer (temperature 77 K, frequency 9.44 GHz, power 5 mW, modulation frequency 100 kHz, time constant 30 ms, number of scans 5, sweep width 7.5 mT).

Scanning Electron Microscopy (SEM)

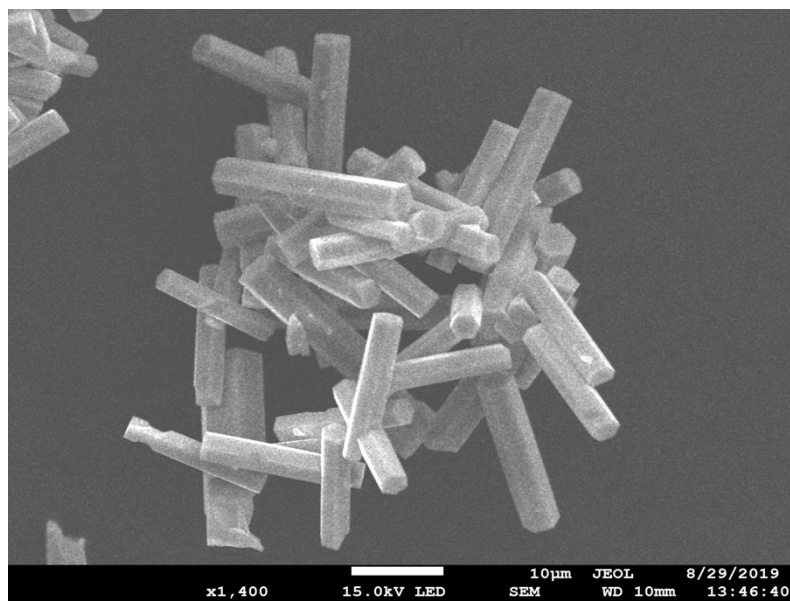


Figure S1: SEM image of MoO₃ nanorods prepared in the absence of starch.

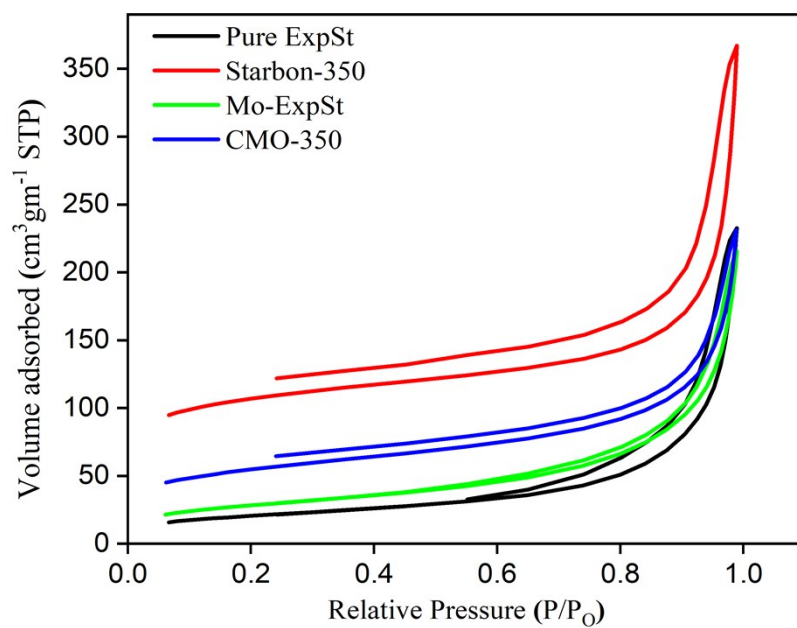


Figure S2: Adsorption-desorption Isotherm of Pure Expst, Starbon-350, Mo-Expst and CMO-350.

N₂ Adsorption Porosimetry

Differential Scanning Calorimetry

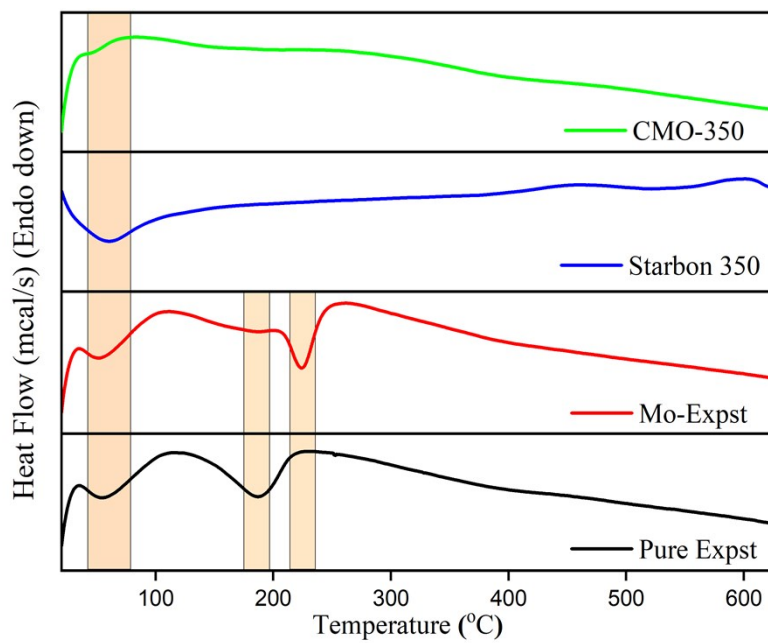


Figure S3: Comparative analysis of the DSC profile of synthesized composites.

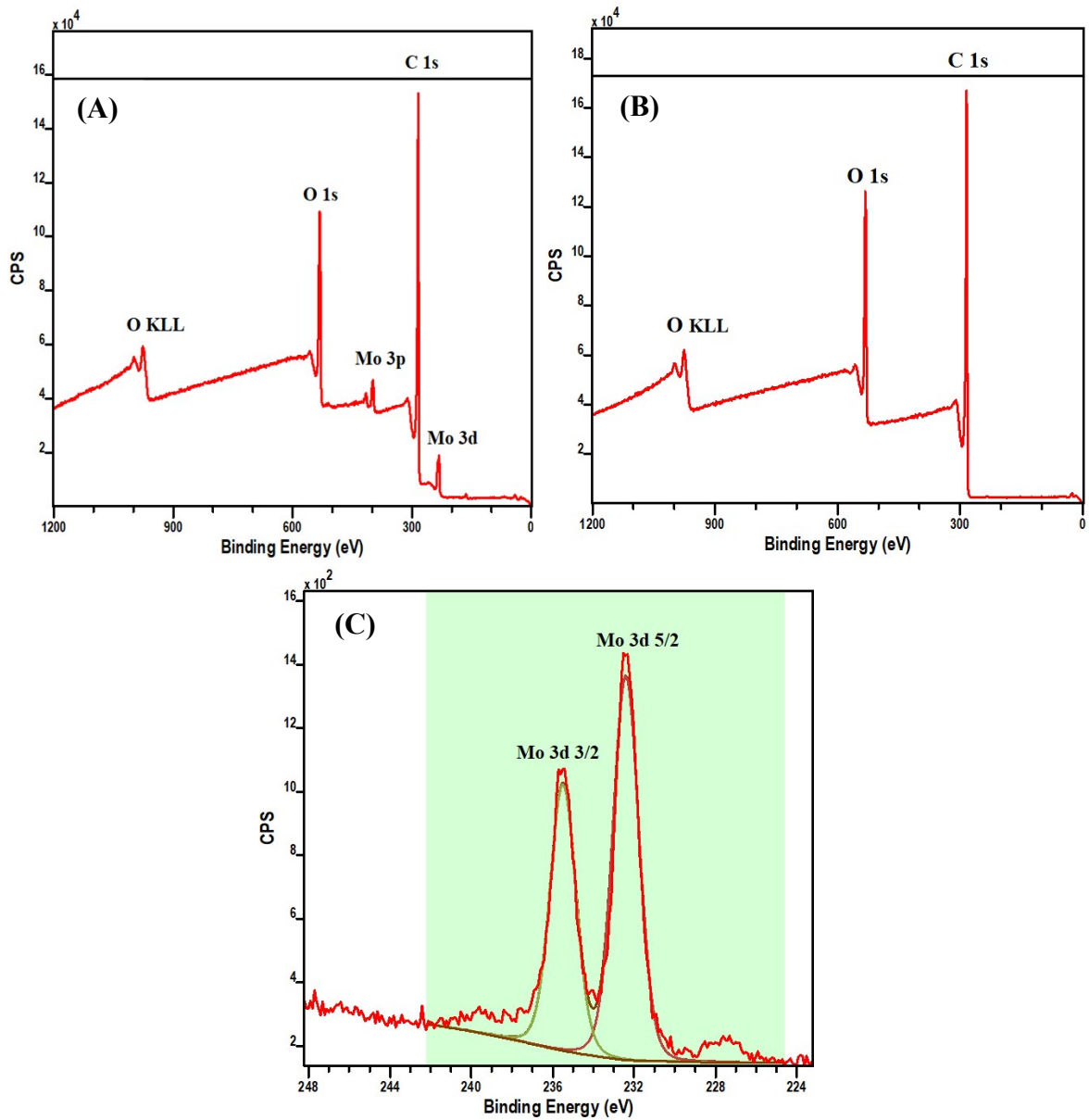


Figure S4: XPS Wide scan of (A) CMO-350 and (B) Starbon-350. (C) represents profile of Mo 3d/2 present in CMO-350

X-Ray Photoelectron Spectroscopy (XPS)

4-NP adsorption study

To study the adsorption of 4-NP in presence of CMO-350, no NaBH_4 was added to it (see Figure S5). The study was conducted at different pH conditions using NaOH and HCl to vary the conditions. At higher pH, the maxima shift from 320 to 400 nm which infer that there was abundance of 4-Nitrophenolate ion in the solution (see Figure S5 (C)). With the addition of the catalyst there was decrease in the maxima and corresponding increase in the absorption band at 320 nm.¹ The 4-Nitrophenolate ion was adsorbed on the surface of the porous matrix. Further, some of the MoO_3 Nanoparticles dispersed in the solution might lead to the formation of molybdic acid which leads to the decrease in the pH of the solution and subsequent formation of peak at 320 nm of 4-NP to maintain the equilibrium.²

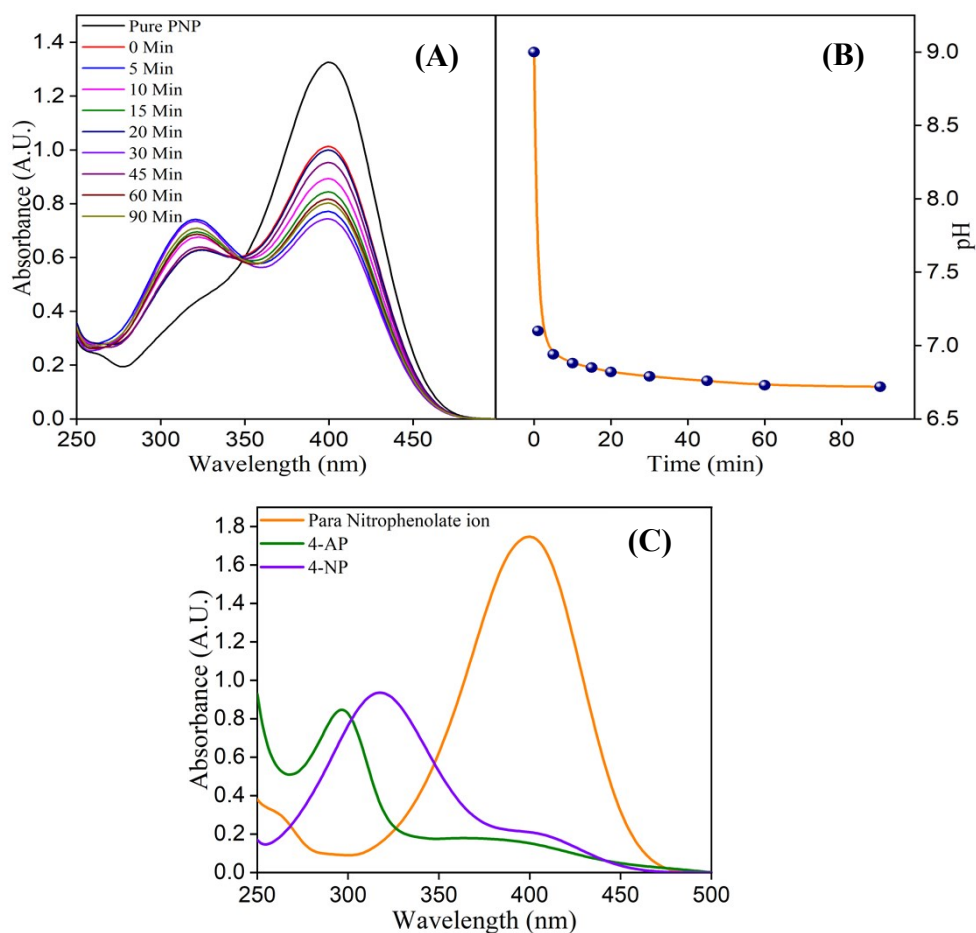


Figure S5: (A) Time study of the adsorption effect of 4-NP on CMO-350, (B) the corresponding change in pH, and (C) UV-Vis peaks of 4-NP, 4-Nitrophenolate ion and 4-AP.

DPPH radical quenching activity

DPPH (2,2-diphenyl-1-picryl-hydrazyl-hydrate) free radical method is an antioxidant assay based on electron-transfer that produces a violet solution in ethanol. This free radical is stable at room temperature but is reduced in the presence of an antioxidant molecule, giving rise to pale yellow solution.³

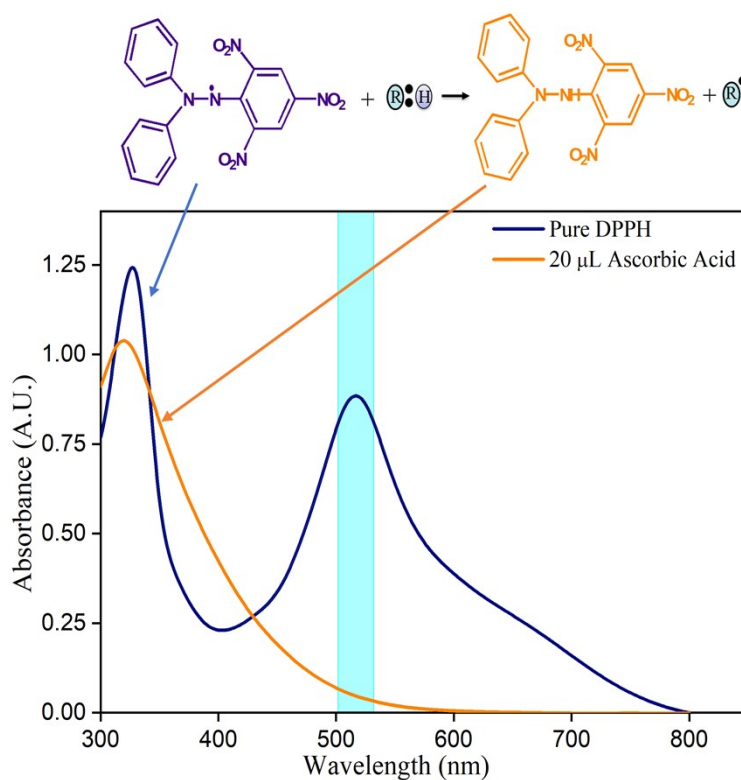


Figure S6: Schematic representation of DPPH radical quenching in presence of antioxidant and its effect on UV absorption band.

References:

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- 2 S. Shafaei, D. Van Opdenbosch, T. Fey, M. Koch, T. Kraus, J. P. Guggenbichler and C. Zollfrank, *Mater. Sci. Eng. C*, 2016, **58**, 1064–1070.
- 3 Y. Deligiannakis, G. A. Sotiriou and S. E. Pratsinis, *ACS Appl. Mater. Interfaces*, 2012, **4**, 6609–6617.