

Fe@g-C₃N₄: An effective photocatalyst for Baeyer-Villiger oxidation under visible light condition

Bharat A. Maru^a, Gaurang J. Bhatt^a, Urvi Lad^a, Pradeep T. Deota^a, Sanjeev Kane^b, U. K. Goutam^c, Chetan K. Modi^{*,a}

^a*Applied Chemistry Department, Faculty of Technology and Engineering, The Maharaja Sayajirao University of Baroda, Vadodara-390001*

^b*Synchrotrons Utilisation Section, Raja Ramanna Centre for Advanced Technology, Indore 452013, India*

^c*Technical Physics Division, Bhabha Atomic Research Centre, Mumbai-400085, India*

E-mail: chetanmodi-appchem@msubaroda.ac.in

Supporting Information

Materials

Urea (>99% pure) and anhydrous FeCl₃ were purchased from SD Fine-Chem Ltd., India. Barbituric acid was purchased from Chemdyes Corporation, India. Hydrogen peroxide (30%) AR was purchased from Loba Chemie Pvt. Ltd., India. Cyclohexanone (purity >99%) was purchased from Merck Ltd., India. A white LED light (12W) was purchased from a local electronic shop, Legrand, Vadodara, India.

Characterization techniques

For the characterization of the as-synthesized materials such as neat g-C₃N₄ and Fe@gC₃N₄ nanocatalysts with varying ratios viz., 1:1, 1:3, and 1:5, different physiochemical techniques were employed. The quantitative analysis of Fe metal ion in Fe@gC₃N₄ nanocatalysts was carried out by ICP-OES analysis (Make: Agilent Model: 5800 VDV) at the Central Salt and Marine Chemicals Research Institute (CSMCRI), Bhavnagar, India. FTIR spectra of as-prepared materials were performed in the range of 4000-500 cm⁻¹ on a model: Spectrum Two Perkin Elmer using KBr pellets. The structure determinations of as-synthesized materials were performed on Rigaku Smart Lab SE X-ray powder diffractometer with a CuKα (λ=1.54058) target and movable detector from the Applied Physics Department, The Maharaja Sayajirao University of Baroda, Vadodara, India. The XPS spectra were performed on a Specs Phoibios 225 spectrometer with Al Kα radiation (1486.6 eV) at the

Synchrotrons Utilisation Section, Raja Ramanna Centre for Advanced Technology, Indore, India. Transmission electron microscopy (TEM) of the as-synthesized materials was performed on Model: JEOL, JEM 2100 from the Centre Salt and Marine Chemicals Research Institute (CSMCRI), Bhavnagar, India. GC of obtained products were performed on model: Thermo, Trace-GC Ultra using DB-5 capillary column (30 m, 0.32 mm id, 0.25 μm film thickness. GC-MS of obtained products was performed on GCMSTQ8040, Shimadzu, QP-2010, from the Centre Salt and Marine Chemicals Research Institute (CSMCRI), Bhavnagar, India.

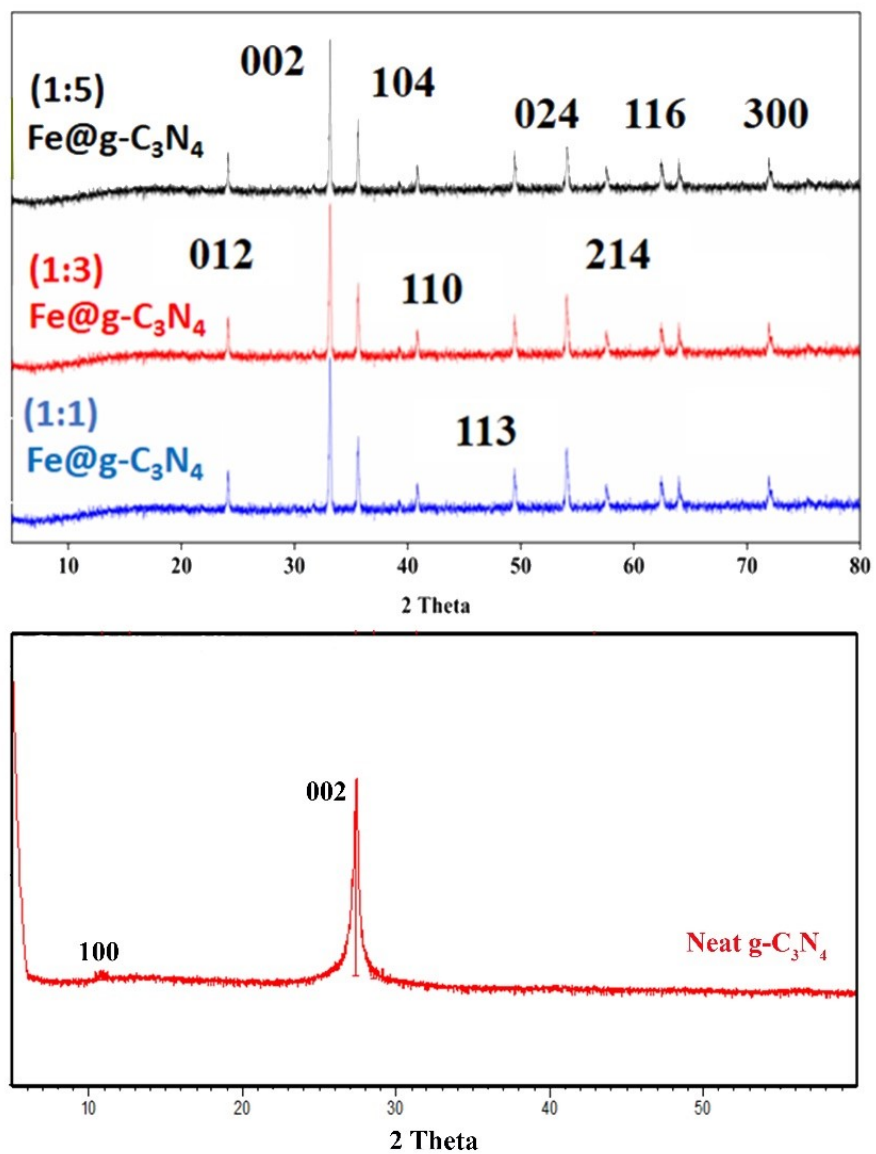


Fig. S1 XRD patterns of neat g-C₃N₄, Fe@g-C₃N₄ (1:1), Fe@g-C₃N₄ (1:3), Fe@g-C₃N₄ (1:5) catalysts

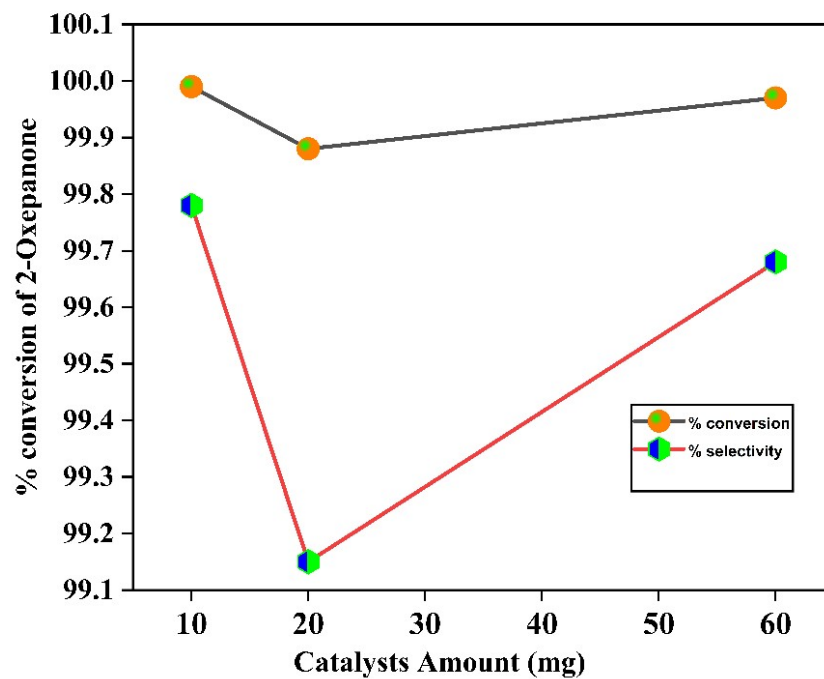


Fig. S2 The effect of catalyst dosage in the Baeyer-Villiger oxidation reaction

Reaction condition: cyclohexanone (8.29 mL, 0.08 mol), 30% H₂O₂ (1.88 mL, 0.08 mol), Fe@gC₃N₄ (1:1) X mg, Without solvent, temp. (Room temperature), time (1 h), under 12 W white LED

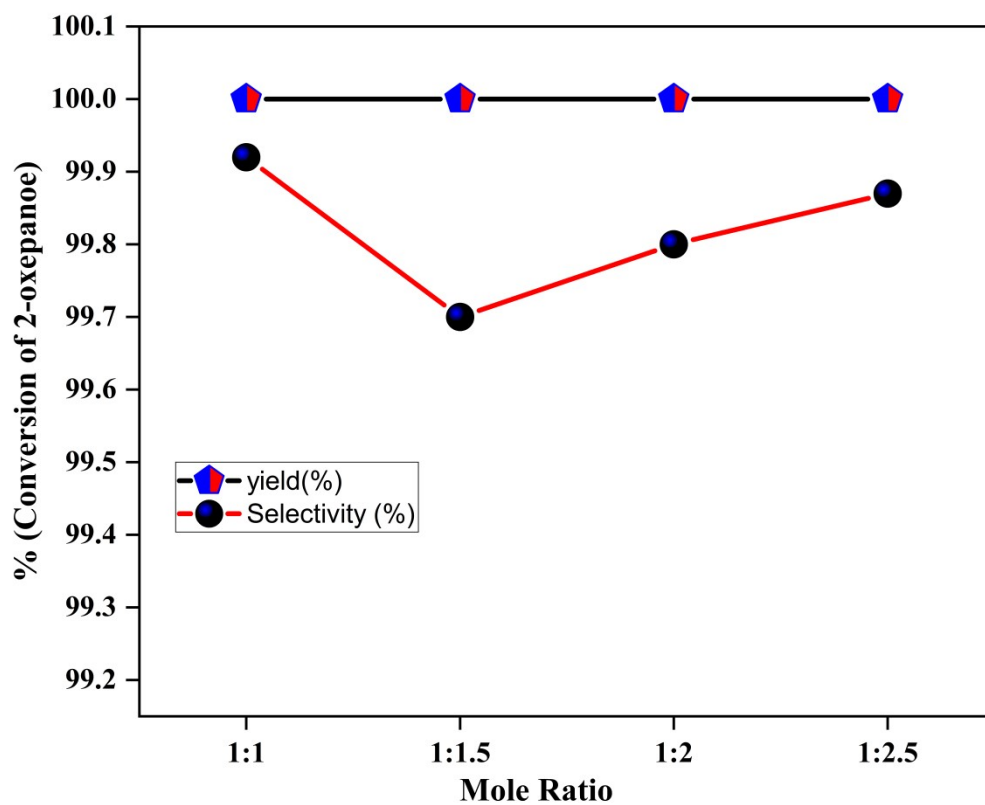


Fig. S3 The effect of mole ratio (cyclohexanone: H₂O₂) in the Baeyer-Villiger oxidation reaction

Reaction condition: cyclohexanone (8.29 mL, 0.08 mol), 30% H₂O₂ (X mol), Fe@gC₃N₄ (1:1) (10 mg, 0.05 mol% of Fe), without solvent, temp. (Room temperature), time (1 h), under 12 W white LED

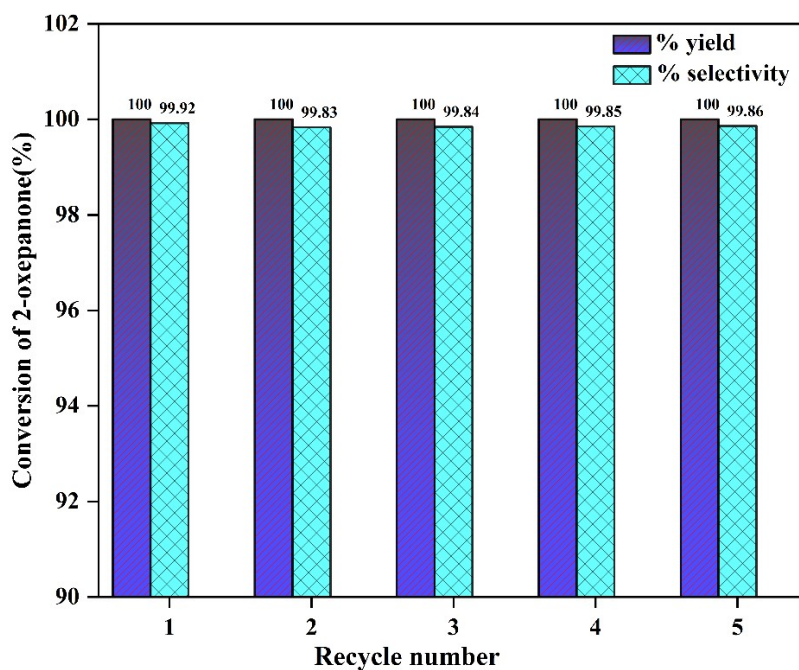


Fig. S4 Recyclability of Fe@gC₃N₄ (1:1) in the Baeyer-Villiger oxidation reaction

Reaction condition: cyclohexanone (8.29 mL, 0.08 mol), 30% H₂O₂ (1.88 mL, 0.08 mol), Fe@gC₃N₄ (1:1) (10 mg, 0.05 mol% of Fe), without solvent, temp. (Room temperature), time (1 h), under 12 W white LED

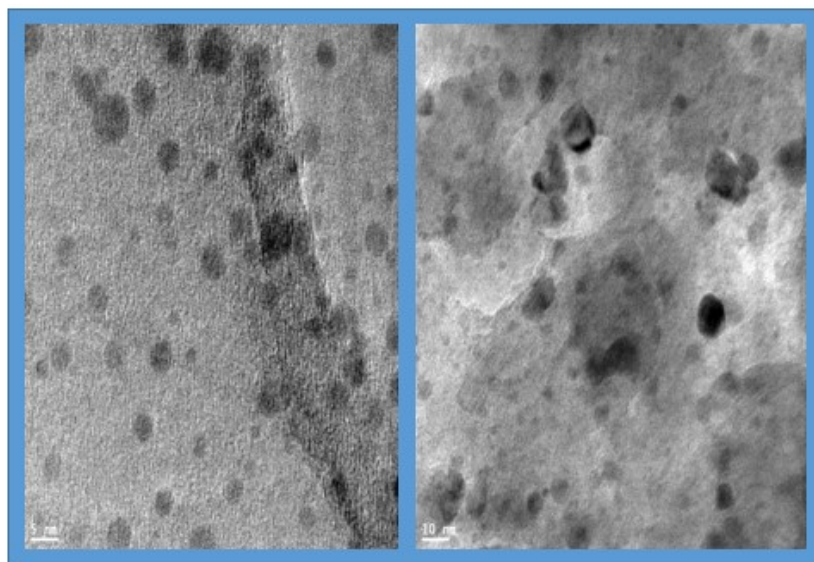


Fig. S5 TEM images of Fe@gC₃N₄ (1:1) catalyst (after 5th cycle)