Redox mediated synthesis of hierarchical Bi₂O₃/MnO₂ nanoflowers: A

non-enzymatic hydrogen peroxide electrochemical sensor

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S1. EXPERIMENTAL SECTION

Electron Microscopy (FESEM) is used with a (Supra 40, Carl Zeiss Pvt. Ltd.) microscope at an accelerating voltage of 20 kV. Transmission electron microscopic (TEM) analyses of the samples are performed on a FEI - TECNAI G2 20S – TWIN transmission electron microscope, operating at 200 kV and energy dispersive X-ray microanalyzer (OXFORD EDAX) has been attached to it for compositional analysis. The nitrogen gas adsorption and desorption study is performed using a Quantacrome autosorb iQ automated gas sorption analyzer. For adsorption study the sample was dried in vacuum for overnight and 20-25 mg amount was loaded in a 6 mm sample holder. In desorption study the sample was used for degassing at 70°C for 2 h and Brunauer–Emmett–Teller (BET) calculations are performed for the analysis of surface area of the sample. Phase purity of the synthesized samples are characterized by recording XRD on a BRUKER-AXS-D8-ADVANCE defractometer with Cu K α radiation ($\lambda = 1.5418$ Å) in the 2 θ range of 10°-80° at a scanning rate of 0.5° min⁻¹. X-ray photoelectron spectroscopy (XPS) analysis is done with a SPECS GmbH, Phoibos 100 MCD Energy Analyzer equipped with an Mg K α excitation source (1283.6 eV).



Fig. S1: FESEM images of (a) Bi₂O₃ and (b) MnO₂.



Fig. S2: Time dependent synthesis of Bi₂O₃/MnO₂ NFs at (a) 1 h, (b) 5 h, (c) 9 h and (d) 12 h.



Fig. S3: (a, c) Nitrogen adsorption and desorption isotherm and (b, d) pore size distribution plot of Bi_2O_3 and MnO_2 materials respectively.



Fig. S4: XRD pattern of as-synthesized Bi(0) nanoparticles.



Fig. S5: $KMnO_4$ concentration dependent synthesis of Bi_2O_3/MnO_2 NFs at (a) 0.05, (b) 0.1, (c) 0.3 and (d) 0.5 mmol.



Fig. S6: Comparative XRD pattern of as-synthesized various BM NFs composites and MnO₂.

Optical property: The optical adsorption of as-synthesized BM NFs is measured using UV-Vis Diffuse Reflectance Spectroscopy (DRS), as shown in Figure S7. All the as-prepared BM NFs samples show a spectral response in visible region, whereas Bi_2O_3 does not have any absorbance in

visible range. In case of BM nanocomposites the humps in visible range rise due to the synergistic effect of Bi_2O_3 photosensitizing and the creation of p-n heterojunction. With increase in Mn content (0.05 mmol to 0.3 mmol), the absorbance in visible region increases due to the enhancement in number of surface Bi_2O_3 nanoparticles. Further rise in Mn amount (0.5 mmol) results gradual decrease in absorbance. From this observation it can be concluded that with a certain amount of Mn (0.3 mmol) the composite shows maximum absorbance in visible region but further increment in Mn content cover up the optical activity of Bi_2O_3 and consequently absorbance in visible region decreases gradually.



Fig. S7: (a) UV-Vis diffuse reflection spectra of the as-prepared various Bi_2O_3/MnO_2 , bare Bi_2O_3 and MnO_2 samples and inset shows enlarged view of various Bi_2O_3/MnO_2 composites.



Fig. S8: Effect of (a) concentration of BM NFs in solution and (b) pH of electrolyte on the amperometric response of BM NFs/Nf/GCE to $5 \mu M H_2O_2$.