Chapter 2

Experimental

2. Experimental

2.1 Reagents

All the reagents used were of analytical reagent grade.

1. Hydrogen tetrachloroauratetrihydrate ($HAuCl_4.3H_2O$), silver nitrate ($AgNO_3$), sodium borohydride ($NaBH_4$), cetyltrimethylammonium bromide (CTAB), manganese acetate tetrahydrate [$Mn(CH_3COO)_2.4H_2O$], zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$], cadmium acetylacetonate [$Cd(acac)_2$], nickel chloride hexahydrate [$NiCl_2.6H_2O$], poly(*N*-vinyl-2-pyrrolidone) (PVP K30), phosphoric acid, ferric chloride hexahydrate ($FeCl_3{\cdot}6H_2O$), ammonium ferrous sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2{\cdot}6H_2O$], Histopaque 1077, 3-(4, 5-dimethylazol-2-yl)-2,5-diphenyl-tetrazolium bromide (MTT) were purchased from Sigma Aldrich and used as received. The molecular probes fluorescein isothiocyanate (FITC), alizarin red (AR), Evans blue (EB) were purchased from Sigma Aldrich and were used without further purification.
2. Poly(*N*-vinyl-2-pyrrolidone) (PVP K30, 40 kDa) (Wako Pure Chemicals, >99% India), PEG-13 (BroadPharm, San Diego, USA), nitroblue tetrazolium (NBT) (Sisco Research Laboratories, India), sulfanilamide (Himedia), *o*-phenylenediamine (Himedia), napthylethylenediamine dihydrochloride (S. D. Fine Chemicals, India) and ammonium hydroxide (S. D. Fine Chemicals, India) were used as received.
3. Methanol, n-heptane and *o*-xylene were purchased from Sisco Research Laboratories Pvt. Ltd., India and were used without further purification.
4. Double distilled water was used throughout the course of this investigation.

2.2 Instruments

1. Absorption spectra were measured in Shimadzu UV-vis 1610PC digital spectrophotometer (Shimadzu, Japan) and PerkinElmer Lambda 750 UV-vis-NIR spectrometer taking the sample in 1 cm well-stoppered quartz cuvette.
2. Diffuse reflectance spectra were measured in a PerkinElmer Lambda 750 UV-vis-NIR spectrometer taking the solid sample in a reflectance port in a 100 mm integrating sphere accessories and base line was corrected by using a reference reflectance port in the integrated sphere.
3. Fluorescence spectra were also recorded with a PerkinElmer LS-45 spectrofluorimeter equipped with a pulsed xenon lamp and a photomultiplier tube with R-928 spectral response. The spectrofluorimeter was linked to a personal computer and utilized the FL WinLab software package for data collection and processing.
4. Transmission electron microscopy (TEM) was carried out on a JEOL JEM 2100 microscope with a magnification of 200 kV. High resolution transmission electron micrograph (HRTEM), dark field scanning tunneling electron micrograph (DF-STEM) and selected area electron diffraction (SAED) pattern were obtained using the same instrument. Samples were prepared by placing a drop of solution on a carbon coated copper grid and allowing the grids to be dried overnight under vacuum.
5. Scanning electron microscopic images were recorded using JEOL **(**JSM-6360) instrument equipped with a field emission cathode with a lateral resolution of approximately 3 nm and acceleration voltage 3 kV.
6. Energy dispersive X-ray analysis was performed on a LEO 1530 field emission scanning electron microscope using X-ray detector.
7. Fourier transform infrared spectra were recorded in the form of pressed KBr pallets in the range (400-4000 cm^{-1}) with Shimadzu-FTIR Prestige-21 spectrophotometer.
8. The powder X-ray diffraction patterns were obtained using a D8 ADVANCE BROKERaxs X-ray Diffractometer with CuK_{α} radiation ($\lambda = 1.4506$ Å); data were collected at a scan rate of 0.5° min^{-1} in the range of 10º-80º.
9. The powder specific surface area was measured by BET analysis using a Micromeritics Tristar 3000 surface area analyzer.

10. Thermogravimetric analysis was carried out on a Perkin-Elmer STA 6000 with the sample amount of 10 mg. The measurements were performed under nitrogen with heating from 40-800 ºC (rate: 10 ºC min^{-1}) and then, maintained at 800 ºC for half an hour. Before TGA measurements, the samples were dried overnight in vacuum oven at 50 ºC.
11. Raman scattering measurements are carried out on silicon substrate in backscattering geometry using a fiber-coupled micro-Raman spectrometer equipped with 488 nm (2.55 eV) of 5 mW air cooled Ar^+ laser as the excitation light source, a spectrometer (model TRIAX550, JY), and a CCD detector.
12. Cyclic voltammetry measurements were performed by a CHI-660C electrochemical workstation. An Ag/AgCl electrode (in 3.0 M KCl) and a Pt wire were used as reference and auxiliary electrodes, respectively. Data have been collected using Pt electrodes as working as well as counter electrode and TOAB/0.1 M KCl solution as supporting electrolyte at temperature 298 K.
13. Photocatalytic reaction was carried out by a 60 Watt tungsten lamp (Institute of Electric Light Source, Beijing) that was positioned inside a cylindrical Pyrex vessel and surrounded by a recirculating water jacket (Pyrex) to cool the lamp.