

# **Chapter 2**

## **Experimental**

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## 2.1 Experimental

### 2.1.1 Reagents

All reagents used were of analytical reagent grade.

1. Hydrogen tetrachloroauratetrihydrate ( $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ ), silver nitrate ( $\text{AgNO}_3$ ), sodium borohydride ( $\text{NaBH}_4$ ), cetyltrimethylammonium bromide (CTAB), trisodium citrate, zinc acetate dihydrate ( $\text{Zn}(\text{OOCCH}_3)_2 \cdot 2\text{H}_2\text{O}$ ), potassium hydroxide (KOH), L(+)-ascorbic acid were purchased from Sigma-Aldrich and were used as received.
2. The molecular probes fluorescein isothiocyanate (FITC), sudan red 7B (SR7B), alizarin red (AR), Evans blue (EB), pyrene (PY), aminopyrene (APY) and aminomethylpyrene (AMPY) were purchased from Sigma Aldrich and were used without further purification.
3. Poly(*N*-vinyl-2-pyrrolidone) (PVP K30, 40 kDa) were purchased from Wako Pure Chemicals, >99% India)
4. Hydrazine hydrate ( $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ ) was purchased from Qualigens' Fine Chemicals, India (>98%) were used as received.
5. Methanol and *o*-xylene were purchased from Sisco Research Laboratories Pvt. Ltd., India and were used without further purification.
6. Double distilled water was used throughout the course of this investigation.

### 2.1.2 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. 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.

3. Time-resolved fluorescence spectra were carried out using the Photonic Technology Industry, Japan.
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) 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 dried overnight under vacuum.
5. Fourier transform infrared spectra were recorded in the form of pressed KBr pellets in the range (400-4000  $\text{cm}^{-1}$ ) with Shimadzu-FTIR Prestige-21 spectrophotometer.
6. 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.
7. 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.
8. Energy dispersive X-ray analysis was performed on a LEO 1530 field emission scanning electron microscope using X-ray detector.
9. The powder X-ray diffraction patterns were obtained using a D8 ADVANCE BROKERaxs X-ray Diffractometer with  $\text{CuK}_\alpha$  radiation ( $\lambda = 1.4506 \text{ \AA}$ ); data were collected at a scan rate of  $0.5^\circ \text{ min}^{-1}$  in the range of  $10^\circ$ - $80^\circ$ .
10. 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  $\text{Ar}^+$  laser as the excitation light source, a spectrometer (model TRIAX550, JY), and a CCD detector.
11. Laser heating was carried out with a continuous wave infrared diode laser (Model: ML-III-785-1 W, Changchun New Industries Optoelectronics Technology Co. Ltd., China) of wavelength 785 nm and output power 1 Watt by taking the sample in 1 cm quartz cuvette.

12. 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.
13. Theoretical calculations were performed using MATLAB 2011a<sup>®</sup>, FreeCAD<sup>®</sup>, Meshlab<sup>®</sup> and ImageJ software packages.
14. 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.