Chapter 2

Experimental

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2.1 Reagents

All the reagents used were of analytical reagent grade.

- Chloroauric acid ($HAuCl_4$, $3H_2O$), cetylpyridinium chloride (CPC), 11-mercaptoundecanoic acid (11-MUA), sodium borohydride ($NaBH_4$), zinc acetate dihydrate ($Zn(OOCCH_3)_2.2H_2O$), potassium hydroxide (KOH), ninhydrine, amino acids, L-Dopa, phosphoric acid, sodium perchlorate were purchased from Sigma Aldrich and were used as received.
- PEG-13 was purchased from Broad Pharm, (USA).
- Potassium carbonate (K_2CO_3) was purchased from Fluka and used without any further purification.
- Anhydrous $FeCl_3$, ammonium ferrous sulphate, and para-aminobenzoic acid (PABA), sodium nitrite, were purchased from Merck, (India) and were used without further purification.
- Chloroform, ammonia, potassium hydroxide were obtained from S. D Fine Chemicals, India.
- Hydrochloric acid obtained from Qualigens' Fine Chemicals was used as received.
- Toluene, methanol, ethanol, *n*-hexane, *n*-heptane, cyclohexane, *o*-xylene, chloroform, dichloromethane, benzene, carbon tetrachloride, cinnamic acid, potassium bicarbonate, sodium acetate, and acetic acid, were obtained from Sisco Research Laboratories, India and were used without further purification.
- Double distilled water was used throughout the course of the investigation.

All the experiments were performed at room temperature 25 ± 1 ºC.

2.2 Instruments

- Absorption spectra were measured with Shimadzu 1601 PC UV-vis spectrophotometer (Shimadzu, Japan) taking the sample in 1 cm quartz cuvette. Solid state UV-vis spectra were recorded by depositing the sample on a glass substrate and base line was corrected by using a similar glass substrate.
- For one set of experiments, Fluorescence spectra were measured with a PerkinElmer LS-45 spectrofluorimeter (PerkinElmer, UK) 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. For another set of experiments, Shimadzu RF-5301 spectrofluorimeter (Shimadzu, Japan) equipped with a 9.9 W xenon flash lamp and a photomultiplier tube with S-20 spectral response was used.

- The 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.
- The optical microscopic images of the membrane were observed with Olympus BX 61, equipped with a high resolution DP70 digital charge coupled device (CCD) camera and images were processed using Aldus Photostyler 3.0 software.
- Fluorescence microscopic images of the membrane were recorded in a Zeiss fluorescence microscope.
- Transmision electron microscopic measurements were performed with a Zeiss CEM 902 operated at 80 kV. The samples were prepared by dropping the gold sol and the membrane on carbon-coated copper grid and allowing the grid to be dried in air.
- 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.
- Energy dispersive X-ray analysis was performed on a LEO 1530 field emission scanning electron microscope using X-ray detector.
- Atomic force microscopic measurements were carried out on Digital Instruments Dimension 3100 microscope operated in tapping mode by taking as-prepared membrane on the silicon wafer.
- Electrochemical 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. Catalytic reactions were performed by the immobilized nanoparticles or nanocomposites over a 4-aminothiophenol monolayermodified gold working electrode where 0.1 M phosphate buffer saline (pH 7.5) was used as electrolyte and the scan rate was 100 mV s^{-1}.
- The powder X-ray diffraction patterns were obtained using a D8 ADVANCE BROKERaxs X-ray Diffractometer with CuK_{α} radiation (λ = 1.4506 Å); data were collected at a scan rate of 0.5° min^{-1} in the range of 10º-80º.

- 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 30-800 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.
- 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.
- Electrical conductivity was measured over the temperature range of 300 – 373 K by conventional multimeter.
- The NMR spectra were recorded in BRUKER ADVANCE II 400 FT-NMR spectrometer using $CDCl_3$ and DMSO (D_6) as solvent.