Background about the metal bimetallic nanoparticles, anisotropic nanostructures and their composites with carbon materials such as graphene and carbon nanotubes, their general properties, applications, strategies for the modification of electrodes, importance of biomolecules and drugs and a complete review on electrocatalytic applications of bimetallic NPs and their composites were discussed in the Chapter I.

Preparation of colloidal Au-Ag and Au-PtNPs and their fabrication on solid substrates, direct attachment of Au-Ag and Au-PtNPs on glassy carbon electrode, SWCNTs, MWCNTs and N-doped graphene and procedure for the growth of anisotropic AuNS and CuNS were given in Chapter II.

Synthesis of Au-Ag and Au-Pt bimetallic NPs with different Ag:Au and Pt:Au compositions in an aqueous medium and their attachment on GCE via 1,6-hexadiamine (HDA) linker for the reduction of HP and dioxygen and oxidation of methanol were described in Chapter III. The Au-AgNPs and Au-PtNPs were prepared by the galvanic displacement of Ag(0) and Pt(0) by AuCl$_4^-$ ions. The composition of the Au-AgNPs and Au-PtNPs was varied by varying the concentration of HAuCl$_4$ and monitored by UV-vis spectroscopy. HR-TEM images showed that the formed Au-AgNPs and Au-PtNPs were spherical in shape with a diameter of ~16 and ~20 nm, respectively. The Au-AgNPs and Au-PtNPs were then attached on GCE and the AFM images showed that they were densely packed on the electrode surface. Further, the electrocatalytic activity of Au-AgNPs
modified electrode was examined by studying the reduction of HP and NB and dioxygen reduction and methanol oxidation at Au-PtNPs modified electrode. The Au-AgNPs modified electrode showed the limit of detection of 0.12 and 0.23 µM ($S/N=3$) for HP and NB, respectively. The Au-PtNPs modified electrode exhibited better electrocatalytic activity towards methanol oxidation and oxygen reduction reactions when compared to their corresponding monometallic NPs.

Direct attachment of Au@Ag and Au@Pt core@shell NPs on GCE and their electrocatalytic activity were described in Chapter IV. It was achieved by catalytic deposition of AgNPs followed by galvanic displacement of AuNPs. The difference in the thermodynamic reduction potential between Ag(0) and Au$^{3+}$ leads to the reduction of Au$^{3+}$ ions by Ag(0) and hence the Au-AgNPs were deposited on GC electrode. The Au@PtNPs were fabricated on electrode surface by substrate catalyzed electroless deposition. The time dependent growth of Au-AgNPs and Au-PtNPs was monitored by SEM and the size of the NPs was increased with increase in deposition time. XPS results confirmed the zero valent nature of Ag, Au and Pt present on the Au-AgNPs and Au-PtNPs modified electrodes. The composition dependent electrocatalytic activity was realized at Au-AgNPs and Au-PtNPs modified electrodes towards the reduction of HP and it was found that the sensitivity of the electrode was found to be 865 and 1306 µA mM$^{-1}$ cm$^{-2}$ for Au-AgNPs and Au-PtNPs modified electrodes, respectively. The Au-PtNPs modified electrode was also exploited to the electrochemical reduction of
dioxygen and oxidation of hydrazine. The Au-PtNPs modified electrode exhibited superior electrocatalytic activity towards dioxygen and hydrazine than the bare and AuNPs modified electrodes.

A simple, facile and fast method for the growth of anisotropic AuNS including bipyramidal and wires on solid and solution phases using in-situ electrochemical reduction of Au$^+$ ions from the growth solution and electrochemical synthesis of CuNS on ITO substrate were demonstrated in Chapter V. The time dependent formation of different AuNS was monitored by HR-TEM and SEM and it was found that the spherical, bipyramidal and nanowires were grown on ITO substrate after 1, 3 and 6 h, respectively. The role of Cu$^{2+}$ ions with different counter ions on the morphology of AuNS was examined and it was found that Cu$^{2+}$ is an effective agent for controlled preparation of different shaped AuNS. The greater SERS enhancement was observed at the Au-nanowires grown substrate when compared to spherical and bipyramidal NS due to higher surface coverage. In the next part of this chapter, fabrication of surfactant-free cubic, spherical, dendritic and prickly CuNS on ITO surface by electrodeposition was described and investigated the role of applied potential, deposition time and pH on the morphology of dendritic CuNS. SEM images confirmed that cubic, spherical, dendritic and prickly CuNS were formed at the applied potentials of +0.10, -0.10, -0.30 and -0.50 V for 400 s, respectively in the presence of 10 mM CuSO$_4$ containing 0.1 M H$_2$SO$_4$. The catalytic activity of the different shaped CuNS
modified ITO was examined towards the oxidation of glucose and hydrazine. The dendritic CuNS modified electrode effectively catalyzed the oxidation of glucose and hydrazine by not only shifting their oxidation potential towards less positive potential and also enhanced their oxidation current when compared other NS.

In Chapter VI, fabrication of Au-PtNPs on both SWCNTs and FMWCNTs by electroless deposition and their electrocatalytic activity towards the oxidation of glucose and methanol and the reduction of HP were demonstrated. Initially, a jungle-gym structured film of SWCNTs was synthesized on the gold wire substrate by catalytic chemical vapor deposition and the fabrication of Au-PtNPs on SWCNTs was achieved by electroless deposition method. The successful deposition of Au-PtNPs on SWCNTs was confirmed by FE-SEM, HR-TEM, XPS, EDAX and CV. FE-SEM images showed that the deposited AuNPs on SWCNTs and PtNPs on SWCNTs/AuNPs were uniform and isotropic. FMWCNTs were fabricated on GC electrode by chemical attachment using OD and DCC. The Au-PtNPs were then attached on the FMWCNTs by substrate catalyzed electroless deposition. XPS confirmed the zero valent nature of both Au and Pt on both SWCNTs and MWCNTs. The homogeneously deposited thin layer of PtNPs over the SWCNTs/AuNPs (Au-20 and Pt-20 nm) electrode effectively catalyzed the glucose oxidation whereas a thick layer of Au-PtNPs (Au-20 and Pt-30 nm) on SWCNTs effectively catalyzed HP reduction when compared to SWCNTs and SWCNTs/AuNPs electrodes. The FMWCNTs/Au-PtNPs modified electrode was
exploited for methanol oxidation. It showed excellent long-term stability and superior electrocatalytic activity towards methanol oxidation than FMWCNTs and Au-PtNPs modified electrodes.

In Chapter VII, preparation of N-GO by intercalating melamine into GO and attach them on GCE via Michael’s reaction followed by its electrochemical reduction and electroless deposition of Au-PtNPs were demonstrated. The aromatic backbone of graphene was retained by the electrochemical reduction of oxygen functionalities of N-GO to form NG. Further, nitrogen doping via the intercalation of melamine with GO was confirmed by XPS. The present modified electrode is utilized towards the oxidation of RT and QR. The NG/Au-PtNPs modified electrode showed better electrocatalytic activity when compared to bare and NG electrode. Further, the comparison of the different bimetallic nanoparticles and their composites towards the reduction of HP was discussed. It was found that the electrocatalytic activity is not only depending on the composition of the bimetallic NPs but also the nature of the substrate in which they were immobilized.

Further, we have also compared the performance of the aforementioned electrodes towards a single analyte HP. It was found that the N-G/Au-PtNPs modified electrode effectively catalyzed the reduction of HP by showing its reduction potential at -0.08 V, which is more positive than the Au-PtNPs and SWCNTs/Au-PtNPs modified electrodes.