Chapter 7

Summary and Future Work

7.1 Summary

The nanomaterials have a very crucial role in the improvement of the performances of the sensors in terms of their catalytic activity. The thesis describes the synthesis of various functional nanomaterials like Molybdenum disulfide quantum dots (MoS_2QDs), Graphitic carbon nitride (g-C_3N_4), Molybdenum diselenide nanosheet ($MoSe_2$), and their composites with metal nanoparticles for the enhancement of their catalytic, electrochemical and optical properties with improved stability, like MoS_2-Qds stabilized silver nanoparticles, Gold nanorod decorated g-C_3N_4 nanosheet, gold nanoflower decorated $MoSe_2$ nanosheet, and synthesis of transition metals based metal-organic frameworks e.g. Cu-Fe Prussian blue analogue nanocube (Cu-Fe-PBA-NC). Further, the electrochemical, optical, and mimicking properties of the synthesized nanocomposites have been explored for designing the sensors of some important biological and chemical species like Dengue biomarker NS1, L-Cysteine, Cholesterol, and Isoniazid drug. This thesis is thoroughly organized and based on the synthesis of the metal nanomaterials, nanocomposites, and their application in sensing. The chapter-wise summary of the thesis is described below.

Chapter 1 deals with the general idea and basic concepts about sensors, components of sensors, sensors types, nanomaterials and their importance, its composites with metal nanomaterials for various sensing applications, nanozymes as a substitute for natural enzymes. The literature survey presents detailed information related to the proposed research topic.

Chapter 2 describes different experimental techniques which have been used for the characterization of developed materials. The main techniques which have been employed for characterizations are Scanning Electron Microscope (SEM), Transmission

Electron Microscope (TEM), X-ray Diffractometer (XRD), X-ray photoelectron spectroscopy (XPS), and Fourier Transform Infrared Spectroscopy (FTIR) used for the morphological and structural investigation. UV-Visible spectrometer has been used for spectroscopic characterizations and optical sensing of different analytes. Cyclic voltammetry (CV) Differential cyclic voltammetry (DPV) and Impedimetric technique (EIS) setup has been used for electrochemical characterization.

Chapter 3 describes a high-performance NS1 immunosensor based on gold nanorod decorated graphitic carbon nitride (AuNRs-g-C_3N_4) modified glassy carbon electrodes (GCE). AuNRs-g-C_3N_4 showed excellent electro-activity, fast electron transfer kinetics, and high catalytic property. NS1 antibody is immobilized onto the surface of the AuNRs-g-C_3N_4 modified GCE and used as an impedimetric sensing probe for the quantitative sensing of NS1 antigen through electrochemical impedance spectroscopy. $[Fe(CN)_6]^{3-/4-}$ was chosen as a redox couple to observe the respective changes in charge transfer resistance (R_{ct}) associated with antigen bonding with an antibody both in PBS buffer as well as in human serum. The constructed biosensor showed a linear response from 0.6 ng/mL to 216 ng/mL in PBS buffer, whereas in human serum, it shows a linear calibration range from 3 ng/mL to 177 ng/mL. The limit of detection was found to be 0.09 ng mL^{-1} and 0.03 ng mL^{-1} in PBS and human serum, respectively. The analytical parameters obtained by using this sensor were found to be superior and comparable to the earlier. The study indicates that the prepared immunosensor can act as a potential impedimetric technique for the point of care diagnosis for the dengue virus in biological samples.

Chapter 4 deals with an electrochemical biosensor assembled with gold nanoflower decorated molybdenum diselenide nanosheet (AuMoSe$_2$) and cholesterol oxidase enzyme for the detection of free cholesterol through Differential pulse voltammetry

(DPV) and electrochemical impedance spectroscopy (EIS). we have successfully synthesized and characterized the $MoSe_2$ nanosheet. Further, in situ surface modification of $MoSe_2$ with gold nanoflower was achieved at room temperature without any use of a reducing agent. The $AuMoSe_2$ shows excellent electrochemical properties and has been utilized to fabricate cholesterol biosensors by immobilizing ChOx. The developed biosensor has been used to detect cholesterol in PBS buffer and human serum using DPV and EIS techniques. a detection limit of 3.2 μM in DPV, and 37 μM in EIS technique respectively. The present study shows that the fabricated can be used as a potential electrochemical technique for the point-of-care diagnosis of free cholesterol in biological samples.

Chapter 5 deals with the hydrothermal synthesis of MoS_2 quantum dots and its application for the formation and stabilization of a nanocomposite with silver nanoparticles (AgNPs) in a single step. we have successfully demonstrated a highly efficient and sensitive colorimetric sensing platform for the detection of Cys in real samples (blood serum), utilizing robust oxidase mimetic activity of MoS_2-QDs stabilized AgNPs stimulated by Hg^{2+}. The developed sensor for Cys detection exhibited a wide dynamic range of 1-100 μM with a lower detection limit of 824 nM. The most astonishing feature of this work is the quick ultra-trace level detection of Cys aided with cost-effective plastic tubes based kit. This approach utilizing the synergistic effect of MoS_2-QDs-AgNPs and Hg^{2+} towards enhancement in catalytic efficiency of the system may prove to be economically beneficial for quick and easy detection of Cys. The presented assay is an efficient, simple, cost-effective, sensitive, and applicative within the clinical health range for accurate identification of Cys.

Chapter 6. deals with a colorimetric technique for the easy quantification of the Isoniazid (INH) in human urine. we have demonstrated the successful synthesis of CuFe-PBA-NC using a simple precipitation method and well-characterized by different techniques like IR, XRD, SEM, and XPS. The nanocubes of average size between 400-500 nm were found to be possessing high oxidase activity to TMB. We have efficiently utilized CuFe-PBA-NC to develop a selective and sensitive colorimetric strategy for sensing the antituberculosis drug INH. The developed colorimetric sensor shows a wide range detection of INH in both acetate buffer and urine sample (1-100 μM) with LOD of 0.44 μM, and 0.77 μM, respectively. We have also developed a portable Eppendorf kit which can be very helpful in the naked eye ultra-trace level onsite detection of INH. This technique shows a rapid, simple, and economical procedure with trace-level selective detection of INH in the human urine.

7.2. Future Work

In the thesis, we have developed nanocomposites with improved electrochemical properties, and optical properties. We have also tried to resolve the issue regarding the stability of the nanoparticles by functionalizing their surface. In the case of nanozymes, we have demonstrated the synthesis of nanoparticles with intrinsic enzyme mimicking properties and also investigated induced mimicking properties which can be achieved through surface engineering. We have demonstrated colorimetric and electrochemical sensors based on nanocomposites for disease identification, drug monitoring, and regular health check-up. Nowadays, the common man's life is greatly threatened by heart and kidney diseases, therefore, our focus will be to assemble some new nanoparticles modified sensors that are simple, economical, portable, efficient, and reliable for point-of-care detection of biomarkers. We will also explore our research

towards the development of FETs-based sensors or the identification of diseases by functionalization of nanoparticles and molecular engineering of some conducting polymers.