Preface

In the present scenario, most of the countries worldwide are facing the burden of various communicable and non-communicable diseases. However, developing countries like India are more vulnerable and more exposed because of multiple factors including demographic, socioeconomic, and geographic factors.

Various lethal diseases are prevalent across the world like Cancer, HIV infection, Malaria, TB, etc. which are responsible for high mortality rates. Several therapeutic drugs are used for the treatment and cure of these diseases. These drugs function effectively and positively when they are present in the human body within a particular concentration window. The amount of drug present in the body is essential to be determined because if the concentration of the drug would be lesser than the appropriate amount, the cure of the disease would be difficult and if the concentration of the drug is higher than the desired amount (due to the drug overdose), it would prove to be fatal and severe adverse symptoms could be observed, and it may lead to death in severe cases. So, there is a need for a cheap, sensitive, and accurate sensor so that the concentration of the drug and its by-products can be quantitatively estimated in the patient's blood on a real-time basis.

A sensor is a device that detects a change in the environment or change on account of the interaction of the analyte and thereby converts it into a readable output signal. We are surrounded by a large number of sensors in our daily life, and they can broadly be categorized into chemical, physical, and biosensors. A physical sensor transforms a physical quantity like temperature, pressure, mass, etc. into a signal**.** A chemical sensor detects a chemical substance by chemical or physical responses and transforms it into a signal. Similarly, a biosensor detects chemical substance by using a biological sensing element.

Basically, a sensor is composed of two important components *viz.* an active layer or the recognition and a transducer. Herein, the active layer (or the recognition layer) represents the heart of a sensor, while the transducer converts the change in the environment into an observable signal such as optical, electrochemical, electrical, etc. On the basis of the method of transduction, the sensor can be categorized into physical, chemical, and electrochemical sensors. In this thesis, we have focused on electrochemical sensors since they are associated with a large number of advantages such as excellent sensitivity, high selectivity, great reproducibility, high stability and portability, and they are easy to operate and economical. In electrochemical sensing, the electrode surface acts as the active layer of the sensor. Efforts are made to synthesize such materials for electrode modification that enhance the efficacy of an electrochemical sensor. These electrode materials include a variety of materials such as polymeric materials, composites, metal complexes, nanomaterials, etc. Among them, nanomaterials present themselves as one of the best choices nowadays because they offer high sensitivity due to great surface-to-volume ratio, rapid electrode kinetics, good catalytic activity, easy functionalization, excellent biocompatibility, etc. On the basis of confinement of directional movement of electrons, nanomaterials are mainly categorized into zero, one, two and three-dimensional nanomaterials. Among these, 2D nanomaterials have regarded as a large number of applications in diverse fields due to their intriguing mechanical, electrical, and thermal properties. However, it possessed tendency to aggregate which reduces their efficacy due to a decrease in the active surface area and the number of active sites. The problem of aggregation can be overcome by a composite formation with other nanomaterials such as metal nanoparticles, metal oxide nanoparticles, polymers, other 2D materials, quantum dots, etc. resulting in the enhancement in the desired properties of these materials.

The main focus of the thesis is to synthesize and characterize the composites of 2D nanomaterials with transition metal dichalcogenides quantum dots (WS_2 QD), conducting polymer (5-carboxypolyindole), and metal nanostructure (gold nanorods). Thereafter, employing the as-synthesized nanocomposites for the modification of commercially available glassy carbon electrodes for voltammetric sensing of various therapeutic drugs like Primaquine, Chloroquine administered to patients suffering from malarial disease, and an immunosuppressive drug, Azathioprine. Thus, on these views, the therapeutic drugs are selected as target analytes in our case of research work, and voltammetric sensors are designed for the trace-level detection of these drugs. Some antimalarial drugs and an anti-inflammatory drug are chosen as target analytes, prolonged exposure of which can cause severe adverse effects on the health and may also prove to be lethal. All work in the thesis has been fractionated into six chapters as following manner:

Chapter 1 pertains to the general terminologies related to the research works like concepts of sensors, categories of sensors, components of sensors, electrochemical sensors and their superiority over other sensors, the importance of nanomaterials and their advantages, transition metal dichalcogenides and their composites for electrochemical sensing applications, the need to produce the sensor for drugs related to harmful diseases and abnormalities. At last, it also includes the literature survey based on the proposed research topic

Chapter 2 illustrates the various experimental techniques utilized for characterizing the synthesized materials. The major techniques that have been employed for the characterizations include Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Energy Dispersive Spectroscopy (EDX), X-ray photoelectron spectroscopy (XPS), and Fourier Transform Infrared Spectroscopy (FTIR) for the structural and morphological investigations. UV-Visible spectroscopy (UV-vis) has been used employed for spectroscopic characterizations. Cyclic Voltammetry (CV), Differential Pulse Voltammetry (DPV), and Electrochemical Impedance Spectroscopy (EIS) have been utilized for electrochemical characterization and sensing of the analytes.

Chapter 3 presents the development of trace level sensor for an antimalarial drug- Chloroquine (CQ) on the tungsten disulfide quantum dots decorated reduced graphene oxide sheets (rGO@WS_2 QDs) modified glassy carbon electrode (GCE) surface. Prior to the trace analysis, this chapter explains about the synthesis of rGO@WS_2 QDs composite and its characterization by UV-Vis., TEM, SEM, EDS, and FTIR. At last, this conducting matrix has shown for excellent electroactivity, high catalytic property, and rapid electron transfer kinetics. CV and DPV were employed for the quantitative determination of CQ in phosphate buffer, human blood serum, and commercially available CQ tablets. The obtained analytical parameters are found to be comparable to or better than previously reported CQ sensors. The proposed sensor is very much sensitive, stable, reproducible, and selective for CQ detection and with great potential for real-time CQ sensing in malarial patients.

Chapter 4 presents an electrochemical sensor based on molybdenum disulfide nanosheets modified with poly(5-carboxyindole) (MoS_2-CPIn) for the trace-level detection of an anti-inflammatory drug Azathioprine (Azp) through DPV. MoS_2 nanosheet has been synthesized through hydrothermal synthesis and characterized by XRD, TEM, SEM, EDS, and FTIR. Further, CPIn chains were grown over the MoS_2 nanosheets to obtain the MoS_2-CPIn composite. Azp was determined in phosphate buffer as well as human blood serum at room temperature. The developed sensor demonstrates an extensively wide linear range and a low limit of detection. The developed sensor shows excellent stability, sensitivity, and good selectivity at physiological pH. The achieved analytical parameters are found to be comparable to or better than previously reported CQ sensors.

Chapter 5 presents an electrochemical sensor based on gold nanorods decorated molybdenum disulfide nanosheets (MoS_2-GNR) for the quantitative estimation of an anti-malarial drug Primaquine (PQ) through DPV. GNR was synthesized through a seed-mediated growth methodology and characterized by XRD, TEM, EDS and FTIR. Further, GNR were decorated over the MoS_2 nanosheets to obtain the MoS_2-GNR composite which possesses excellent electroactivity, high catalytic properties, and rapid electron transfer kinetics. PQ sensing was validated in phosphate buffer, human blood serum, and commercially available PQ tablet. The proposed electrochemical sensor shows a wide concentration range and a low limit of detection and offers a fascinating sensitivity, selectivity, stability, and reproducibility at physiological pH conditions with great potential to be used as a detection tool in diagnostic centers. The analytical parameters obtained are found to be comparable to or better than previously reported PQ sensors.

Chapter 6 includes the conclusive remarks and future prospects of the thesis.