SUMMARY OF THE THESIS

The main theme of this thesis is to study the versatility of carbon paste electrode in terms of application to investigate the electrochemical behavior of different organic compounds of biological relevance and elucidation of the sequence of the electron transfer that occur at or near the electrode surface by cyclic voltammetry.

The organic compounds of biological relevance chosen in the present study were dopamine, amlodipine besylate, ascorbic acid, mesoionic 3-aryl-4-bromo sydnone, and its derivatives.

Organization of the Thesis

The work carried out on the above topic is described in seven chapters.

Chapter 1

Introduction, Review of Cyclic Voltammetry and Theoretical Considerations

The first chapter carries the introduction, basics and a brief historical preview, present trend, transfer of electro-chemical phenomena into technological devices and process, applications, theory and theoretical considerations of cyclic voltammetry. A brief review of cyclic voltammetric investigations of certain organic compounds has been presented. Objective and scope of the present work is included in this chapter.

Chapter 2

Experimental

This chapter describes the instrumentation, devices, the electrode systems with special emphasis on carbon paste electrode, experimental techniques and procedures used in the present work, modified and unmodified carbon paste electrode. Some practical aspects of preparation of unmodified carbon paste electrode have been discussed. This chapter also includes details about the basic equipment needed for electrochemistry such as potentiostat, a recording device, and an electrochemical cell
Chapter 3 A

Cyclic Voltammetric Studies of Potassium Ferricyanide and Dopamine Using Triton X-100 at Carbon Paste and Ceresin Wax Carbon Paste Electrode

Triton X-100 \([C_{14}H_{22}O(C_2H_4O)n]\) is a nonionic surfactant, which has a hydrophilic polyethylene oxide group (on average it has 9.5 ethylene oxide units) and a hydrocarbon lipophilic or hydrophobic group. Triton X-100 is used as a detergent and it is considered 100% active and biodegradable in liquid form. It has numerous general uses as a wetting agent, emulsifier or even as a mild detergent.

Dopamine is a chemical naturally produced in the body. In the brain, dopamine functions as a neurotransmitter, activating dopamine receptors. Dopamine is also a neurohormone released by the hypothalamus. Its main function as a hormone is to inhibit the release of prolactin from the anterior lobe of the pituitary. Dopamine can be supplied as a medication that acts on the sympathetic nervous system, producing effects such as increased heart rate and blood pressure.

The focus in this chapter is on the electrochemical response of potassium ferricyanide and dopamine at carbon paste and ceresin wax carbon paste electrode in 1M KCl medium. The process was carried out by introducing Triton X-100 in two ways i.e. onto the surface (immobilization) and into the solution (mobilization) for potassium ferricyanide and dopamine. The increase in the current signal was observed only in the case of immobilization method. This is because, the Triton X-100 molecules will diffuse along with potassium ferricyanide and dopamine. Where as in the case of mobilization method the increase in the current signal was observed only in the case of dopamine but not for potassium ferricyanide.
Chapter 3 B

Cyclic Voltammetric Studies of Potassium Ferricyanide and Dopamine Using Cetyl Trimethyl Ammonium Bromide at Carbon Paste and Ceresin Wax Carbon Paste Electrode

Cetyl trimethyl ammonium bromide \[\text{[(C}_{16}\text{H}_{33})\text{N(CH}_3\text{)}_3\text{Br}\] is one of the components of the topical antiseptic cetrimide. The Cetyl trimethyl ammonium bromide cation is an effective antiseptic agent against bacteria and fungi. Its uses include providing a buffer medium for the extraction of DNA. It is also widely used in hair conditioning products.

The presentation in this chapter is on the electrochemical response of potassium ferricyanide and dopamine at carbon paste and ceresin wax carbon paste electrode in 1M KCl medium. Here we carried out our work by introducing CTAB in two ways i.e. onto the surface (immobilization) and into the solution (mobilization) for potassium ferricyanide and dopamine. The increase in the current signal was observed only in the case of immobilization method. This is because, the CTAB molecules will diffuse along with potassium ferricyanide and dopamine. Where as in the case of mobilization method the increase in the current signal was observed only in the case of dopamine but not for potassium ferricyanide.

Chapter 4

Electrocatalytic Oxidation of Amlodipine besylate with Phenylhydrazine as a Mediator at Carbon Paste Electrode

Amlodipine besylate is chemically described as 3-ethyl-5-methyl (±)-2-[(2-aminoethoxy) methyl]-4-(2-chlorophenyl)-1,4-dihydro-6-methyl-3,5pyridindicarboxylate, monobenzene sulphonate. Its emperical formula is \(\text{C}_{20}\text{H}_{25}\text{Cl N}_2\text{O}_5 \text{ C}_6\text{H}_6\text{O}_3\text{S}\). Amlodipine besylate is a white crystalline powder with a molecular weight of 567.1. It is slightly soluble in water and sparingly soluble in ethanol. Amlodipine is a dihydropyridine calcium antagonist (calcium ion antagonist or slow channel blocker) that
inhibits the transmembrane influx of calcium ions vascular smooth muscle and cardiac muscle.

This chapter describes the electrochemical oxidation pathway of Amlodipine besylate at carbon paste electrode by using phenylhydrazine as a mediator in pyrophosphate buffer medium. The dihydropyridine moiety undergoes two electron oxidation to give pyridine derivative. The effect of varying pH, sweep rate, concentration of the electroactive species, concentration of phenylhydrazine, effect of CTAB and SDS surfactants have been discussed. The increase in the current signal was observed in the case of phenylhydrazine mediated carbon paste electrode compared to carbon paste electrode, showing that phenyl hydrazine acts as a good mediator catalyst for the oxidation various electroactive species.

**Chapter 5**

**Electrochemical Responses of Potassium Ferricyanide and Dopamine Separately by Sodium Dodecyl Sulfate Modified Carbon Paste Electrode**

Sodium dodecyl sulfate (C_{12}H_{25}SO_{4}Na) is an anionic surfactant that is used in household products such as toothpastes, shampoos, shaving foams, and bubble baths for its thickening effect and its ability to create a lather. The molecule has a tail of 12 carbon atoms, attached to a sulfate group, giving the molecule the amphiphilic properties required of a detergent.

Dopamine is a chemical naturally produced in the body. In the brain, dopamine functions as a neurotransmitter, activating dopamine receptors. Dopamine is also a neurohormone released by the hypothalamus. Its main function as a hormone is to inhibit the release of prolactin from the anterior lobe of the pituitary. Dopamine can be supplied as a medication that acts on the sympathetic nervous system, producing effects such as increased heart rate and blood pressure. However, since dopamine cannot cross the blood-brain barrier, dopamine given as a drug does not directly affect the central nervous system. To increase the amount of dopamine in the brains of patients with diseases such
as Parkinson's disease and Dopa-Responsive Dystonia, a synthetic precursor to dopamine such as L-DOPA (levodopa) can be given, since this will cross the blood-brain barrier. Dopamine has many functions in the brain. Dopamine affects the basal ganglia motor loop which in turn affects the way the brain controls our movements. Shortage of dopamine, particularly the death of dopamine neurons in the nigrostriatal pathway, causes Parkinson's disease, in which a person loses the ability to execute smooth, controlled movements.

The focus of the work in this chapter is on the electrochemical response of each potassium ferricyanide and dopamine at bare carbon paste and SDS modified carbon paste electrode in 1M KCl supporting electrolyte medium. The effect of varying sweep rate, concentration of electroactive species, and concentration of SDS on peak current and peak potential have been discussed. The peak potential difference each of potassium ferricyanide and dopamine at bare carbon paste electrode are 77 and 129mV and at SDS modified carbon paste electrode are 34 and 63mV respectively. The SDS modified carbon paste electrode shows enhancement in the current signals for potassium ferricyanide and dopamine. The concentration of SDS shows increase for 4 mg for potassium ferricyanide and 6 mg for dopamine. The application of this electrode creates a new approach to determine the importance neurotransmitter dopamine sensitivity.

Chapter 6

Electrocatalytic Oxidation of Ascorbic Acid and Dopamine with Phenylhydrazine as a Mediator at Carbon Paste Electrode

Dopamine has the chemical formula C₆H₃(OH)₂-CH₂-CH₂-NH₂. Its chemical name is 4-(2-aminoethyl) benzene-1,2-diol. Dopamine is a hormone and neurotransmitter occurring in a wide variety of animals, including both vertebrates and invertebrates. In chemical structure, it is a phenylamine. In the brain, dopamine functions as a neurotransmitter, activating the five types of dopamine receptors D₁, D₂, D₃, D₄, and
Summary of the thesis

D5, and their variants. Dopamine is produced in several areas of the brain, including the substantia nigra. Dopamine is also a neurohormone released by the hypothalamus. Its main function as a hormone is to inhibit the release of prolactin from the anterior lobe of the pituitary.

This chapter deals with the simultaneous determination of ascorbic acid and dopamine at carbon paste electrode using phenyl hydrazine as a mediator in 0.1M KCl. The catechol moiety undergoes two electron oxidation to give dopaquinone. The effect of varying sweep rate, phenyl hydrazine concentration on to the surface, and directly in to the solution for dopamine and ascorbic acid has been discussed. Many authors have been reported that it is very difficult to determine ascorbic acid directly at ordinary (carbon or metal) electrodes because of its large over potential and fouling by oxidation products. Moreover, at bare electrodes, the oxidation of dopamine, which is always present with ascorbic acid in biological tissues, occur at a potential close to that of ascorbic acid. Surprisingly we separated both ascorbic acid and dopamine at bare carbon paste electrode itself only. From this we can say that immobilization is more favourable than mobilization because in immobilization method good increase in the current signal was noticed compare to mobilization method. The effect of addition of phenyl hydrazine can be a consideration for the selective determination of dopamine in the presence of ascorbic acid present in biological systems.

Chapter 7

Cyclic Voltammetric Investigations of 3-Aryl-4-Bromo Sydnone and its Derivatives at Glassy Carbon Electrode

Sydnones are a novel class of mesoionic compounds containing 1,2,3-oxadiazole ring system. The study of sydnones still remain a field of interest because of the
electronic structure and also varied types of biological activity associated with them. Sydnones are the most important representatives of mesoionic compounds because they possess physiological activity of different types depending on substituents in heterocyclic ring. Sydnones find their use as anti inflammatory, antimicrobial, anticancer, and analgesic agents.

This chapter discusses how the mesoionic 3-aryl-4-bromo sydnone and its derivatives undergo one electron reduction in aqueous and nonaqueous media by using 50mM sulphuric acid and 0.1M tetra butyl ammonium iodide as a supporting electrolyte at glassy carbon electrode. Mass spectral data was used to characterize the reduced product by bulk electrolysis confirm the proposed mechanism. The influence of variation of pH on the peak current and peak potential has been studied in acidic range. The shift in the cathodic wave with increase in pH, the substituent effect and the effect of variation of scan rate and concentration on peak parameters have been discussed. The cathodic shift in different organic co-solvents using 50mM sulphuric acid as the supporting electrolyte has been examined using different organic solvents such as acetonitrile, DMF, and DMSO. The electrochemical reduction of 3-aryl-4-bromo sydnone was also carried out in cetyl trimethyl ammonium bromide and triton x-100 as the cationic and non-ionic surfactants respectively.

Specific conclusions are incorporated at the end of each chapter.