CHAPTER I
INTRODUCTION

1.1 Introduction

Electrochemical detection is a technique based on electrical sensors that usually involve an interface between a solid or liquid electronically conducting phase (the electrode) and a solid or liquid ionically conducting phase (the electrolyte) [1]. The types of electrical measurements include potential, current, charge, capacitance, conductance and impedance. Electrochemical methods provide great advantages that make this technique popular such as high sensitivity, simplicity, quickness, and low cost. Proper use of electrochemical detection requires knowledge of redox reactions of the target analytes and their dependence upon the composition of the supporting electrolyte as well as the working-electrode material. In the present chapter, current-based measurements at solid electrodes in liquid electrolytes were focused.

Electrochemical methods are in principle simpler than optical detection, as this is the most direct route from chemical information to the electrical signal required for the processing. Electrochemical detection is also an attractive alternative to optical detection for species that do not absorb in the UV-VIS range or do not fluoresce. Over the past two decades, electrochemical detectors have achieved wide acceptance for monitoring a flowing liquid stream from various systems such as flow injection analysis [2], the liquid chromatographic system [3], and capillary electrophoresis [4]. Electrochemical detectors are suitable in flowing liquid systems because of their selectivity, sensitivity and linear response over a wide concentration range [5]. Furthermore, this sensor has some features that make this technique especially suitable for implementation in a miniaturized analytical system because it is modern and ideally suitable and compatible with microfabrication technology.

Recently, highly boron-doped diamond thin film electrodes have been realized as outstanding electrode material for variety of electrochemical applications such as wide potential window, very low background current, chemical and physical stability. These attributes make conductive diamond well suited for current-based electrochemical measurements.

1.2 Research Objective

There are two targets for this dissertation.

1. To develop a new voltammetric detector using the boron-doped diamond thin film electrode for analysis of thiocompounds.

2. To develop and study the application of microchip capillary electrophoresis by amperometry for the separation and detection of environmental pollutants.

1.3 Scope of Research

To achieve the research objectives, the following scope was set.

- The as-deposited boron-doped diamond thin film electrodes were used for studying the electrochemical properties of thiol-containing drugs consisting of captopril and tiopronin. Particular attention was given towards studying the electrochemical properties of these anlytes using cyclic voltammetry. Comparison results were obtained by glassy carbon electrode. The effect of pH, concentration and scan rate were studied in detail.

- The as-deposited boron-doped diamond thin film electrodes were used as the detector for quantitative analysis of thiol-containing drugs by the amperometric method with the flow injection system. The effect of detection potential, linear dynamic range was also studied.

- The developed method was applied for the determination of thiol-containing drugs in some pharmaceutical preparations. Analytical recovery was determined as well as the precision.

- The anodized boron-doped diamond thin film electrodes were utilized for studying the electrochemical properties of homocysteine using cyclic voltammetry. The effect of pH, concentration of homocysteine and scan rate was investigated in detail.

- The anodized boron-doped diamond thin film electrode was employed as a detector in the HPLC system for separation and detection of homocysteine. Chromatographic and electrochemical condition for the separation and detection of homocysteine were studied as well as the analytical figures.

- The microchip capillary electrophoresis with electrochemical detection was developed for the separation and detection of contaminated hydrazine compounds. The electrophoresis conditions and the detection potentials were investigated.
- The microchip capillary electrophoresis and electrochemical system were designed for rapid screening and identification of phenolic compounds. Also, pH effect, separation voltage, sampling time, and switching time were investigated.
- The microchip capillary electrophoresis for continuous monitoring was developed for the separation and detection of explosive and phenilic compounds. The effect of sample flow rate, applied voltage, and other relevant variables, were described in detail.

There are six chapters in this dissertation. Chapter I is the introduction. Chapter II is the principle of electrochemical technique related to the detection and analysis of chemical compounds. This chapter also covers the theoretical aspects of separation in HPLC, and microchip CE. Chapter III gives details of the experiments for this dissertation including the chemicals, instruments, and chemical procedure used in this work. Chapter IV reports on the use of the boron-doped diamond (BDD) thin film electrode for electroanalysis of thiocompounds. Chapter V presents the microchip capillary electrophoresis coupled with electrochemical detection for the separation and detection of environmental pollutants. Lastly, Chapter VI is the conclusion and future perspectives.