CHAPTER I

INTRODUCTION

1.1 Introduction

Due to the growing interest on micro total analytical system (μTAS), fast analysis of small amount samples with high throughout is highly desired. Separation techniques, such as capillary electrophoresis (CE), show a great potential for miniaturisation in order to perform fast separations at low cost. Microchip capillary electrophoresis is an exciting approach for a rapidly increasing numbers of analytical problems in life sciences and environmental applications.

The first paper on microchip capillary electrophoresis (CE) was published in 1992 and demonstrated that a significant improvement in speed is obtained by miniaturizing conventional separation methods (1). In the broader concept of micro total analysis systems (μTAS), CE separations form an integral part enabling the analysis of complex samples (2-5). The steadily growing number of publications on microchip CE shows the sustained interest and continuing development. A significant amount of research is invested in the development of different functional components that form the building blocks of microchip systems.

Heavy metals are a kind of greatly major contaminants in food and environment. In general, they are not only biodegradable but also have long biological half-lives. There are the potential for accumulation in the different plants and natural sources causing serious health hazards such as renal failure, symptoms of chronic toxicity, and liver damage. According to the stipulation of the World Health Organization, lead, cadmium, chromium, and other heavy metals must definitely be controlled in food in order to assure the safety in food.

Lead and cadmium are among the most abundant heavy metals and are particularly toxic. The excessive content of these metals in food is associated with etiology of a number of diseases, especially with cardiovascular, kidney, nervous as well as bone diseases (6,7). In addition, they are also implicated in causing carcinogenesis, mutagenesis and teratogenesis (8). Another important metal is copper. It is an essential trace element for the human body and contributes to important

intracellular metabolic events (9). Either an excess or a deficiency of copper can result in severe ailments to human (10). One of the major reasons to monitor levels of toxic metals in food is that the contamination of metal has been increasing.

Contemporary methods for metal ion analysis include atomic absorption and emission spectrometry, mass spectrometry, electrochemical methods (potentiometry, voltammetry), colorimetry and ion chromatography. Most equipments are operated by qualified personnel only and are not suitable for portable use. Samples are collected and transported to a central laboratory, which substantially adds to the overall cost of the analysis. Miniaturization using microchip technology offers the opportunity for on-site analysis with equipment that is simple to operate.

Following the trend of miniaturization, microchips have been developed to measure metal ions in aqueous samples based on methods developed initially for conventional CE (11). Microchips for capillary electrophoresis (CE) can be fabricated from a variety of materials. The important property of materials is that the material is electrically insulating and impermeable to water. Predominantly, glass is used. However, polymers such as Poly (dimethylsiloxane) (12), poly(methyl methacrylate (13), polycarbonate (14), cyclic olefin polymers (15), SU-8 (16), polyimide (17) quickly gain more popularity because of the potential to rapidly replicate devices using hot embossing, injection molding or lithography (18). Each polymer materials has unique properties that make them suitable for different applications. The processes for fabrication microfluidic chips from plastics are ,however, not established as well as they are for glass. In this thesis glass chips are used for measuring metal ions in complex mixtures with microchip CE.

Another example of a field in which metal ions analysis plays an important role is in water quality monitoring and environmental analysis. Water companies need to be sure that their products is suitable for consumption. Strict regulations are imposed on the maximum allowed concentration of many potential contaminants like heavy metals. Microchip electrophoresis provides the throughput needed for semi-continuous analysis. The capillary electrophoresis microchip forms the heart of the system for measuring inorganic ions. The design of the microchip and the optimization of the separation conditions are critical to reach the goal of analyzing complex samples.

In this thesis, a microchip capillary electrophoresis system is presented for measuring metal ions. The main goal is to develop the analysis system for determination the metal ions in aqueous samples. Speed, accuracy, robustness and user-friendliness are the criteria which the system has to fulfill. Furthermore, the application of the system for monitoring beverage quality was investigated.

1.2 Research objectives

There are two targets for this thesis

1. To develop an inexpensive, field portable and sensitive analysis device for metal ions analysis.
2. To use the proposed system for the application of microchip capillary electrophoresis by amperometry for the fast screening and detecting of metals in beverage.

1.3 Scopes of the research.

Therefore, this work addresses the need to achieve the research objectives for onsite metal ions analysis, which could assist in the fast screening and detecting of metal ions in contaminated food or waste water. The detection of metal ions was accomplished using a microchip CE / amperometric detection system, in which a screen-printed carbon electrode was placed at the end of channel. This approach simplified the fabrication of the working electrode and also provided a convenient and sensitive means for the determination of metal ions by amperometry. Furthermore, we can easily remove or clean the detection electrode, which is prone to contamination. The microchip CE/amperometric system provided a good portable device for screening or simultaneously analysis of a complex system.

In this thesis, the set up of the glass microchips and the factors affecting the performance of CE microchips for metal ion analysis were studied. The electrochemical properties of these analytes were also investigated using cyclic voltammetry. In addition, The effect of pH, concentration and properties of

background electrolytes were studied. The screen-printed carbon electrode was employed as a detector in the microchip CE system for detection of metal ions. Based on these considerations a microchip with amperometric detection was designed. The effect of detection potential, separation potential, buffer concentration, injection time, linear dynamic range were also studied in micro analysis system. Analytical recovery was determined as well as the precision. The optimization, characterization, attractive performance characteristics of such microchip CE, and its successful application to complex samples (such as vegetable juices) are reported in the thesis.