UNIVERSITI PUTRA MALAYSIA

EMBEDDED MINI-ANALYZER DEVICE FOR IN SITU WIDE RANGE HEAVY METAL IDENTIFICATION AND CONCENTRATION DETECTION

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By

AMIN MAZAHERI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATIONS

First and foremost I would like to thank God, my creator, for giving me the intellectual capacity to learn about His creation. I dedicate my thesis work to my loving parents, Akram and Ali, whose words of encouragement and push for tenacity ring in my ears. In addition, I have a special feeling of gratitude to my loving brother supporting me entire my life. I also dedicate this grateful work to my loving wife, Zeinab whose motivate me to complete my research efficiently. I also would like to thanks her for love, encouragement, admiration, kindness and support.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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April 2018

Chairman : Maryam binti Mohd Isa, PhD
Faculty : Engineering

Metal toxicity is a critical concern in both human health and ecosystem. Many heavy metals are lethal at high concentration. It can also be harmful at trace concentration since accumulating such materials in human organs lead to long-term negative health effects such as cancer, heart disease and high blood pressure. Therefore, heavy metal detection of trace concentration is very important. Heavy metals can be detected using electrochemical detection system. It consists of electrodes, potentiostat that controls the electrode and signal processing block. With the advancement of integrated technology, in-situ electrochemical systems provide feasible solution for sensitive detection and miniaturized platform. The potentiostat as main part of the system; read, amplify and control the current flow through the electrodes.

In this study, the fully differential variable gain potentiostat, would be able to measure wide range current of different types of electro chemicals, typically from 100 nA to 100 mA and can generate an excitation potential between -3V and +3V. This potentiostat is designed with a fully differential operational amplifier and rail-to-rail common-mode range buffer for linearity of output signal.

Voltammetry as electrochemical technique is used in this project for the heavy metals detection. This designed device was able to perform differential pulse anodic stripping voltammetry (DPASV) as a sub techniques of voltammetry. Among many types of voltammetry techniques, differential pulse anodic stripping voltammetry (DPASV) technique was chosen where a pulse shaped voltage is applied on the sen- sor and the current through the sensor is measured to determine

the concentration and types of heavy metal.

To achieve the ability of in-situ processing of detection, embedded algorithms like digital FIR filter, multiple peaks finding, peaks classification and linear regressions have been implemented on an ARM processor. The resulted signals known as voltammogram and the concentration value will be displayed on a graphical LCD. Voltammogram is a plot of current reaction with applied voltage. Tests were carried out for solution with different heavy metals like cadmium (Cd), lead (Pb) and copper (Cu). A concentration range from 0.5 ppm to 10 ppm of lead have been used to test the system accuracy and detection limits. The system was able to detect the heavy metal with correlation factor of 0.99, between the concentration value and voltammogram current peaks.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PERANTI MINI-ANALISIS TERBENAM UNTUK MENGENALPASTI DAN MENGESAN KEPEKATAN LOGAM BERAT DALAM LINGKUNGAN LUAS DI TEMPAT ASAL

Oleh

AMIN MAZAHERI

April 2018

Pengerusi : Maryam binti Mohd Isa, PhD
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Ketoksikan logam adalah keprihatinan kritikal dalam kesihatan manusia dan eko-sistem. Banyak logam berat yang mematikan pada kepekatan yang tinggi. Ia juga boleh memudaratkan kepekatan mengesan sejak mengumpul bahan-bahan sedemikian dalam organ manusia menyebabkan kesan kesihatan jangka panjang yang negatif seperti kanser, penyakit jantung dan tekanan darah tinggi. Oleh itu, pengesanan logam berat kepekatan jejak adalah sangat penting. Logam berat dapat dikesan menggunakan sistem pengesanan elektrokimia. Ia terdiri daripada elektrod, potentiostat yang mengawal elektrod dan blok pemprosesan isyarat. Dengan kemajuan teknologi bersepadu, sistem elektrokimia di-situ menyediakan penyelesaian yang layak untuk pengesanan sensitif dan platform mini. Potentiostat sebagai ba- hagian utama sistem; membaca, menguatkan dan mengawal aliran semasa melalui elektrod. Dalam kajian ini, pembolehubah pembolehubah pembolehubah sepenuh- nya, dapat mengukur arus pelbagai jenis bahan kimia elektro, biasanya dari 100 nA hingga 100 mA dan boleh menghasilkan potensi pengujaan antara -3V dan + 3V. Potentiostat ini direka bentuk dengan penguat operasi kebezaan sepenuhnya dan penampan pelbagai mod rel kereta api-railiiito untuk linieriti isyarat keluaran. Voltammetry sebagai teknik electrochemical digunakan dalam projek ini untuk pengesanan logam berat. Peranti yang direka ini dapat melakukan voltmeter pelucutan anodik pulsa (DPASV) sebagai sub teknik voltammetri. Antara jenis teknik voltammetri, teknik tegasan denyutan anodik tegangan denyutan (DPASV) dipilih di mana voltan berbentuk nadi digunakan pada sensor dan semasa melalui sensor diukur untuk menentukan kepekatan dan jenis logam berat. Untuk men- capai keupayaan pemprosesan pengesanan in-situ, algoritma terbenam seperti pe- napis FIR digital, penemuan puncak pelbagai, klasifikasi puncak dan regresi linier telah dilaksanakan pada pemproses ARM. Isyarat yang dihasilkan dikenali sebagai voltammogram dan

nilai tumpuan akan dipaparkan pada LCD grafik. Voltammo- gram adalah plot tindak balas semasa dengan voltan yang digunakan. Ujian telah dijalankan untuk penyelesaian dengan logam berat yang berbeza seperti kadmium (Cd), plumbum (Pb) dan tembaga (Cu). Pelbagai kepekatan dari 0.5 ppm hingga 10 ppm plumbum telah digunakan untuk menguji ketepatan dan had pengesanan sistem. Sistem ini dapat mengesan logam berat dengan faktor korelasi 0.99, antara nilai tumpuan dan puncak voltammogram semasa. .

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy.The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ADC	Analog to digital converter
ASV	Anodic stripping voltammetry
CE	Counter electrode
CPU	Central processing unit
CV	Cyclic Voltammetry
DAC	Digital to analog converter
DPASV	Differential pulse anodic stripping voltammetry
DPV	Differential pulse voltammetry
GUI	Graphical user interface
HMDE	Hanging mercury drop electrode
IC	Integrated circuit
I2C	Inter integrated circuit
I/O	Input/Output
LCD	Liquid crystal display
LED	Light emitting diode
Op-Amp	Operational amplifier
PC	Personal computer
PCB	Printed circuit board
RE	Reference electrode
SWASV	Square wave anodic stripping voltammetry
SNR	Signal to noise ratio
WE	Working electrode

CHAPTER 1

INTRODUCTION

1.1 Introduction

In modern century, electrochemical devices are widely used in several areas, including the biotechnology, physics and chemistry laboratories as well as industrial, food and environmental monitoring. These instruments are used to detect, identify, monitor and analyze critical parameters of chemical reactions [3, 13]. In recent years, there is an increasing interest in employing electrochemical electrode sensors in portable devices. The electrochemical detection techniques are recommended, not only because they are cost effective, rather they can be used for real-time portable devices with high reliability [14–20].

Electrochemical devices utilize electrode sensors for detecting chemical elements; for example they can be used as an implantable microchip to check the content level of blood, such as oxygen, glucose and cholesterol [21] or to identify toxic metals in drinking waters [5, 14]. Generally, an electrochemical sensor reacts with the chemi- cal elements of interest by exchanging an electrical current which is proportional to the concentration of that species. There are different electro-analytical techniques that can be used to control the chemical reaction between the sensor and solution. Voltammetry is one of the common method that use for the quantitative determina- tion of substances in solutions. These technique can be used for identifying transfer of electron in a variety of solvent.

Figure 1.1 illustrates the block diagram of the electrochemical instrumentation sys- tem [5]. This electrochemical instrument includes electrochemical sensor, data conversion, microcontroller and potentiostat. Potentiostat is an electronic circuit that utilized to perform the electro-analytical techniques by applying an excitation po- tential on an electrode sensor and then read the produced current from the sensor. Basically, a potentiostat has two main functions, controlling the potential difference between working electrode (WE) and reference electrode (RE) and measuring the current flowing between working electrode and counter electrode. The excitation signal is generated by the microcontroller in digital form and is then converted to analog form using a digital to analog converter (D/A) [6]. It is applied to the counter electrode (CE) and reference electrode (RE) via a potentiostat which acts to control the applied potential. The signal output, in the form of current, is obtained from working electrode (WE). The generated current is the result of electrochemical reactions occur at the surface of the electrode. The amount of current is related to the concentration of electro-active elements, applied voltage on the sensor and area of the electrode sensor. at the data acquisition process, the current is digitized by an analog to digital converter (A/D) under the control of the microprocessor. These digital numbers are then stored in the memory for storage and further processing.

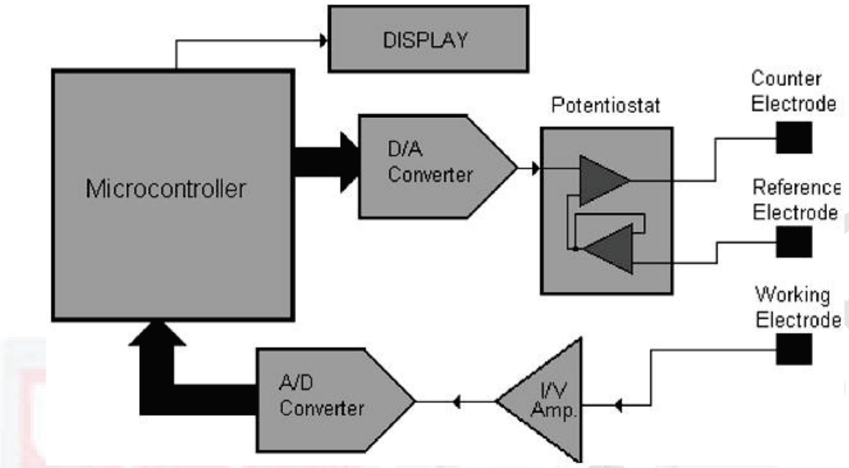

Figure 1.1: A Simplified Block Diagram of the Electrochemical Instrumentation System

1.1.1 Heavy Metals Toxicity and Environment Monitoring

Metal toxicity is a critical concern in both human and ecosystem health. Many heavy metals are lethal at high concentrations. It can also be harmful at low concentration and lead to long-term negative health effects such as heart disease, high blood pressure and cancers [7-9]. In fact, after the penetration of these metals into the body, accumulate in tissues such as fat, muscle, bones and joints and cause many diseases and bring various other aggravating problems to human [10, 11]. Environmental pollution from industry is the main source of heavy metals in the environment. As shown in Figure 1.2, the effect of heavy metals for example lead and cadmium in the human body is often associated with some complications as in the following: Getting cold feet, immunodeficiency, skin rashes, digestive problems, fatigue, heart disease, high blood pressure, irritability, allergy, forgetfulness and dizziness.

Environmental monitoring is important for evaluating and mitigating threats to the environment and public health, tracking natural resources for reducing the costs associated with waste treatment. The need for inexpensive analytical tools is not thus not limited to the area of health care. Such tools will also be useful in food safety testing and a variety of applications from environmental monitoring.

Current monitoring methods required by regulatory bodies are often expensive, time-consuming and require skilled personnel and a laboratory equipped with ex-pensive analytical tools. In some large scale projects laboratory analysis are more costly of the remediation effort [22]. In addition to the costs, the integrity of the analyses can also be compromised at any point within the usually multiple day pro- cess of sample collection, storage, transport and analysis. Portable instrumentation that allows on-site sampling and analysis has the potential to make environmental monitoring simpler, faster and inexpensive. The instruments can be designed to work unattended and to store data for periodic retrieval. Devices with wide range voltage control capability will enable more routine monitoring of environments to support more electro chemicals. These field portable devices installed for environ- mental monitoring could also incorporate intrusion sensors to detect intentional damage such as might occur in a biological or chemical leakages. Furthermore, the resources and diagnostic tools required for rapid identification of electro-chemicals.

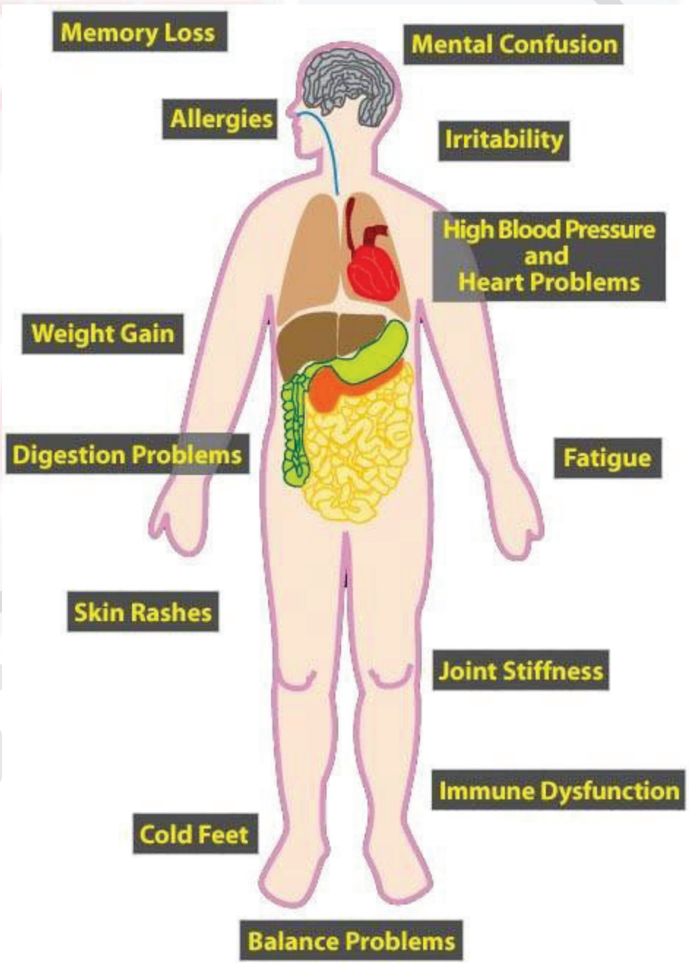

Figure 1.2: Effect of Accumulation of Heavy Metals in Human Body [1]

1.1.2 Electrochemistry

In this work, a fully differential potentiostat is utilized to accomplish differential pulse anodic striping voltammetry (DPASV) method. Differential pulse anodic stripping voltammetry is a technique in which a pulse that placed on a ramp voltage is applied on sensor electrodes. The electrodes are dropped in a solution with an electro-active chemical element. The combination of electrodes and solution are named as electrochemical cell. When electrochemical reaction occur, the sensor generates a current peak at a specific voltage level. Electrochemical reaction current is known as faradaic current. The potentiostat is responsible for measuring this current which normally is in mili to nano-amper range.

The plot of measured current versus cell voltage graph is called voltammogram and for a DPASV experiments looks like a bell shape curve. At the voltammogram the voltage position of measured peak is unique for each elements which is called $E_{1/2}$ and at that specific voltage have the maximum electrochemical reaction. This property can be used for identification of an unknown chemical. Additionally, the height of the voltammogram peak which representing the current of sensor, provides a measure of the concentration of chemical.

The proposed system are able to apply a wide range of DPASV excitation signal from -3V to +3V that based on references can cover the $E_{1/2}$ voltage of 30 different kinds of traced chemicals [1]. On the other hand the device is targeted to support a wide range of sensor current readout from nA to mA, which is essential for different range of chemical concentrations.

1.2 Problem Statement

The effect of contamination by highly toxic metals on human health, economic, agriculture products and natural water, forcing industry and regulatory bodies to monitor pollutant levels at different points of natural water, industrial, and cities. Therefore, significant number of properly trained staff members are used by several organizations to monitor pollutant in areas. Typically, pollutant monitoring needs sample collection and specific laboratories. The instruments currently used for the analysis of samples are expensive devices and involve trained personnel to carry out the analysis and to understand the results. Additionally, these systems are usually too large to be used in the field. It has to be connected to a computer and in some application, it is a longtime process. It is also not user friendly where, only graphs with peak value was given with unknown type of heavy metal and the unknown level of concentration. In a number of applications, the longtime delays related with this procedure are unsatisfactory, and make online monitoring necessary.

Almost all the validated devices currently on the market are still too costly for the limited resource communities that have great need for such equipment. Professional companies offer a range of equipment for general purpose electrochemistry appli- cations that can be used in many forms of electrochemical analysis. The price of those stats differs in the range of $15k to $20k for big research style analyzers with 20-30 kg weigh. For less complex devices weighing l-3 kg, the cost is approximately

$5k to $10k, less compact systems use desktop/laptop computers to interface to the system to run the analysis. Furthermore, developing a field-ready, online systems needs real time electrochemical data analysis. Basically, research on potentiostats is categorized into three parts which are the potentiostats designed to improve accu- racy and detection limit, potentiostats integrated for biological array applications, and potentiostats integrated with mixed-signal functionality. The voltage gain, input offset voltage, output voltage swing and input referred noise of the poten- tiostat are defined the potentiostat accuracy. Recently researchers have developed potentiostat based on CMOS technology but for the detection of limited type of heavy metals [18, 46]. In order to detect trace concentration of heavy metals, the potentiostat should be able to detect wide range current typically in the range of mA to nA. Scaled down CMOS technology which tends to operate at lower current may be useful for detecting low concentration of heavy metals. Although down- scaling trend of CMOS technology has significantly improved the performance of digital system, but, the decreasing supply voltage imposes challenges to analog de- sign and limited the range of required voltage for wide range detection. Therefore, an inexpensive miniaturized instrument capable of performing in-situ measuring of different electro-active samples by generating a wide range of excitation signal, is demanded. Also, stand-alone analyzing of data with real time plotting is necessary for a portable system.

1.3 Research Objective

The aim of this dissertation is to design a prototype hand-held automated elec-trochemical analyzer system that could perform electrochemical measurements for the purpose of heavy metal detection by using electronic circuit for control, sig- nal processing and data storage. Numerous commercial instruments that perform such analysis are available; however, their size and cost inhibit their application for on-site testing. The resulting prototype addresses the problem of detecting heavy metals with a low-cost hand-held device. It has considerable advantages of stand-alone data analyzing over the laboratory-based system and could be used for inexpensive electrochemical experiments. The costs, energy efficiency, and ease of use were considered as part of the system design. In order to achieve this aim, the following objectives have been set:

1. To design a fully differential potentiostat that is able to generate a wide range of potential over the electrochemical three-electrode sensor from -3V to +3V and read the current of chemical reactions. The variable gain circuit, allows measurement of a wider range of currents from 100nA to 100mA.

2. To implement the sub-units of the portable device as like power unit, filters, level shifters and the signal processing block to verify the functionality and the performance of the system. Implements embedded signal processing algorithms to automatically analyzing sampled data in order to detect a heavy metal and identify its concentration. The algorithms contain FIR filtering, peaks detection, statistical prediction and linear regression.

3. To evaluate the system performance by testing several types of heavy metals with different concentration. Additionally, the portable device have touch screen graphical LCD to shown the online experiment signals and processed data.

1.4 Scope of the Work

The mini-analyzer device is able to detection heavy metals such as cadmium (Cd), lead (Pb) and copper (Cu) with the concentration range from 0.5 ppm to 10 ppm. Those three heavy metals are used to verify device processing functionality and comparing with commercial micro-auto lab device. The performed statistic and calibration data of Lead, in a concentration range of 0.5ppm to 10ppm is stored on the system memory. Because of portable processing ability and online display of results, the sample tests can be done on site.

1.5 Thesis Organization

The outline of the thesis is as follows:

Chapter 1 gives an introduction to electrochemical systems, highlighting the importance of the research for heavy metal detection and monitoring are presented.

Chapter 2 provides an overview of the electrode sensors, methods configuration and detection principles. Detail description of the analytical electrochemistry theory and equations associated with the electrochemical analyzer. The configuration of different potentiostat and its fundamental operations in the perspective of heavy metal detection, are outlined.

Chapter 3 includes details of experimental procedures. The procedures outline the electrochemical experiments including the sensor, materials to be deposited, the deposition method, voltammetry techniques employed and method of detection. The chapter also explains the design and development of the portable hand-held electrochemical analyzer system with details of the analog interface circuit, the dig- ital circuit design, PCB layouts, embedded software and user interface. Details are presented on the analog and digital hardware of the system and the embedded algo- rithms executed on the digital hardware. The analysis methods used for analyzing the data obtained from electrochemical analyzer system is presented.

Chapter 4 provides the results and discussion acquired from the controlled testing of the electrochemical device and illustrate the electrochemical experiments using the portable device.

Chapter 5 concludes and gives potential direction for the future research on elec- trochemical analyzer systems.

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LIST OF PUBLICATIONS

Enhancing Real Time Heavy Metal Detection Using Embedded Portable Signal Processing Unit

Real-Time Processing of Differential Pulse Voltammetry (DPV) Data Using LPC1768

A comparison between two different FPGA-based topologies of first order sigma-delta modulator

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