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VOLTAMMETRY DETERMINATION OF ZINC IN VARIOUS TYPES OF SOIL

Dissertation submitted in partial fulfillment for the Bachelor of Science (Hons) in Forensic Science

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LIST OF ABBREVIATIONS

Zn: Zinc

ASV: Anodic stripping voltammetry

CSV: Cathodic stripping voltammetry

AdSV: Adsorptive stripping voltammetry

DPASV: Differential pulse anodic stripping voltammetry

AAS: Atomic absorption spectrometry

ICP-MS: Inductively coupled plasma mass spectroscopy

HDME: Hanging mercury drop electrode

MFE: Mercury thin film electrode

Ag: silver

AgCl: silver chloride

KCl: potassium chloride

Pt: platinum

NH_3: ammonia

NH_4Cl: ammonium chloride

NaAc: sodium acetate

H_2SO_4: sulphuric acid

H_2O_2: hydrogen peroxide

HNO_3: nitric acid

NaOH: sodium hydroxide

HCl: hydrochloric acid

I: current

U: potential

LIST OF SYMBOLS

°C: degree celcius

rpm: revolutions per minute

mV: millivolt

A: ampere

s: second

mV s^{-1}: millivolt per second

cm: centimetre

µm: microgram

g: gram

%: percent

µL: microlitre

mL: mililitre

µg/L: microgram per litre

mg/L: milligram per litre

g/L: gram per litre

ppb: part per billions

R^2: correlation coefficient

ABSTRACT

Zinc plays an important role in plant growth as the plant micronutrient. The soil samples were collected from different location to compare the concentration of Zn. Then, the soil sample was digested using H_2SO_4 and H_2O_2. Anodic stripping voltammetry (ASV) technique was used to determine the concentration of Zn and the parameters used for the determination of Zn are as follows: start potential: -1.15 V, end potential: -0.7999 V, deposition potential: -1.15 V, deposition time: 90 s, equilibrium time: 10 s, voltage step: 0.005951 V, voltage step time: 0.1 s, sweep rate: 0.0595 V/s, pulse amplitude: 0.05005 V, and pulse time: 0.04 s. One mL acetate buffer with pH 4.64 was used as supporting electrolytes. The peak potential of Zn was found at -0.995 V and the concentration of Zn was determined by the standard-addition method. From ten samples of soil, the underground soil showed the lowest concentration of Zn measured at 2.95 ppm while the concentration Zn in soil from the area of waste incinerator is the highest measured at 57.47 ppm. As the conclusion, ASV technique was successfully applied for the determination of Zn in various types of soil and the concentration of Zn in soil was found to be different from one location to another location.

ABSTRAK

Zink memainkan peranan penting dalam pertumbuhan tumbuhan dimana Zn bertindak sebagai mikronutrien kepada tumbuhan. Sampel tanah telah dikumpulkan dari lokasi yang berbeza untuk membandingkan kepekatan Zn. Kemudian, sampel tanah telah dicerna menggunakan H_2SO_4 dan H_2O_2. ASV telah digunakan untuk menentukan kepekatan Zn dan parameter yang digunakan untuk menentukan kepekatan Zn adalah seperti berikut: keupayaan awal: -1.15 V, keupayaan akhir: -0.7999 V, keupayaan pengumpulan: -1.15 V, masa pengumpulan: 90 s, masa keseimbangan: 10 s, langkah voltan: 0.005951 V, masa langkah voltan: 0.1 s, kadar imbasan: 0,0595 V/s, amplitud denyutan: 0.05005 V, dan masa denyutan: 0.04 s. 1 ml buffer asetat dengan pH 4.64 telah digunakan sebagai elektrolit sokongan. Keupayaan puncak Zn didapati pada -0.98 V dan kepekatan Zn telah ditentukan oleh kaedah penambahan piawai. Dari sepuluh sampel tanah yang dikaji, tanah bawah tanah menunjukkan kepekatan terendah Zn iaitu 2.95 ppm manakala kepekatan Zn dalam tanah dari kawasan pembakaran sampah adalah yang tertinggi iaitu 57.47 ppm. Sebagai kesimpulan, teknik ASV berjaya digunakan untuk penentuan kepekatan Zn dalam pelbagai jenis tanah dan kepekatan Zn dalam tanah adalah berbeza dari satu lokasi ke lokasi yang lain.

CHAPTER 1

INTRODUCTION

1.1 Zinc

Zinc is a silvery-white and lustrous metal. According to the Jefferson National Linear Accelerator Laboratory, the properties of zinc are zinc has the symbol Zn with the atomic number 30 and the atomic mass is 65.38. The density of Zn is 7.134 g/cm^3 which relatively heavy and will sink in water while the melting point and boiling point is 419.53 °C and 907 °C respectively. It is in solid phase at room temperature.

There are many uses of Zn such as to prevent rusting by undergo a process known as galvanization in which Zn galvanized other metals, such as iron that are applied in car bodies, street lamp posts, safety barriers and suspension bridges (Emsley, 2011). Zn is also used to produce die-castings, which are important in the automobile, electrical and hardware industries. Brass, nickel silver and aluminium solder are the example of alloys that used Zn (Emsley, 2011).

Other than that, zinc oxide is widely used in the manufacture of many products such as paints, rubber, cosmetics, pharmaceuticals, plastics, inks, soaps, batteries, textiles and electrical equipments. Zinc sulfide is used in making luminous paints, fluorescent lights and x-ray screens as it will glows when exposed to ultraviolet light (Emsley, 2011).

1.2 Soils

Soil is a product of the physical chemical and biochemical breakdown of the Earth's surface into small wreckages, including sand, silt, and clay. The product of organic matter decomposition such as the composting of dead plant and animal debris also made up the soil composition (Bradshaw-Rouse and Judith, 2013). The classification of soil is based on the soil profile and its formation. Agronomic use, colour, organic matter content, texture, and moisture condition are the characteristic that are used for grouping the soils (Bradshaw-Rouse and Judith, 2013).

The formation of soil takes hundreds or even thousands of years. It depends on parent material, climate, organisms, topography, and time. The igneous, sedimentary, and metamorphic rocks where the fragments of which may be deposited by water, wind, and ice and also plant and animal deposits are sources of parent material (Bradshaw-Rouse and Judith, 2013).

According to North Carolina Department of Agriculture and Consumer Services portal, 2014, in general, nutrients are essential for most plants to grow by absorbing it from the soil and the nature of the soil affected their ability to do this. The extent to which nutrients are available to plants is depend on the soil texture and its acidity (pH) and it also affects how well nutrients and water are retained in the soil. As the example, clays and organic soils hold nutrients and water much better than sandy soils. A condition called leaching usually occurs in sandy soils where the nutrients will carries along with water as water drains from sandy soils. As a result, the nutrients are not available anymore for plants to use.

1.3 Role of zinc as the plant micronutrient

Micronutrients are those elements essential for plant growth which are needed in only very small (micro) quantities. These elements are sometimes called minor elements or trace elements, but use of the term micronutrient is encouraged by the American Society of Agronomy and the Soil Science Society of America.

Manganese, copper, boron, iron, zinc, chlorine, molybdenum and nickel are trace elements that are important for normal, healthy growth and reproduction of plants. Zn is required as a structural component of a large number of proteins, such as transcription factors and metalloenzymes (Figueiredo *et al.*, 2012). With insufficient amount of Zn, the plants will suffer from physiological stresses due to the failure of metabolic processes in which Zn plays a critical role. Zn in soils can be separated into fractions based on particle size distribution and/or chemical analysis procedures (Sadeghzadeh, 2013).

Zn is essential for the growth in animals, human beings, and plants it is vital to the crop nutrition as required in various enzymatic reactions, metabolic processes, and oxidation-reduction reactions. In addition, Zn is also essential for many enzymes which are needed for nitrogen metabolism, energy transfer and protein synthesis (Hafeez *et al.*, 2013).

Zn plays a very important role in plant metabolism by influencing the activities of hydrogenase and carbonic anhydrase, stabilization of ribosomal fractions and synthesis of cytochrome (Tisdale *et al.*, 1984). Plant enzymes activated by Zn are involved in carbohydrate metabolism, maintenance of the integrity of cellular membranes, protein synthesis, regulation of auxin synthesis and pollen formation (Marschner, 1995). The regulation and maintenance of the gene expression required for the tolerance of environmental stresses in plants are Zn dependent (Cakmak, 2000).

1.4 Voltammetric technique

In 1922, the Czech chemist Jaroslav Heyrovsky developed voltammetry technique from the discovery of polarography and in 1959, he received the Nobel Prize in chemistry. At the early development, this method having a number of difficulties that make it less than ideal for routine analytical use (Kounaves, 1997). However, the significant advances were made including the theory, methodology, and instrumentation in the 1960s and 1970s, which improved the sensitivity and expanded the range of analytical methods. The coincidence of these advances with the advent of low-cost operational amplifiers also facilitated the rapid commercial development of relatively inexpensive instrumentation (Kounaves, 1997).

The application of a potential (E) to an electrode and the monitoring of the resulting current (I) flowing through the electrochemical cell that involved in this technique are the common characteristics of all voltammetric techniques. The applied potential is varied or the current is monitored over a period of time (t). Thus, all voltammetric techniques can be described as some function of E, I, and t. The applied potential forces a change in the concentration of an electroactive species at the electrode surface by electrochemically reducing or oxidizing it and considered as active techniques (Kounaves, 1997).

The electrochemical cell that involved in voltammetric experiment consists of a working (indicator) electrode, a reference electrode, and a counter (auxiliary) electrode. In general, an electrode provides the interface across which a charge can be transferred or its effects felt. The working electrode is the electrode where the reaction or transfer of interest is taking place. The reduction or oxidation of a substance at the surface of a working electrode, at the appropriate applied potential, results in the mass transport of new material to the electrode surface and the generation of a current. The fundamental principles and applications of various types of voltammetric techniques are derived from the same electrochemical theory (Kounaves, 1997).

There are three common variations of stripping technique including anodic stripping voltammetry (ASV), cathodic stripping voltammetry (CSV), and adsorptive stripping voltammetry (AdSV). These techniques have the lowest limits of detection of any of the commonly used electroanalytical techniques while the sample preparation is minimal but sensitivity and selectivity are excellent (Kounaves, 1997). Mercury is usually chosen as the electrode for the stripping voltammetry. In ASV, the species of interest can be is reduced into the mercury, forming amalgams while in CSV is adsorbed to form an insoluble mercury salt layer. Stripping voltammetry is a very sensitive technique for trace analysis. As the other quantitative technique, care must be taken in order to obtain the reproducible results. The electrode surface, rate of stirring, and deposition time are the important conditions that should be held constant (Kounaves, 1997).

CHAPTER 2

LITERATURE REVIEW

Micronutrients play an important role to stimulate enzymes activities in the plants for physiological and biochemical activities (Fageria *et al.*, 2011). Manganese, copper, boron, iron, zinc, chlorine, molybdenum and nickel that are the eight essential micronutrient for normal, healthy growth and reproduction of plants (Figueiredo *et al.*, 2012).

Zn is constituent of several enzymes that essential in carbohydrate and protein synthesis, maintaining the integrity of membranes and regulating auxin synthesis, photosynthesis, conversion of sugars to starch, resistance to infection by certain pathogens and also important in pollen formations (Alloway, 2008). Plants will suffer from physiological stress if the amount of Zn is not sufficient and under Zn-deficient conditions, flowering and fruit development are reduced, and growth period is prolonged resulting in delayed maturity, leading to lower yield, poor quality and suboptimal nutrient use efficiency (Suresh *et al.*, 2013).

In every part of the world, Zn deficiency can be found and almost all crops respond positively to application of Zn (Welch, 2002). Normal soils inherit their trace elements which include Zn primarily from the rocks through geochemical and pedochemical weathering processes. Besides mineralogical composition of the parent material, the total amount of Zn present in the soil is also dependent on the type, intensity of weathering, climate and numerous other predominating factors during the process of soil formation (Saeed and Fox, 1977).

The total Zn is low in highly leached, acidic or sandy soils such as in many coastal areas. The concentrations of Zn in quartz are very low as reported that quartz in the soil will dilutes Zn, which the concentrations ranged from 1.0 $\mu g\ g^{-1}$ to < 5 to 8 $\mu g\ g^{-1}$ (Helmke *et al.*, 1977). According to the Food and Agriculture Organization (FAO), about 30% of the cultivable soils of the world contain low levels of plant available Zn (Sillanpaa, 1990). Usually, the amount of Zn in calcareous soils, sandy soils, peat soils, and soils with high phosphorus and silicon are insufficient (Alloway, 2008).

There are a few techniques that have been used for analysis of trace metal with varying degrees of success and convenience. These techniques include UV-visible spectrophotometry, flame atomic absorption spectrometry, electrothermal atomic absorption spectrometry, inductively coupled plasma atomic emission spectrometry, total reflection X-ray fuorescence spectrometry and electrochemical stripping analysis. Among the various techniques, stripping analysis offers the advantages of species characterization and of utilizing inexpensive instrumentation and low operating cost (Wang, 1985).

When compared with other spectrometric methods, only electrothermal atomic absorption spectrometry has nearly the same sensitivity but it is costly. The instrument for stripping analysis is small in size, has very low power demand, and requires no special installation such as cooling or ventilation (Wang, 1985). These features make stripping analysis suitable for in situ measurement at which contamination or sample loss from adhesion to sample bottle during storage and transport can be minimized. None of the other techniques for trace metal quantitation can compete with stripping analysis on the basis of sensitivity per money invested (Lau and Cheng, 1998).

The literature on the use of the anodic stripping voltammetry method (ASV) for the determination of metals in soil is very limited based on a paper reported by Opydo in 1989. Reddy *et al.* (1982) determined the total content of lead, cadmium, zinc and copper in $HCl+HNO_3$ extracts by differential pulse anodic stripping voltammetry (DPASV) while Forbes *et al.* (1979) determined selenium in soils by the same method. The concentrating metals on the hanging mercury drop in ASV method makes it possible to determine very low concentrations of lead, cadmium, zinc and copper.

The limit of detectability of anodic stripping voltammetry with reference to amalgamated metals is 10^{-9} M. The selection of the appropriate base electrolyte makes it possible to determine several ions at the same time and the necessary equipment is easily available. The precision of this method is very good and comparable to the AAS method, whereas the limit of detectability in ASV is generally lower (Opydo, 1989).

Barbeira and Stradiotto (1997), reported the determination of traces of heavy metals which are Zn, Pb and Cu in rum samples, pure and oak cask matured, by anodic stripping voltammetry (ASV) with a hanging mercury drop electrode without previous treatment and in the absence of a supporting electrolyte. Then, the results were compared with those obtained by atomic absorption spectrometry (AAS) as shown in Table 2.1. The instrument used for the ASV analysis was an IBM-PC interfaced instrument consisting of a BASIC programme (Machado *et al.*, 1992), a converter interface and a Metrohm Model E506 polarograph coupled with Metrohm Model 663VA stand. The polarographic cell contained a working electrode (Metrohm Model 6.1246.020 multi-mode electrode), a carbon rod (Metrohm model 6.123.345) as auxiliary electrode and an $Ag/AgC1/KCl_{sat}$ reference electrode. The AAS measurements were carried out in an atomic absorption/flame emission spectrophotometer

(Shimadzu Model AA-680), equipped with an air-acetylene burner of 10 cm slot burner head and metal cathode-hollow lamps (Zn, Pb and Cu). The optimum instrumental conditions for each element were fixed automatically by the equipment (Barbeira and Stradiotto, 1997).

Table 2.1: Concentration (μg/L) of Zn, Pb and Cu ions in rum samples

Sample	**Element**	**ASV**	**AAS**
Sample 1	Zn	64 ± 4	70 ± 15
	Pb	65 ± 4	Not detected
	Cu	3 ± 1	Not detected
Sample 2	Zn	44 ± 3	48 ± 11
	Pb	32 ± 3	Not detected
	Cu	14 ± 2	Not detected
Sample 3	Zn	69 ± 3	62 ± 14
	Pb	52 ± 2	Not detected
	Cu	45 ± 3	Not detected
Sample 4	Zn	59 ± 6	68 ± 17
	Pb	23 ± 6	Not detected
	Cu	20 ± 2	Not detected

The study about determination of zinc in environmental samples by anodic stripping voltammetry has been done by Oi-Wah Lau and Oi-Ming Cheng (1998). Anodic stripping voltammetric measurements were carried out with a portable digital voltammeter PDV 2000 (Chemtronics, Bentley, WA). The instrument consists of a power unit, a microprocessor and a cell module. The cell consists of an inverted working electrode (glassy carbon), reference electrode (Ag/AgCl/ KCl(3M)) with double junction, auxiliary electrode (Pt), stirrer, sample inlet and drain. The working parameters were programmed by the built-in microprocessor for semi-automatic operation. The analytical parameters used to determine zinc are as follows:

stirrer speed, 2000 rpm; plating potential, -1400 mV; plating time, 60 s; holding potential, -1400 mV; holding time, 15 s; sweep rate, 375 mV s^{-1}; final potential, -100 mV; strip time, 2 s; rest potential, -100 mV; buffer, 0.25 M NH_3/1M NH_4Cl. A staircase waveform was used during the stripping step (Lau and Cheng, 1998). It was applied to determine zinc in tap water, contaminated natural water, domestic wastewater, air filter, oyster tissue, sewage sludge and sediment samples, and the results were compared with FAAS. The results are shown in Table 2.2.

Table 2.2: Concentration of Zn in environmental samples

Sample	**ASV**	**FAAS**
Tap water	295 µg/L	290 µg/L
Contaminated natural water	2693 µg/L	2590 µg/L
Domestic wastewater	66 µg/L	70 µg/L
Air filter	214 µg	206 µg
Oyster tissue	910 µg/kg	936 µg/kg
Sewage sludge	3830 µg/kg	4000 µg/kg
Sediment samples	114 µg/kg	110 µg/kg

Jothimuthu *et al.* (2013) reported a study about zinc detection in serum by anodic stripping voltammetry on microfabricated bismuth electrodes. The authors found that the conventional methods for measuring Zn in fluids are accurate, but costly and time consuming. For example, Rahman and Wahid (2009) used instrumental neutron activation analysis (INAA) and atomic absorption spectrophotometry (AAS) to validate quantification of Zn in whole blood of cardiovascular diseases (CVD) and malignant hypertension (MH) patients. Barany and Bergdahl (1996) used inductively coupled plasma mass spectroscopy (ICP-MS) to measure multiple trace elements including Zn in blood and serum of adolescents. The most critical challenge of these conventional methods is the time delay from sample collection, shipment to a certified metals laboratory, and reporting results to a clinician. Also, laboratory costs associated with these repeated measurements may be high. These challenges are particularly important when dealing with critically ill patients (Jothimuthu *et al.*, 2013).

Stripping analysis is a powerful yet simple approach to measuring trace metal concentrations. It offers very low limits of detection and simple instrumentation. Thus, it is preferred for “on-site” applications and can be miniaturized for portable and inexpensive field operations (Kissinger and Heineman, 1996). Mercury electrodes are typically used in stripping analysis and produce reliable measurements. Different forms of mercury electrodes have been reported in literature for the detection of zinc including hanging mercury drop electrode (HDME), mercury thin film electrode (MFE), chemically modified mercury film electrode and mercury film on screen printed carbon-paste electrodes (Martinotti *et al.*, 1995). However, a critical challenge for “on-site” applications is the toxicity of mercury electrodes leads to strict safety and handling requirements (Jothimuthu *et al.*, 2013).

CHAPTER 3

OBJECTIVES

3.1 To study and understand the working procedure of voltammetric technique for determination of zinc.

3.2 To determine the concentration of zinc in various types of soil.

CHAPTER 4

MATERIALS AND METHODS

4.1 Instruments

4.1.1 A Metrohm 797 VA processor with three electrodes system comprising a hanging mercury drop electrode (HMDE) as the working electrode, a KCl reference electrode, a Pt auxiliary electrode were used to obtain the voltammogram. 797 VA Computrace is a modern voltammetric work station connected to the PC via USB port. The complementary PC software which is 6.6053.030 VA Computrace 797 software 1.3x is used to control the determination, recorded and evaluated the measured data. The measuring technique that has been used is Anodic Stripping Voltammetry technique.

4.1.2 A pH meter (Hanna Instrument pH211 Microprocessor) was used to determine the pH of supporting electrolyte.

4.1.3 An electronic balance (Shimadzu ATX224) was used to weigh soil samples, KCl and NaAc.

4.1.4 Hot plate

4.2 Apparatus

4.2.1 Volumetric flasks (50 mL and 100 mL)

4.2.2 Conical flask (100 mL)

4.2.3 Weighing plate

4.2.4 Spatula

4.2.5 Micropipette (100 µL, 200 µL and 1000 µL)

4.2.6 Glass pipette (5 mL)

4.2.7 Pipette bulb

4.2.8 Dropper

4.2.9 Sieve

4.2.10 Mortar and pestle

4.2.11 Soil sampler

4.2.12 Plastic bags

4.3 Chemicals and reagents

4.3.1 96% H_2SO_4

4.3.2 30% H_2O_2

4.3.3 Deionised water

4.3.4 Acetate buffer, pH= 4.6

4.3.5 Zinc standard

4.3.6 KCl

4.3.7 NaAc

4.3.8 0.01M HNO_3

4.3.9 1.0M NaOH

4.3.10 1.0M HCl

4.4 Samples

10 types of soil sample were collected randomised from different places using soil sampler in a depth of approximately 0-20 cm. Soil samples were dried at 100 °C and grinded to pass through a 60 μm-mesh sieve.

4.4.1 Sample 1: Commercial soil (from nursery)

4.4.2 Sample 2: Soil from paddy field

4.4.3 Sample 3: USM's hostel soil

4.4.4 Sample 4: Muddy soil (flooded Dec 2014)

4.4.5 Sample 5: Burned soil (from area of waste incinerator)

4.4.6 Sample 6: Underground soil (about 10ft)

4.4.7 Sample 7: Soil from beach area

4.4.8 Sample 8: Soil from batik factory area

4.4.9 Sample 9: Soil from agricultural area

4.4.10 Sample 10: Soil from river

4.5 Procedures

4.5.1 Soil digestion

Approximately 250 g of soil sample was weighed and transferred into 100 mL conical flask. 4 ml of 96% H_2SO_4 was added and the mixture was heated. After that, 1 mL of 30% H_2O_2 was added and the mixture was heated. H_2SO_4 was continue added until the solution turn to colourless and clear. The addition of H_2O_2 were different for some types of soil as shown in Table 4.1 and it might be due to different reaction that occurs among the soils. Then, the solution was left evaporated to 5 mL and cooled at room temperature. The solution was then diluted with deionised water into 100 mL volumetric flask.

Table 4.1: The addition of H_2SO_4 and H_2O_2

Sample	H_2SO_4 (ml)	H_2O_2 (ml)
1	4	4
2	4	3
3	4	3
4	4	3
5	4	4
6	4	3
7	4	3
8	4	3
9	4	3
10	4	2

4.5.2 Preparation of Zn standard (10mg/L)

0.5 mL of 1000 mg/L Zn standard was taken and mixed with 0.01M HNO_3 into 50 mL volumetric flask.

4.5.3 Preparation of acetate buffer (pH= 4.6)

11.18 g of KCl and 4.18 g of NaAc were weighed and dissolved in 100 mL volumetric flask with deionised water. The pH of acetate buffer was adjusted by adding 1.0M HCl and/or 1.0M NaOH.

4.5.4 Voltammetry determination of zinc in sample

5 mL of the digested soil solution was diluted in a 100 mL volumetric flask with the addition deionised water. After dilution, 10 mL of sample was put into the electrochemical cell and 1 mL of acetate buffer with pH 4.6 was added. The measurement cycle using the parameters as listed in Table 4.2 was repeated thrice on the same solution, with a new mercury drop for each measurement. The concentration of Zn was determined by the standard-addition method, by marking three successive additions of the known volume of Zn standard solution which is 0.1 mL with the concentration of 10 mg/L to the solution, repeating the measurement cycle each time, beginning from the deposition stage. From the linear plots of peak height versus amount of Zn standard added, the amount of Zn in the sample was read and measured. The parameters of the voltammetric analysis are in Table 4.2.

Table 4.2: Parameters for voltammetric analysis

Parameter	
Initial purge time (s)	300
Deposition potential (V)	-1.15
Deposition time (s)	90
Equilibration time (s)	10
Start potential (V)	-1.15
End potential (V)	-0.7999
Pulse amplitude (V)	0.05005
Pulse time (s)	0.04
Voltage step (V)	0.005951
Voltage step time (s)	0.1
Sweep rate (V/s)	0.0595
Potential peak (V)	-0.98

4.5.5 Voltammetry determination of zinc in deionised water

10 mL of deionised water was put into the electrochemical cell and 1 mL of acetate buffer with pH 4.6 was added. The measurement cycle used is the same as the parameters as listed in Table 4.2 that used for sample analysis.

CHAPTER 5

RESULTS

5.1 The concentration of Zn in soils for sample 1 – 10

From the voltammetric determination of zinc by using the parameters listed in Table 4.2, the results show that the amount of zinc content were varied in different types of soil. The results are shown in Table 5.1.

Table 5.1: Results for determination of zinc in soils and deionised water

Samples	Reading	Concentration (ppm)	Dilution factor	Actual concentration ([Zn] x dilution factor - [Zn] in dH_2O) (ppm)	Mean concentration (ppm)	Standard deviation (n=3)
1 (commercial)	1	0.02	400x	8.38	7.99	0.43
	2	0.02	400x	8.06		
	3	0.02	400x	7.53		
2 (paddy field)	1	0.01	400x	4.97	4.69	0.27
	2	0.01	400x	4.44		
	3	0.01	400x	4.65		
3 (USM's hostel)	1	0.02	400x	8.23	7.98	0.39
	2	0.02	400x	7.54		
	3	0.02	400x	8.18		
4 (muddy)	1	0.02	400x	7.59	7.29	0.28
	2	0.02	400x	7.03		
	3	0.02	400x	7.26		

5 (burned)	1	0.15	400x	59.63	57.47	3.62
	2	0.15	400x	59.49		
	3	0.13	400x	53.29		
6 (underground)	1	0.01	400x	3.18	2.95	0.28
	2	0.01	400x	3.04		
	3	0.01	400x	2.64		
7 (beach)	1	0.01	400x	4.05	3.95	0.27
	2	0.01	400x	3.65		
	3	0.01	400x	4.16		
8 (batik)	1	0.07	400x	29.51	29.17	0.31
	2	0.07	400x	28.91		
	3	0.07	400x	29.09		
9 (agricultural)	1	0.04	400x	17.20	17.62	0.77
	2	0.05	400x	18.50		
	3	0.04	400x	17.16		
10 (river)	1	0.02	400x	6.50	4.99	1.36
	2	0.01	400x	4.65		
	3	0.01	400x	3.84		
Deionised water	1	0.004	-	-	0.004	-
	2	0.005				
	3	0.003				

5.2 The voltammogram and the calibration curve

The amount of Zn have been determined based on the internal standard addition method and the internal standard addition calibration curve that obtained from the analysis. The voltammogram and the calibration curve of each sample are shown in Figure 5.1 to Figure 5.11.

5.2.1 Sample 1

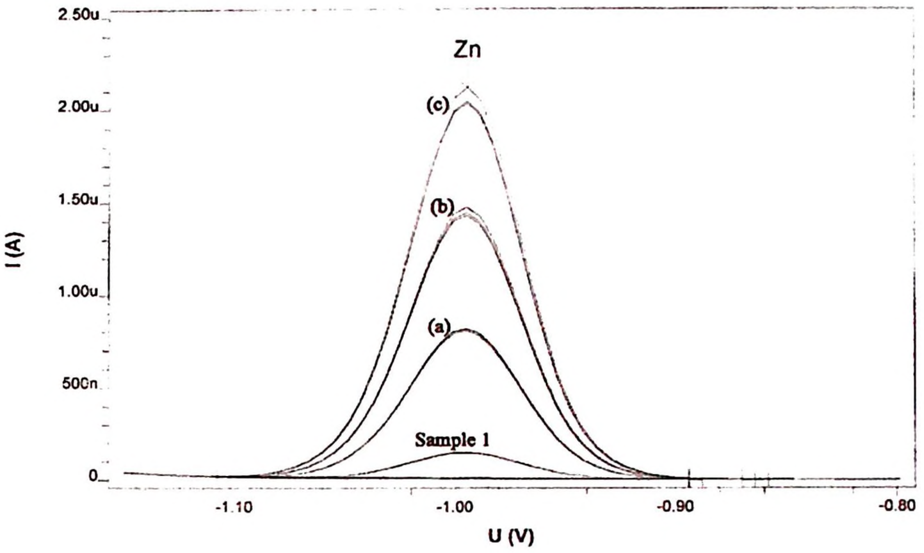
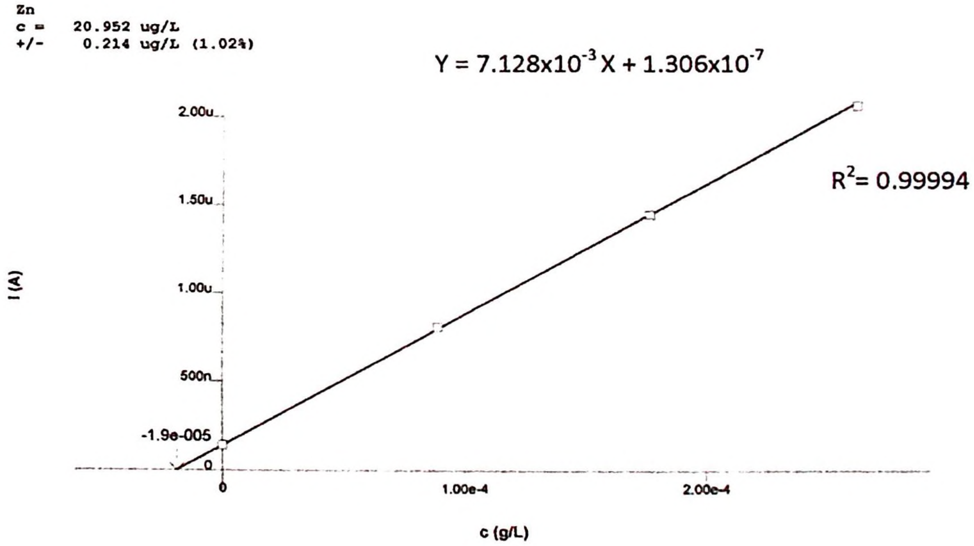


Figure 5.1(i): Voltammogram of Sample 1 with internal Zn standard addition

(a): standard addition 1; (b): standard addition 2; (c): standard addition 3



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Zn
c =    20.952 ug/L
+/-     0.214 ug/L (1.02%)
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Figure 5.1(ii): Internal standard addition calibration curve

5.2.2 Sample 2

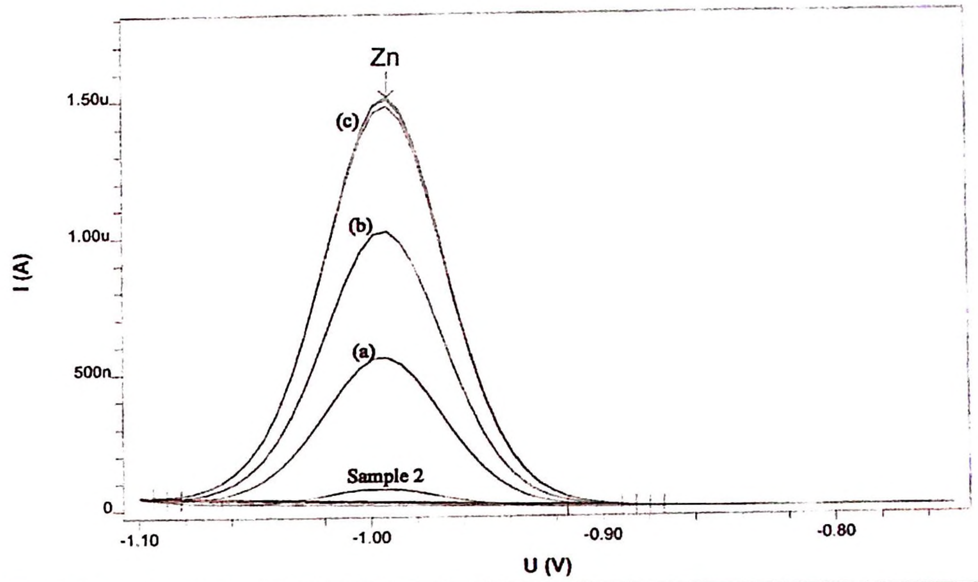
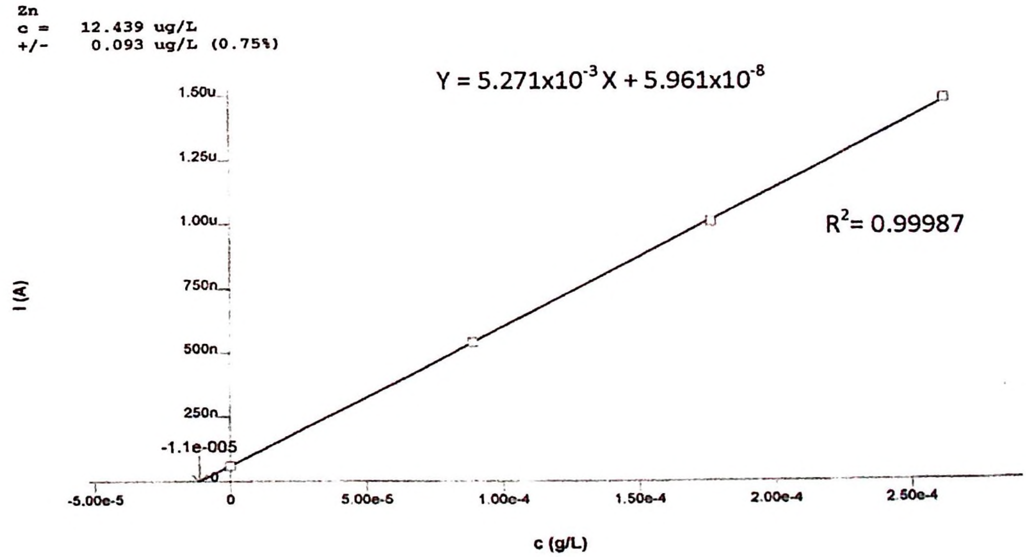


Figure 5.2(i): Voltammogram of Sample 2 with internal Zn standard addition

(a): standard addition 1; (b): standard addition 2; (c): standard addition 3



```
Zn
c =    12.439 ug/L
+/-     0.093 ug/L (0.75%)
```

Figure 5.2(ii): Internal standard addition calibration curve

5.2.3 Sample 3

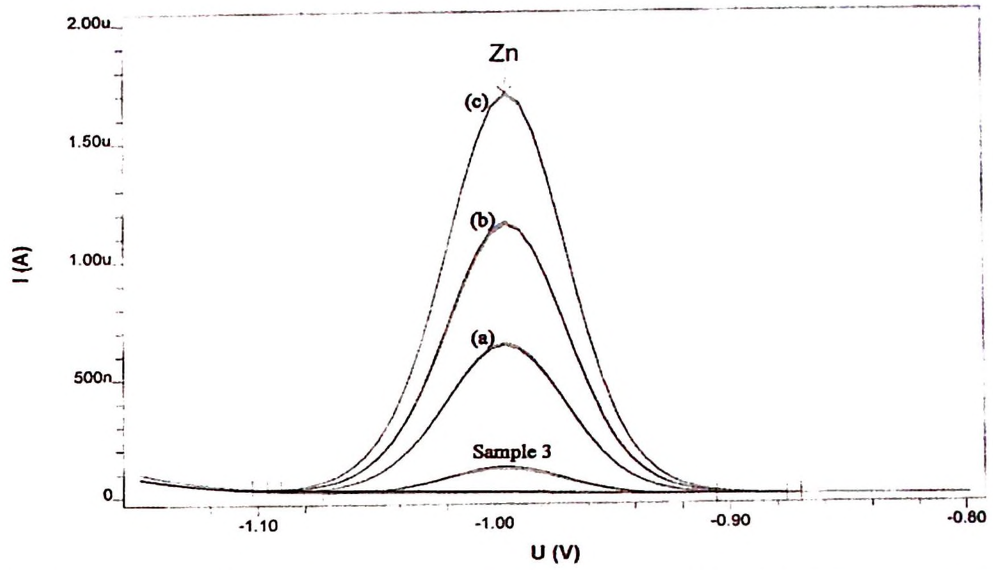
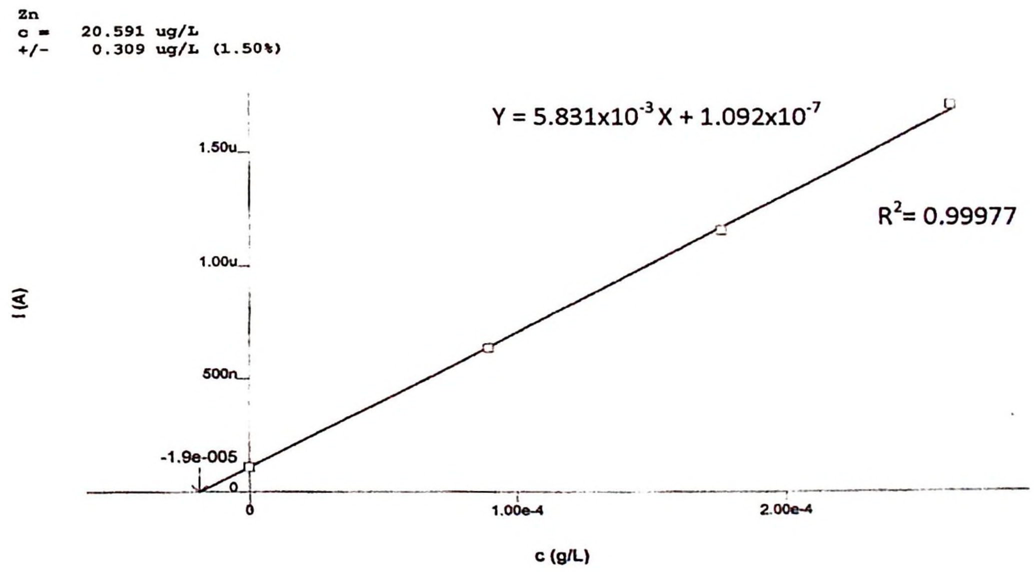


Figure 5.3(i): Voltammogram of Sample 3 with internal Zn standard addition

(a): standard addition 1; (b): standard addition 2; (c): standard addition 3



```
Zn
c =    20.591 ug/L
+/-     0.309 ug/L  (1.50%)
```

Figure 5.3(ii): Internal standard addition calibration curve

5.2.4 Sample 4

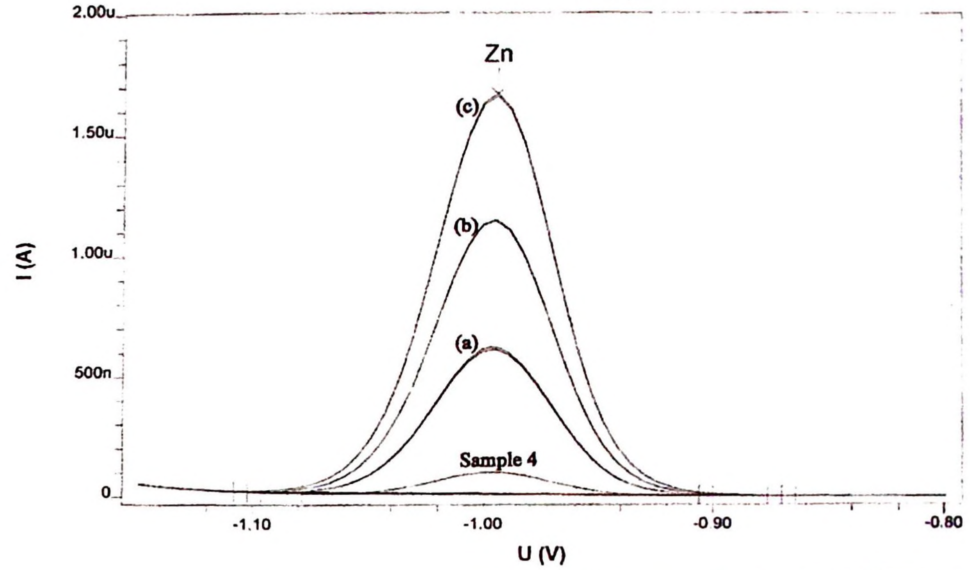
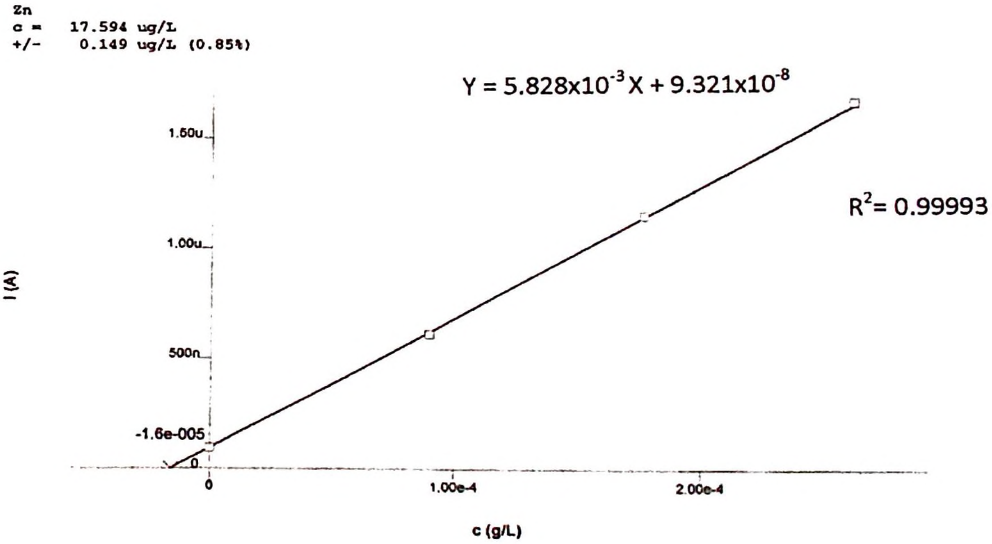


Figure 5.4(i): Voltammogram of Sample 4 with internal Zn standard addition

(a): standard addition 1; (b): standard addition 2; (c): standard addition 3



```
Zn
c =    17.594 ug/L
+/-     0.149 ug/L (0.85%)
```

Figure 5.4(ii): Internal standard addition calibration curve