DETERMINATION OF HEAVY METAL CONCENTRATION IN TOP SURFACE AND SUB SURFACE SOIL AROUND ULU TUALANG CLOSED LANDFILL, TEMERLOH PAHANG

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JUDUL: DETERMINATION OF HEAVY METAL CONCENTRATION IN TOP SURFACE AND SUB SURFACE SOIL AROUND ULU TUALANG CLOSED LANDFILL, TEMERLOH PAHANG

SESI PENGAJIAN: 2010/2011

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DETERMINATION OF HEAVY METAL CONCENTRATION IN TOP SURFACE AND SUB SURFACE SOIL AROUND ULU TUALANG CLOSED LANDFILL, TEMERLOH PAHANG

AHMAD KHUSAIRI BIN AYOP

A thesis submitted in fulfillment

of the requirements for the award of the Degree of

Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering

Universiti Malaysia Pahang

NOVEMBER 2010

“I hereby declare that I have read this thesis and in my/our opinion this thesis has fulfilled the qualities and requirements for the award of Degree of Bachelor of Chemical Engineering”

Signature : …………………………

Name of Supervisor : MR. AZIZAN BIN RAMLI
Date : 3 December 2010

I declare that this thesis entitled “D*etermination of Heavy Metal Concentration in Top Surface and Sub Surface Soil around Ulu Tualang Closed Landfill, Temerloh Pahang*” is the result of my own research except as cited in references. The thesis has not been accepted for any degree and it is not concurrently submitted in candidature of any degree.

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Date : 03 DECEMBER 2010

“

Allah! There is no God but Him;
The living, the Self-subsisting, the Eternal.
No slumber can seize Him, nor sleep.
All things in heaven and earth are His.
Who could intercede in His presence without His permission?
He knows what appears in front of and behind His creatures.
Nor can they encompass any knowledge of Him except what he will.
His throne extends over the heavens and the earth,
and He feels no fatigue in guarding and preserving them,
for He is the Highest and Most Exalted.

”

(Surah Al-Baqarah: 255)

Special Dedication to my beloved mother (Paridah Bt Hj. Mat Isa) and father (Ayop Bin Awang), for their love and encouragement.

And,

Special Thanks to my friends, my fellow course mates and all faculty members.

For all your care, support and best wishes.

Sincerely,
Ahmad Khusairi Bn Ayop

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Last but not least, to my family members. Thank you.

ABSTRACT

There are variety type of heavy metal may contain in the soil at the landfill. Heavy metal is the metallic chemical element that has a relatively high density and it is toxic and poisonous at low concentrations. The objectives of this research are to determine and analyses the distribution of heavy metal that present in the soil obtained from newly-closed landfill at Ulu Tualang Temerloh and to determine which heavy metal have higher amount of concentration in sub and top surfaces of the soil and also to compare it with the Environmental Protection Agency (EPA) Compost Standard. Heavy metal that will be analyses is zinc (Zn) and lead (Pb). For this experiment, after we pulverized and sieved the sample, we extract the sample using Acid Digestion Method, then both Zn and Pb concentration in the sample is determined using Atomic Absorption Spectroscopy (AAS). The result obtained is concentration of Pb in the soil is in range 5.65 mg/kg to 60.6 mg/kg while concentration range of Zn is between range 0.761 mg/kg to 16.365 mg/kg. As a conclusion, for overall the concentration of Pb is highest compared to Zn in top and sub surface of the soil and this heavy metal distribution data is under EPA Compost Standard (Zn and Pb) and also is very useful to be a good reference and guideline for soil remediation.

ABSTRAK

Terdapat pelbagai jenis logam berat yang mungkin terkandung di dalam tanah. Logam berat adalah unsur kimia logam yang mempunyai kepadatan relatif yang tinggi dan ianyatoksik dan beracun pada kepekatan rendah. Objektif kajian ini adalah untuk menentukan dan menganalisis pembahagian logam berat yang terkandung di dalam tanah yang diperolehi dari tempat pelupusan sampah di Ulu Tualang Temerloh dan untuk menentukan logam berat manakah yang mempunyai jumlah kepekatan yang lebih tinggi pada permukaan atas dan di dalam tanah serta untuk membandingkannya dengan Environmental Protection Agency (EPA) Kompos Standard. Logam berat yang akan analisis adalah Zink (Zn) dan Plumbum (Pb). Di dalam eksperimen ini, setelah sampel ditumbuk dan diayak, sampel kemudiannya diekstrak menggunakan kaedah Pencernaan Asid, seterusnya kepekatan Zn dan Pb di dalam sampel di tentukan denagn mengunakan Atomic Absorption Spektrometer (AAS). Keputusan yang diperoleh adalah konsentrasi Pb di dalam tanah adalah di dalam julat 5.65 mg/kg to 60.6 mg/kg manakala julat kepekatan Zn adalah antara 0.761 mg / L menjadi 16.365 mg / L. Sebagai kesimpulan, secara keseluruhannya kepekatan Pb lebih tinggi berbanding dengan Zn pada permukaan dan di dalam tanah dan data pembahagian logam berat ini adalah di bawah EPA Kompos Standard (Zn dan Pb) dan ianya juga sangat berguna untuk menjadi rujukan yang baik dan panduan untuk perbaikan tanah.

TABLE OF CONTENTS

2 **LITERATURE REVIEW**

3 **METHODOLOGY**

4 **RESULTS AND DISCUSSIONS**

5 **CONCLUSION AND RECOMMENDATION**

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

LIST OF ABBREVIATIONS

AAS	-	Atomic Adsorption Spectrometer
Zn	-	Zinc
HCL	-	Hydrochloric Acid
Ppm	-	Part per million
HNO_3	-	Nitric Acid
Cr	-	Chromium
Cu	-	Cuprum
Pb	-	Lead
M	-	Concentration
V	-	Volume
EPA	-	Environmental Protection Agency
MSW	-	Municipal Solid Waste
HWL	-	Hazardous Waste Landfill
PCDD/Fs	-	polychlorinated dibenzo-p-dioxins and dibenzofurans
Cd	-	Cadmium
Cr	-	Chromium
Cu	-	Copper
Ni	-	Nickel
Fe	-	Ferum
Mn	-	Manganese
Al	-	Aluminum

B - Barium
K - Pottasium
Na - Natrium
BOD - Bio-oxygen demand
COD - Chemical oxygen demand
TCLP - Toxicity Characteristics Leaching Procedure
Hg - Mercury
As - Arsenic
USEPA - United State Environmental Agency Protection
OM - Organic Matter
NAA - Neutron Activation Analysis
NO_2 - Nitrous Dioxide
EU PEL - European permissible exposure limit
CCME - Canadian Council of Ministers of the Environment

CHAPTER 1

INTRODUCTION

1.1 Research Background

Nowadays landfill has accepted as a reasonable and widely useful technology that uses at most of developing country. Beside it can accommodate large amount of waste disposal, it also give low investment and operational cost (Chai Xiaoli *et al.*, 2007). But for a certain time, landfill also is a major factor that gives bad effect to the environmental pollution. The soil at the landfill may become toxic and poison because of presence of higher concentration of heavy metal (M.S Li *et al.*, (2007)). Even though there is some metal that good to the soil which is may come from dumping waste, (such as nitrate that can act as fertilizer to the soil), but most of the soil will accepted more type of dangerous heavy metal in the soil that will lead to the negative effects. This situation will make the soil have more dangerous and toxic heavy metal rather than good heavy metal.

After a long time, waste that decomposed at the landfill will make the soil become polluted and may contain large amount of heavy metal. There are some of heavy metal can react with rain rich oxygen to form sulphur and then produces macromolecule of sulphides. These hazardous molecules will influences the quality of atmosphere thus may affect health and life of animal and human being that closed to the landfill. Usually after the landfill is closed, that placed is usually reconstructed as recreational places or residential area. A lot of problems will occur if the soil becomes porous and not stable because of the presence of heavy metal in the soil. Even though there are some heavy metal that may good for the soil, but most of heavy metal that come from industrial waste are toxic and dangerous to human. This issues then will make the reconstructed area are not safe and not suitable for human lives.

1.2 Problem Statement

The dangerous possibility may occur such as contamination of the crop (in term of heavy metal uptake by the plant), pollution of underground water, influence the plant growth and make the soil generally considered toxic(Peter Ryser *et al.,* 2006). In agricultural activity, if the soil that contains a large amount of heavy metal is used, the plant cannot grow well and may be intoxicated. It will affect the health of the human or animal when they took the plants as their meals. The critical issues mentioned above should be avoided to make sure that if the soil is used for human activity, it must be safe and not contain toxic heavy metal that may dangerous and would lead to environmental pollution.

1.3 Objectives

The objectives of this result are:

I. Determine and analyses distribution of heavy metal that present in the soil cause by decomposition of waste in the landfill
II. Determine which heavy metal have higher amount of concentration in sub surfaces and top of the soil

1.4 Scope of Study

1.4.1 Area

For this study, the soil sample that use is from closed landfill at Kampung Ulu Tualang Temerloh, Pahang. This landfill is near to the Temerloh Industrial Estate and Mentakab Industrial Park that focusing in stainless steel fabrication and wood base industry.


Closed landfill at Kg. Ulu Tualang

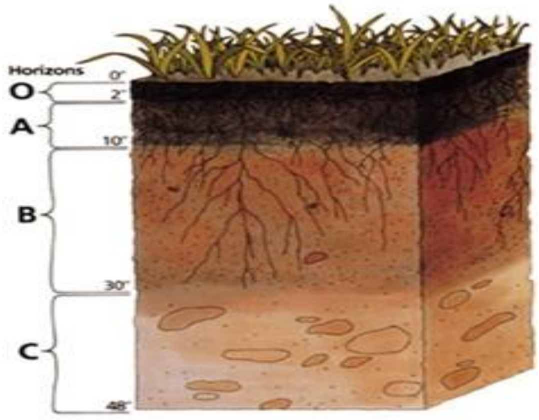
Figure 1.1: Ulu Tualang closed landfill map

The company list that probably using the landfill ;

i. SQ Wooden Picture Frame Moulding Sdn Bhd (Picture Frames - Wholesaler & Manufacturers).
ii. Mentakab Stainless Steel Works (Stainless Steel Fabricators).
iii. Syarikat Perniagaan Boon Wee (Biscuits - Wholesaler & Manufacturers,Food Products).
iv. Megaply Industries (M) Sdn Bhd (Plywood & Veneers).
v. Intan Suria Sdn Bhd (Frames - Picture,Wood Products).
vi. Mentakab Agricultural Machinery Sdn Bhd (Agricultural Equipment & Supplies, Tractor Distributors & Manufacturers).
vii. LCS Precast Sdn Bhd (Piling).

1.4.2 Soil Sample Region

- Soil sample are taken at O and A region



Region A = within depth 2-10 inch

Region B = within depth 10 – 30 inch

1.4.3 Type of Heavy Metal

- Lead is a bluish-white lustrous metal. It is very soft, highly malleable, ductile, and a relatively poor conductor of electricity. It is very resistant to corrosion but tarnishes upon exposure to air. Lead isotopes are the end products of each of the three series of naturally occurring radioactive elements.

- Zinc is a lustrous bluish-white metal. It is found in group IIb of the periodic table. It is brittle and crystalline at ordinary temperatures, but it becomes ductile and malleable when heated between 110°C and 150°C. It is a fairly reactive metal that will combine with oxygen and other non-metals, and will react with dilute acids to release hydrogen.

1.4.4 Method

In this experiment, we divide the experiment into three phases which is sampling and pre-treatment stage, sample digestion or extraction stage using Acid Digestion Method and analysis of the sample by using Atomic Absorption Spectrometer (AAS) in order to determine the concentration of heavy metal in plant and also soil.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Nowadays there are many developing countries that use landfill to provide a place for waste disposal management. A research done by Chai Xiaoli *et al.,* (2007), it shows that more than 90% refused are disposed in landfill at China but this developing country has recently closed more than 1000 landfills because of environmental concern. An example from Chai Xiaoli *et al.,* (2007) research, they state that after the largest landfill in China, Shanghai Laogang Refuse Landfill that constructed in 1985 along the shore of the East China Sea was closed after handling and treats about 30 million tones of refused up to 2005, they preferred Municipal Solid Waste (MSW) landfill to replaced previous landfill because of its offers potential economic benefits by creates additional disposal capacity for the placement of fresh refuse, and recycles the stabilized, valuable resource. This is important for promoting the sustainable environmental development of a society. But before the reclamation of a landfill can be implemented and the residues can be reused outside the landfill environment, it is necessary to comprehensively characterize heavy metals in the landfill. By analyzing heavy metal content in soil reclaimed from an MSW landfill, we can assess the possible limitation of reuse of the reclaimed soil in the landfill and also avoid the environmental pollution effect that may comes because of heavy metal contamination in that soil .

2.2 Effect of Heavy Metals

A research done by M.S Li *et al.,* (2007) also done in China, large amount of heavy metal also found in Guangxi, South China cause by mining process and decomposition of coal in landfill soil. Mining, in particular the metal ore extraction is the second source of heavy metal (especially manganese) contamination in soil after sewage sludge. Even thought there are some perception that told manganese are non-toxic metal element, but the fact is exposure too much to manganese may cause health damage such as Parkinson-like symptoms. Manganese ore also can accompany with other heavy metal mixture such as Pb, Zn, Cd, Ni, Co and Fe and extraction of this combine element will lead to release of more toxic metals into the environment.

2.3 Previous Research / Methodology

In Catalonia, Spain, in Montse Mari *et al.,* (2009) research, they collect information from European Companion and found that 1.3 billion tons of waste including 36 million tonnes hazardous waste was produced there. It may come from different origins such as households, commercial activities and industry and they must be properly managed to minimize their impact on the environment and human health. Their research was done is in Castellolí (Barcelona Province, Catalonia) and this place is classified as class III hazardous waste landfill (HWL). Heavy metal contained there may come from hazardous material such municipal waste incinerator fly ashes, asbestos, as well as other inert wastes. Fly ashes are characterized by a high content of heavy metals and organic compounds such as polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/Fs). In their research, they concern about the human risk health that been living around the HWL area so that the analyze soil and air condition at the landfill. They found relatively higher concentrations of PCDD/Fs, as well as some metals such as Pb, in soil samples of the HWL of Castellolí and also soil samples at the villages located in the vicinity than in those obtained in samples collected from the other places. So that they finally conclude this HWL area is did not safe for human population but in term of environmental issue, this pollution matter should be monitored well in order to avoid contamination happen there will not experiencing and show an increasing trend.

In this new millennium world, there are more research and technique that use to study and analyze presence of heavy metal in the soil. In a research done by A.Kasassi *et al.,* (2008), they found that extensive and long industrial operation that done in landfill site at Efkarpia, Thessaloniki gave higher concentration amount of toxic and dangerous heavy metal to the soil which is cadmium (Cd), chromium (Cr), copper (Cu,) nickel (Ni), lead (Pb) and zinc (Zn). Most of the soil is obtained from deeper site of the landfill, it shows that the waste is decomposed for a long time and it reaches the deeper site in the soil. The higher value of heavy metal found is in depth over 2.5 m. The soil samples collected from this area, which is one of the largest industrial areas where metal-processing, chemical, furniture and wood, textiles and clothing, food, chemical and cement industries are located there. The related produced wastes are 240, 220, 200, 150, 110 and 40 tons/year respectively. For the method to collect and prepared the samples, they drill the site in range of 2.5 m to 17.5 m. Then all samples were stored in a covered place and left to dry out. They were pulverized and sorted all the samples with a 2-mm sieve. For the extraction method, 2 g of each pulverized sample were weighed and placed in conical bottles of 100 or 200 mL. After the addition of 25 mL of HNO3 4 M, the bottle was placed in a sand bath; funnels with filters were placed in the neck of the bottle, in order for the digestion to take place under constant volume. The solution was heated in 70 – 80 ^{o}C for 16 h. After heating, the sample was filtered in a volumetric bottle of 50 mL. Finally, the bottle was filled up to the slit with deionized water. The samples were refrigerated for maintenance (4 ^{o}C). This method was applied to all 40 samples as well and finally analyzed by Atomic Absorption Spectrometer (AAS).

Same thing state by research of M. Ostman *et al.,* (2006), they found that heavy metal at Sweden landfill are dissolved as organic metal and it transported to downward of the landfill. The landfill studied is situated at Hogbytorp, 40 km northwest of Stockholm, Sweden. Wastes that come to this landfill can be classified into five main classes; building and construction waste, household waste, sludge, filling material (inert material) and also ash and slags. This landfill starts it operation in 1964 and the closure is estimated to be in 2005, when the wastes contain approximately 4 million tons of waste. From its starting operation at 1964 to the beginning of 1983 the major proportion of the waste was household waste, but from 1984 building and construction waste began to dominate the waste. For the collecting the samples in this landfill site, they drilled the landfill until depth of 18 m. Then all the samples have been drying follow next steps which is by crushing and sieving. Larger metal items were removed from the sample including gravel and stones. The samples then were carefully mixed. For the analyses of heavy metals, all samples was extracted using nitric acid soluble speciation, which was chosen to get an estimate of the metal content as near as possible to the total content in the material. Silicate minerals are not soluble in nitric acid, so for Al, B, K and Na only a small amount of the total content was dissolved in the nitric acid, but for the rest of the elements analyzed, approximately about 70–90% of total content was obtained. At the end of their research, they confirm and conclude that soils from this landfill area contain large number of heavy metals. This condition makes the stability of the landfill difficult to predict. Furthermore, they also assumed that some of the metals can bounds with sulphur element and other organic materials in the macromolecules. When it reacts with oxygen-rain water, these elements will release from both organic material and sulphides which is a very hazardous material. It then makes the area of the landfill and nearest area there polluted, did not safe for living and this environmental issue must be handle well regard to the fate of metals present there.

Most of the researcher has done drilling method in order to take the sample. In a research done by L. Borgna *et al.,* (2009), they take sample from top surface of the soil until depth of 20cm. For the extraction method, we use acid digestion of sediments, sludge's, and soils method. This method is a very strong acid digestion method that wills dissolves almost of elements that could become environmentally available. It also don't have higher operation cost. The need for accurate and precise analyses of toxic metals is becoming important aspect, as we learn more about their behavior and causes if they're in the environment and in the food chain. The harmful effects of many of these metals to man and nature are well known while the number of elements that to be determined continues to increase, and also in a demand for low detection limits. According to research done by Timo Myöhänen *et al.*, (2002), they use another type of digestion method which is aqua-regia digestion method. This method is quite popular method that always uses to determine concentration of heavy metal., aqua-regia method give shorten time for analyzing method. But, in term of precision, it cannot determine concentration of heavy metal accurately if we want proceed to last method using atomic absorption spectroscopy (AAS).

2.4 Previous Research / Result and Discussion

In a research done by A.F Al-Yaqout *et al.*, (2003), they compared the soil contamination at the leachate landfill from developed country which is USA, Germany and Italy in term of amount of pH, bio-oxygen demand (BOD), chemical oxygen demand (COD) and distribution of heavy metal with their country.

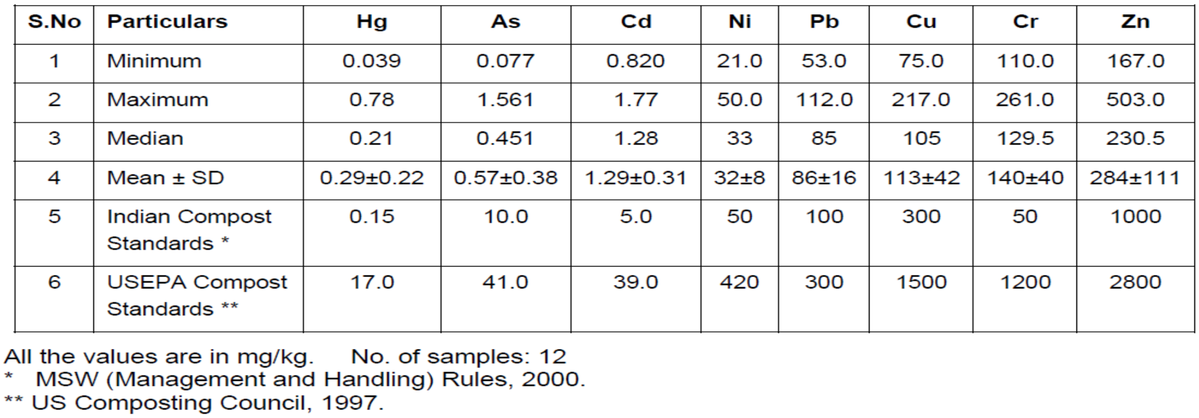
Table 2.1: Comparison of leachate quality of Kuwait (Al-Sulaybiya) with other leachates reported for developed countries

Parameter (mg/l)	Kuwait	USA	Italy	Germany
Alkalinity (as CaCO3)	250–6340	240– 8965	4250– 8250	-
pH	6.9– 8.2	5.1–6.9	6.0–8.5	5.7– 8.1
BOD	30– 600	13,400	2125– 10,400	400–45,900
COD	157.9–9440	1340– 18,100	7750– 38,520	1630–63,700
Sulfate	55– 3650	0.01– 1280	219–1860	1– 121
Zn	0.0– 4.8	18.8– 67	5 – 7	-
Pb	0– 0.2	0 –4.46	–	–
Cu	0– 0.2	0 –0.1	–	–
Fe	0.3– 54.1	4.2–1185	47–330	8– 79
Ca	5.6– 122	254.1– 2300	0 – 175	70– 290
Mg	5.2– 268	233– 410	827–1469	100–270

From the table, it show that developed country produce higher amount of heavy metal thus lead to more environmental pollution. This is due to the amount of waste produce is higher from this four developing country.

The Ulu Tualang closed landfill gets most of waste disposal similiar with research done by S. Esakku *et al.*, (2003). The dumping site at Chennai India get the unused waste about 3500 tons/day such as electronic goods, electro plating waste, painting waste, used batteries and this various waste sources may come from developed such as USA, Singapore and South Korea. It will lead to the environmental issue matter if unorganized dumping of solid waste management authorized there. It also will cause serious health damaged due to the released of toxic heavy material that comes from this landfill. People that exposed to this environment can be affected to their health such as blood and bones disorder, kidney damage, decreased of mental capacity and also damage the neurological systems. In their research, for solid waste sampling procedures, they drilled about 3m depth in the landfill to get the sample. The sample then was analyzed by using water extract method, Toxicity Characteristics Leaching Procedure (TCLP) and also aqua reqia acid digestion method. As a result, they found that the soil have higher concentration of zinc compare than other heavy metals (Hg, As, Cd, Ni, Pb, Cu and Cr) and the heavy metal distribution was found most at top surface of the soil within depth 1-2 meter. The summary of the distribution data can be explained in Table 2.1.

Table 2.2: Summary of statistics of heavy metal that done in Chennai India



S.No	Particulars	Hg	As	Cd	Ni	Pb	Cu	Cr	Zn
1	Minimum	0.039	0.077	0.820	21.0	53.0	75.0	110.0	167.0
2	Maximum	0.78	1.561	1.77	50.0	112.0	217.0	261.0	503.0
3	Median	0.21	0.451	1.28	33	85	105	129.5	230.5
4	Mean ± SD	0.29±0.22	0.57±0.38	1.29±0.31	32±8	86±16	113±42	140±40	284±111
5	Indian Compost Standards *	0.15	10.0	5.0	50	100	300	50	1000
6	USEPA Compost Standards **	17.0	41.0	39.0	420	300	1500	1200	2800

All the values are in mg/kg. No. of samples: 12

* MSW (Management and Handling) Rules, 2000.

** US Composting Council, 1997.

From the table, we can see that from their research heavy metals that was found did exceed the India Compost Standard but when comparing with United State Environmental Agency Protection (USEPA) standard, all are them is within the standard limits. Another research done by Abdelatif Mukhtar Ahmed *et al.,* (2001), they compared the concentration of heavy metal that found in Seri Petaling landfill with USA, Poland and China landfill. According to them, concentration of heavy metal in landfill at Malaysia is quite low compare than the other country. It proves that as a developing country, Malaysia produced less waste disposal compare than the other 1st World Country. Besides that, a research done also in Malaysia done by Md. Mizanur Rahman *et al.*, (1997), they found that industrial places such as Taman Beringin landfill produced more waste disposal then lead to decomposition of variable heavy metals in the soil compare than the other places.

Heavy metal accumulation in soils makes the soils becomes loss of its functions then leading to concerns about environmental quality protection, maintenance of human health and productivity. Soil pollution can have implications in phytotoxicity at high concentrations and as a result in the transfer of heavy metals to the human diet from crop uptake or soil ingestion by grazing livestock. A research done at Yocsina, Córdoba Province Argentina by Gonzalo M.A. Bermudez *et al.,* (2009), they studied about soil located near to the cement plant. Cement plants are important emission sources of pollution of both organic and inorganic chemicals, and it produce a large number of metals and metalloids elements such as As, Cd, Ca, Co, Cr, Cu, Ni, Pb and Zn. They top surface soil sample was collected within the depth 0 – 10 cm in 25 meter per square and within this gridding area, nines sub soil samples was randomly choose. All the samples then was analyzed to determine ph value, organic matter (OM) content and also distribution of heavy metal analysis done by Neutron Activation Analysis (NAA) after extracted by 0.5 M HCl-extractable heavy metals method. This method has been successfully used to get the total non-residual or non-latticeheld heavy metal concentrations in streams, road sediments and also in soils. As a result, they found that most of the heavy metal contains in the soil at the cement plant there and people that live near to this cement plant have high risk to expose to the toxicity of dangerous affects of heavy metal such as Ba and Cr that highly detected in this plant.

2.5 Conclusion

Although the previous literature gave a lot of experimental data with variable technique done, it still has a room to make an improvement in order to achieve perfectness. Data that will be obtained can be more precise and accurate because of latest technology compare to their time. This important aspect should be done continuously time by time so that we can always alert to the dangerous possibility that may comes from the waste dumping site. Therefore, another research study about presence of heavy metal and their quantity in the soil should be done to avoid any unexpected thing that may happen.

In this study, statistical data can be plotted and it will be very useful for landfills treatment and become references for the land developer when they start to reconstruct this closed landfill. Decomposition of this heavy metal may lead to more good effect or vice versa to human lives near to the landfill. It can act either good for fertilizer to the soil or leads to more bad effect that cause environmental pollution. When the soil is detected contain large amount of heavy metal that totally dangerous and contain higher toxicity, it is important to us to make that the soil safe first before reconstructed it for human activity there to avoid any unexpected incident occur.

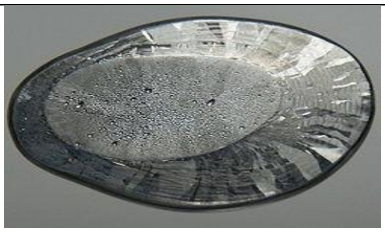
Table 2.3: Information of zinc

General information 	- Lustrous bluish-white metal - brittle and crystalline at ordinary temperatures, but it becomes ductile and malleable when heated between 110°C and 150°C - Reactive metal that will combine with oxygen and other non-metals, it will react with dilute acids to release hydrogen
Application	- Always used in principally for galvanizing iron, in the preparation of certain alloys, used for the negative plates in some electric batteries and for roofing and gutters in building construction, used in die casting in the automobile industry - White pigment (zinc oxide) - used in plastics, cosmetics, photocopier paper, wallpaper, printing inks - Can also act as catalyst during manufacture and as a heat disperser in the final product in rubber production
Health effects of zinc	- can experience a loss of appetite, decreased sense of taste and smell, slow wound healing and skin sores - cause birth defects - high amount of zinc can cause stomach cramps, skin irritations, vomiting, nausea, anemia, damage the pancreas, disturb the protein metabolism and can cause respiratory disorders
Environmental effects of zinc	- increase the acidity of waters - Water-soluble zinc that is located in soils can contaminate groundwater - Decrease the plant survival chances - Interrupts activity of microorganisms in soils

Table 2.4: Information of lead

General information 	- bluish-white lustrous metal - very soft, highly malleable, ductile - poor conductor of electricity - a radioactive elements
Application	- used in piping systems - constituent of the lead-acid battery used extensively in car batteries - used as a coloring element in ceramic glazes - commonly uses is in the glass of computer and television screens, where it shields the viewer from radiation - Other uses - in sheeting, cables, solders, lead crystal glassware, ammunitions, bearings and as weight in sport equipment
Health effects of lead	- - Disruption of the biosynthesis of hemoglobin and anemia, a rise in blood pressure, kidney damage, disruption of nervous systems, brain damage - Declined fertility of men through sperm damage - Diminished learning abilities of children - Behavioral disruptions of children, such as aggression, impulsive behavior and hyperactivity
Environmental effects of lead	- increase the acidity of waters in corrosive pipe - disturb the soil functions by lead intervention - poison the water and soil organisms

Table 2.5: Properties of Zinc and Lead

	Zinc (Zn)	Lead (Pb)
Picture		
Name, symbol, number	zinc, Zn, 30	lead, Pb, 82
Element category, transition metal	Alternatively considered a post-transition metal	post-transition metal
Group, period, block	12, 4, d	14, 6, p
Standard atomic weight	65.38(4) g·mol^{-1}	207.2 g·mol^{-1}
Phase	solid	solid
Melting point	692.68 K, 419.53 °C, 787.15 ° F	600.61 K, 327.46 °C, 621.43 °F
Boiling point	1180 K, 907 °C, 1665 °F	2022 K, 1749 °C, 3180 °F
Oxidation state	+2, +1, 0 (amphoteric oxide)	4, 2, -4 (amphoteric oxide)
Thermal conductivity	(300 K) 116 W·m^{-1}·K^{-1}	(300 K) 35.3 W·m^{-1}·K^{-1}
Toxicity info (in a large amount)	Poison , cause body damage to plant, human and animal, high possibility to cause death	Poison, cause failure of respiratory system, nervous system, cardiovascular system

CHAPTER 3

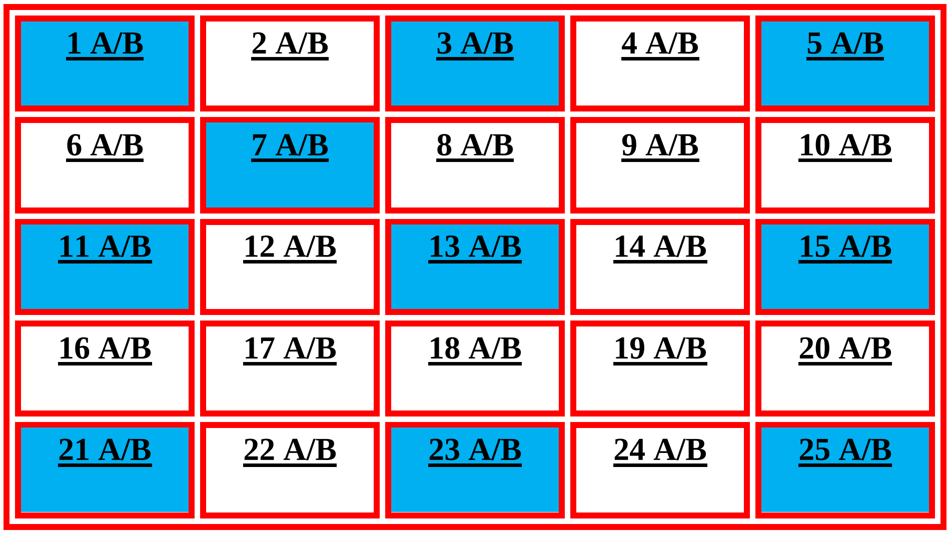
METHODOLOGY

3.1 Procedure

In this experiment, we divide the experiment into three phases which is sampling and pre-treatment stage, sample digestion or extraction stage and analysis of the sample by using Atomic Absorption Spectrometer (AAS). All of these stages will be clearly explained below.

Stage 1 (Sampling and Pre-treatment)

- ✓ Gridding the landfill area (50m x 50m)
- ✓ 25 sampling point was divided within this area
- ✓ Sample collecting from sampling (2 samples for each point)
- ✓ Sample stored and left to dry out
- ✓ Wet samples dried using drying oven
- ✓ Sample pulverized & sieved (2-mm sieve)
- ✓ Mix sample to homogeneity



1 A/B	**2 A/B**	**3 A/B**	**4 A/B**	**5 A/B**
6 A/B	**7 A/B**	**8 A/B**	**9 A/B**	**10 A/B**
11 A/B	**12 A/B**	**13 A/B**	**14 A/B**	**15 A/B**
16 A/B	**17 A/B**	**18 A/B**	**19 A/B**	**20 A/B**
21 A/B	**22 A/B**	**23 A/B**	**24 A/B**	**25 A/B**

Figure 3.1: Sampling Point Gridding Area

Stage 2 (Acid Digestion Method)

This method required 1.00-4.00 g of dried sample to be digested in a mixture of 9 mL of concentrated hydrochloric acid and 3 mL of concentrated acid at ambient air in a 50 mL digestion vessel. This mixture must be prepared in a fume hood due to the volatility of the acids. The sample and reaction mixture were slowly heated to 95°C to prevent an overly vigorous reaction. For the vapor recovery, a watch glass had been used. The digestion was continued until the disappearance of NO_2 (reddish brown) fumes and no more changes in appearance (Kimbrough and Wakakuwa, 1992). The sample must be brought to near dryness. The times taken for the fumes disappear vary due to the type of soil. Most of the samples take 30 minutes to 2 hours to achieve the condition. The digestate is the filtered using AAS grade filter paper (Whatman 41 or equivalent) and collected in a 100 mL volumetric flask. To enhance the filtration rate, a vacuum filter had been used for replacing the conventional filtration method.

Stage 3 (Sample Analysis)

In this stage, after the digestate was filtrated, it will be analyzed using AAS. Before we run the samples, the entire sample must be added with 3 to 5 droplet of nitric acid, HNO_3 and the standard solution must be prepare first. There are 4 standard solutions which is 1 part per million (ppm), 5 pmm, 30 ppm and 100 ppm that must prepared to analyze Zn and Pb element in the sample

- ✓ Filter,make to volume.
- ✓ Add 3 droplet of $HNO_{3.}$
- ✓ Prepare the standard solution.
- ✓ Analyze by AAS.
- ✓ Calculations & Data collection.

3.2 Equipment/Apparatus/Material

In completing the study, some equipment is required. They are:

1. Atomic Absorption Spectroscopy (AAS) unit
2. Beaker
3. Vacuum pump
4. Filter paper Whatman 41
5. Oven
6. Weighing scale
7. Sieve 0 – 2 mm
8. Vial
9. Mortar crusher
10. Measuring cylinder
11. Volumetric flask
12. Dropper
13. Aluminum Foil
14. Hot plate

3.3 Reagents

Reagents or chemical are required. There are:

1. Soil Samples
2. Ultra pure water
3. Distilled water
4. Hydrochloric Acid (HCL)
5. Nitric Acid (HNO_3)

3.4 Preparation of Standard Curve

Calibration or standard curve is a method used to determine the unknown concentration of heavy metal in the wastewater solution by using Atomic Adsorption Spectrophotometer (AAS). AAS is the standard method for the analysis of specific metal. The concentration of single heavy metal such as zinc, Zn in the wastewater can be determined if it occur within the range of standard solution of zinc that we prepared.

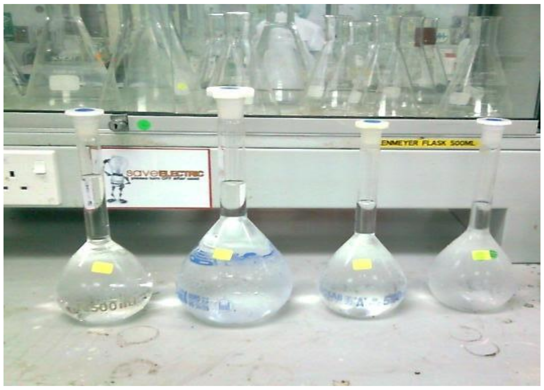


Figure 3.2: Standard solution of zinc, Zn

Standard solution was prepared by dilution of standard zinc (Zn) and nickel (Ni) of 1000ppm into 4 difference concentration of standard solutions. Standard solution of , 1, 5, 30 and 50 ppm for zinc and lead prepared by using 100ml of volumetric flask.

The most accurate formula for making dilutions of solutions is the following:

$$M_1V_1 = M_2V_2$$

M_1= percentage you have

M_2= percentage you want

V_1= unknown volume

V_2= volume wanted

The time estimation for analyses of 10 samples for each specific metal is about 10 minutes using Atomic Adsorption Spectrophotometer (AAS). The graph and the value of specific metal concentration are then print out automatically after the analysis finished.

3.5 AAS Spike Method

- 10 of the sampling point (blue color) was chosen to go through AAS spike method because of negative value get in the 1st test from AAS [Christidis *et. al.* (2007)]

- Spike method procedure

  - Mix 1ml of the sample with 250µL of 1000 ppm stock solution (Pb & Zn) in 50ml volumetric flask
  - Shake well the mixture
  - Analyze samples using AAS

- Spike 5 ppm calculation

  $\mathbf{M_1V_1 = M_2V_2}$

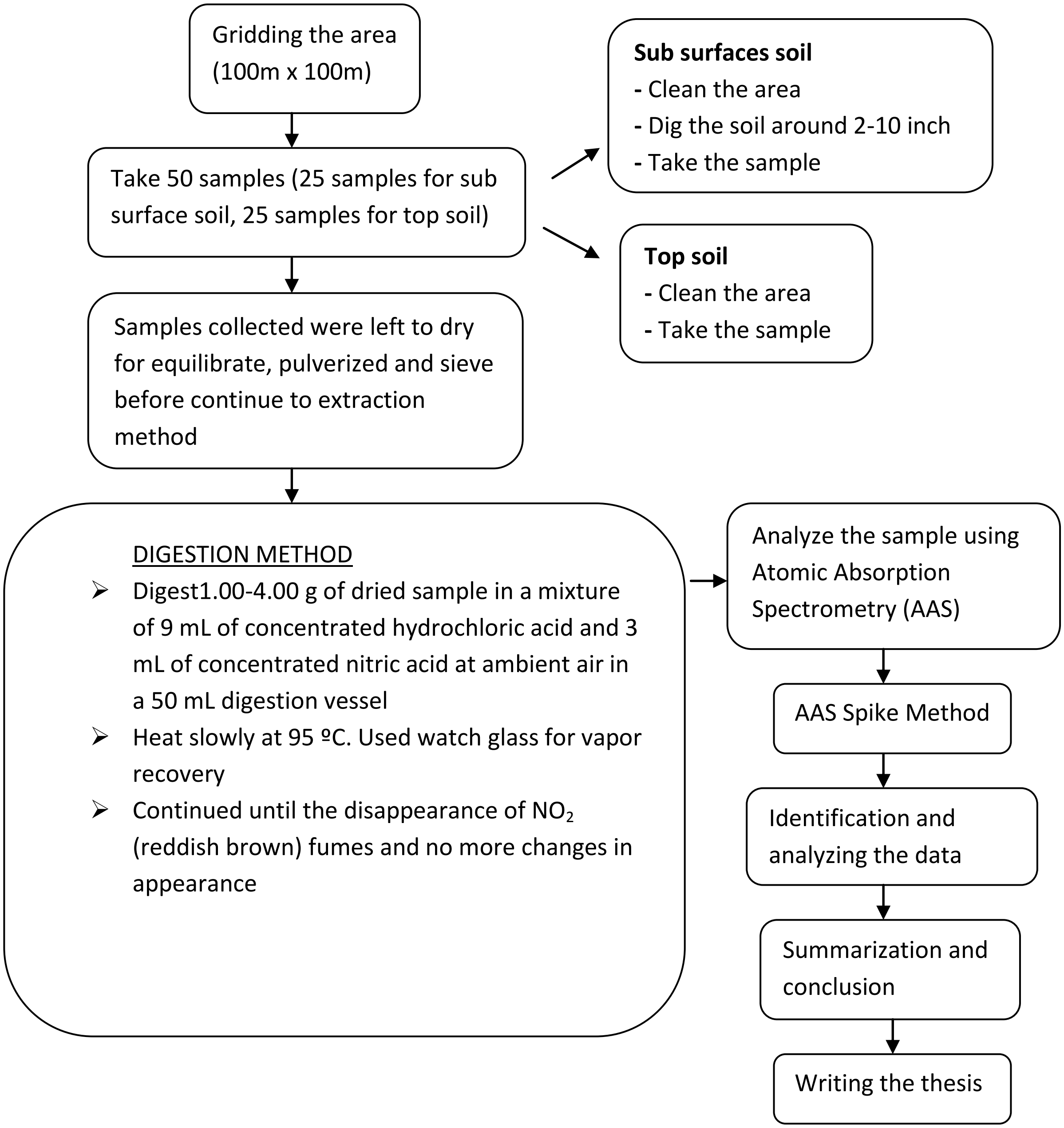
  (1000 ppm) x V = (5 ppm) x (50 ml)

  V = 0.25 ml

  = 250µL

- The value that get from AAS analysis then must be subtract with 5 ppm to get the sample actual concentration value

3.6 Summary of Leads and Zinc Sample Analysis



Gridding the area (100m x 100m)

Take 50 samples (25 samples for sub surface soil, 25 samples for top soil)

Sub surfaces soil
- Clean the area
- Dig the soil around 2-10 inch
- Take the sample

Top soil
- Clean the area
- Take the sample

Samples collected were left to dry for equilibrate, pulverized and sieve before continue to extraction method

DIGESTION METHOD

- Digest1.00-4.00 g of dried sample in a mixture of 9 mL of concentrated hydrochloric acid and 3 mL of concentrated nitric acid at ambient air in a 50 mL digestion vessel
- Heat slowly at 95 ºC. Used watch glass for vapor recovery
- Continued until the disappearance of NO_2 (reddish brown) fumes and no more changes in appearance

Analyze the sample using Atomic Absorption Spectrometry (AAS)

AAS Spike Method

Identification and analyzing the data

Summarization and conclusion

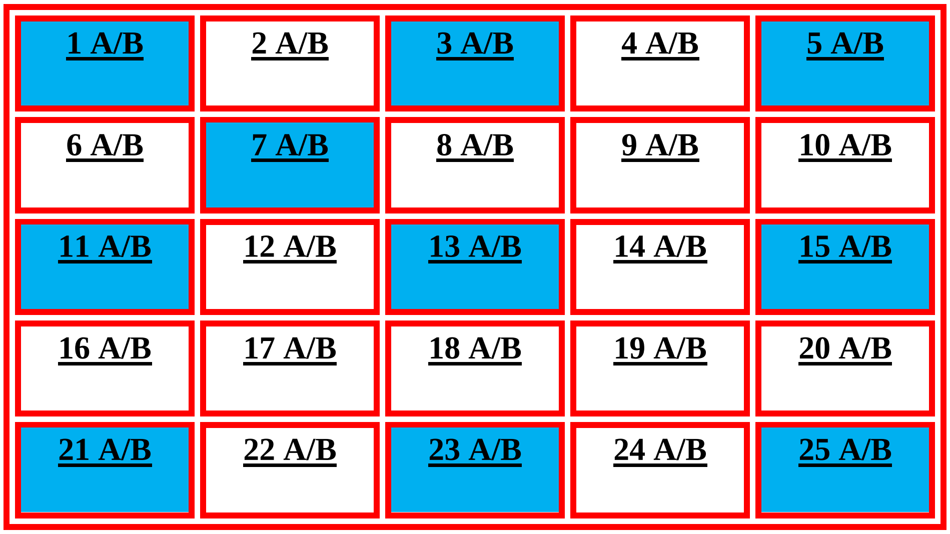
Writing the thesis

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

After the first run testing by AAS, Zn reading by AAS show positives value while for Pb reading, it show the negatives value. In order to get Pb actual amount of sample concentration, the samples must go through AAS spike method. 20 samples from top surface and sub surface soil were chosen randomly (blue one) that scattered through all the gridding area to go through AAS spike method.



1 A/B	2 A/B	3 A/B	4 A/B	5 A/B
6 A/B	7 A/B	8 A/B	9 A/B	10 A/B
11 A/B	12 A/B	13 A/B	14 A/B	15 A/B
16 A/B	17 A/B	18 A/B	19 A/B	20 A/B
21 A/B	22 A/B	23 A/B	24 A/B	25 A/B

Figure 4.1: Sampling Point Gridding Area

Figure 4.2: Sample Collecting

4.2 Spike 5 ppm Method

Procedures

- Dilute1ml of filtered sample + 250 µL of 1000 ppm Pb stock solution in 50 ml volumetric flask. Shake well
- Analyze using AAS

Spike 5 ppm calculation (Pb)

$$(\text{A ppm}) = \frac{\text{A mg}}{\text{1000 L}} \text{ x } \frac{\text{volumetric flask vol}}{\text{weight of sample}} \text{ x } \frac{\text{1000 g}}{\text{1 kg}} \text{ x dilution factor}$$

A = detected value – 5 ppm

$$\text{Dilution factor} = \frac{\text{volumetric flask volume}}{\text{weight of sample}}$$

Eg :

$$(14.37 - 5) \text{ ppm} = \frac{\text{9.37 mg}}{\text{1000 L}} \text{ x } \frac{\text{0.1 L}}{\text{1 g}} \text{ x } \frac{\text{1000 g}}{\text{1 kg}} \text{ x } \frac{\text{50 ml}}{\text{1 g}}$$

$$= 46.85 \text{ mg / kg}$$

4.3 Lead

Table 4.1: Lead (Pb) reading by AAS after Spike Method

Sampling Point	Top Surface concentration (mg / kg)	Sub Surface concentration (mg / kg)
1.	46.85	5.65
3.	54.4	46.45
5.	43.05	50.3
7.	45.4	46.45
11.	51.35	48.9
13.	54.1	53.95
15.	49.1	46.95
21.	47.75	60.6
23.	54.05	56.75
25.	54.75	53.45

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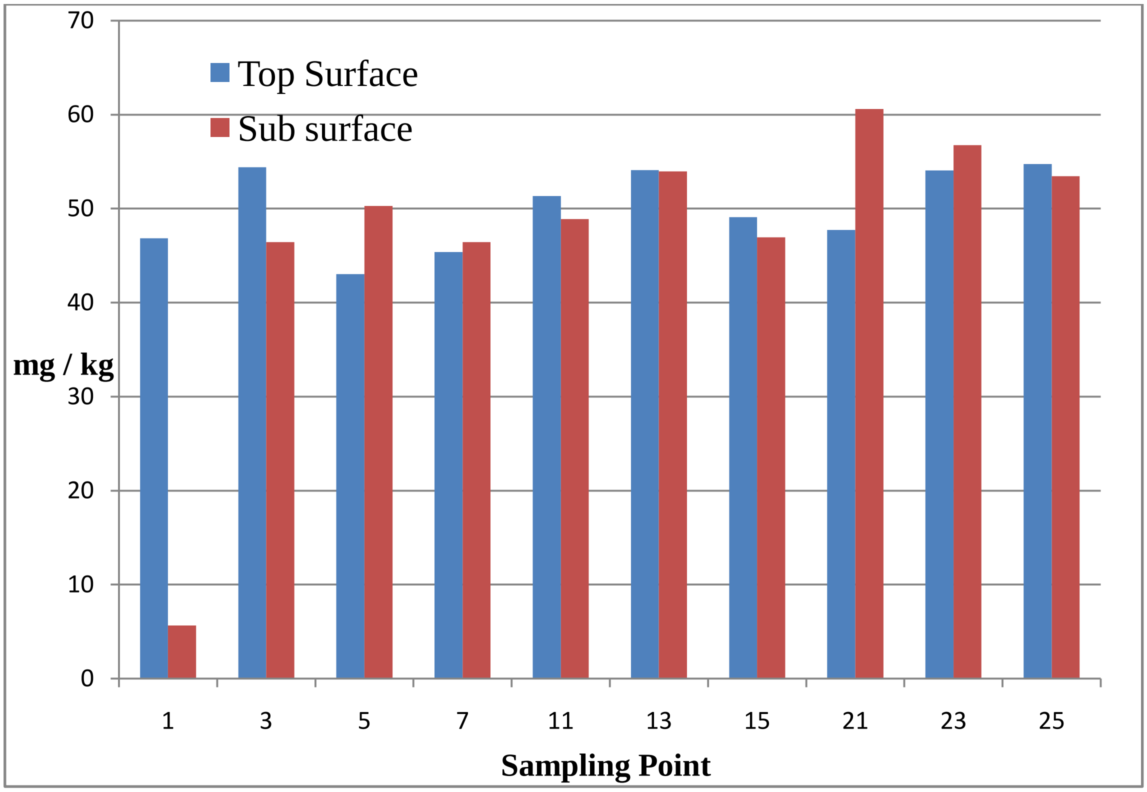

Figure 4.3: Graph of Pb concentration vs sampling point

Table 4.2: Lead concentration data

	Top Soil (mg / kg)	**Sub-surface (mg / kg)**
Minimum	43.05	5.65
Maximum	54.75	60.6
Median	52.725	51.425
Mean	50.08	46.945
EU PEL CCME (Canadian Council of Ministers of the Environment, 1991)	**375**	
US PEL (*Adapted from U.S. EPA, 1993)	**420**	

From the graph, we can see that highest concentration of Pb in sub surface soil at sampling point no. 21 (60.6 mg/kg) and lowest concentration of Pb also in sub surface soil at sampling point no.1 (5.65 mg/kg. For sampling point number 1, 3, 11, 13, 15 and 25, the top soil concentration is higher than the sub surface soil. It can be explained by the metal solubility of the elements (Esakku et al 2003). The pH of the sample that we get is in range 5.4 to 7.20 for top soil and 4.11 to 6.90 for sub surface soil. According to Esakku et al (2003), solubility of heavy metal is affected by pH value of the soil (higher pH will lower the metal solubility due to the precipitation of metals ions). While for sampling point number 5, 7, 21, and 25, the sub surface soil concentration is higher compare than top surface soil. This situation also explained by Esakku et al (2003), where phytoextraction of metals (Pb) attributed by natural vegetation. Other possibility that may give this pattern of data also is because the major part of heavy metals is already going downward deep away in the landfill. There’s only minor part that not yet going downward to the landfill. With this reason, we can see from the data obtained, lead concentration in Ulu Tualang soils that been studied did not exceed the EU permissible exposure limit (PEL) value and US EPA standard limit.

4.4 Zinc

To determine Zn concentration in the collected samples, it did not go through AAS spike method because we obtained the positive value in first test analysis. In order to synchronize the Zn concentration data with Pb concentration data, we also choose 20 samples from same lead sampling point.

Table 4.3: Zinc (Zn) reading by AAS

Sampling Point	Top Surface concentration (mg / kg)	Sub Surface concentration (mg / kg)
1.	8.656	6.076
3.	3.046	2.402
5.	5.69	0.498
7.	10.491	5.909
11.	14.154	16.365
13.	2.022	7.043
15.	7.159	7.513
21.	5.052	6.876
23.	14.138	7.063
25.	6.025	6.873

Zn Calculation

$$(A\ \text{ppm}) = \frac{A\ \text{mg}}{1000\ \text{L}}\ \text{x}\ \frac{\text{volumetric flask vol}}{\text{weight of sample}}\ \text{x}\ \frac{1000\ \text{g}}{1\ \text{kg}}$$

A = detected value

Eg :

$$(86.56)\ \text{ppm} = \frac{86.56\ \text{mg}}{1000\ \text{L}}\ \text{x}\ \frac{0.1\ \text{L}}{1\ \text{g}}\ \text{x}\ \frac{1000\ \text{g}}{1\ \text{kg}}$$

$$= 8.656\ \text{mg / kg}$$

(S. Esakku *et al.*, 2003)

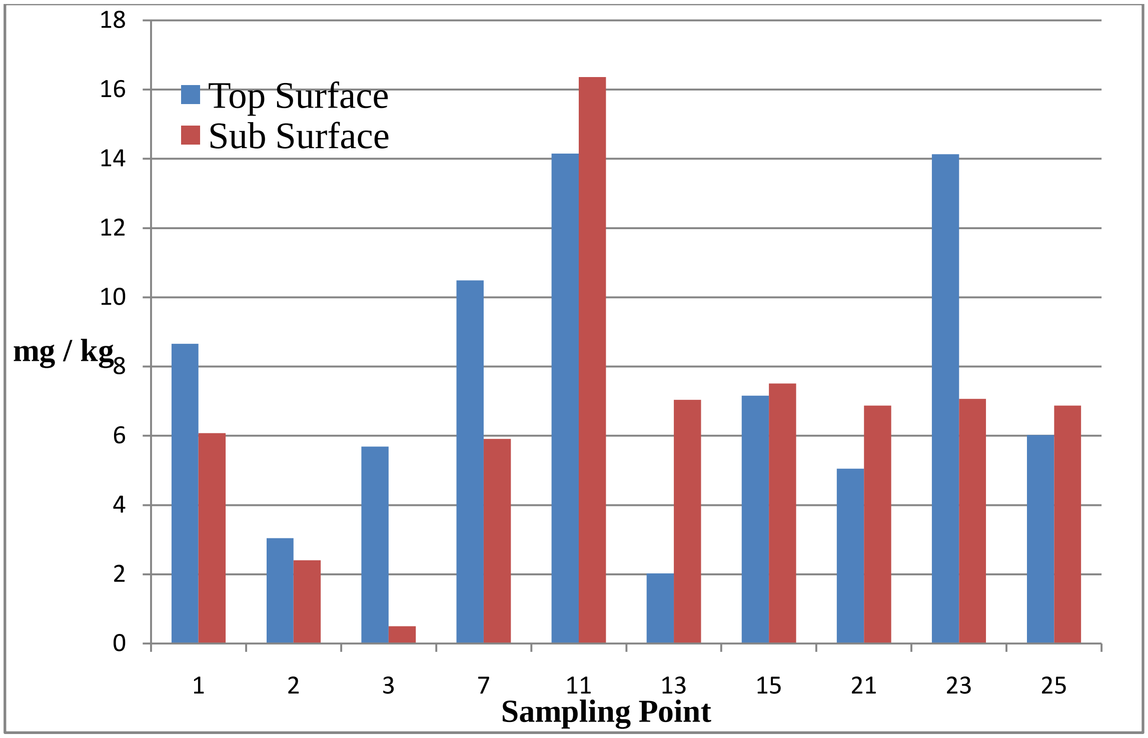

Figure 4.4: Graph of Zn concentration vs sampling point

Table 4.4: Zinc concentration data

	Top Soil (mg / kg)	**Sub-surface (mg / kg)**
Minimum	2.022	0.498
Maximum	14.154	16.365
Median	8.088	11.702
Mean	7.6433	6.6618
EU PEL CCME (Canadian Council of Ministers of the Environment, 1991)	**600**	
US PEL (*Adapted from U.S. EPA, 1993)	**2800**	

The pH value for Zn also same with Pb which is in range 5.4 to 7.20 for top soil and 4.11 to 6.90 for sub surface soil. From the plotted graph above, we can see that at sampling point number 1, 3, 5, 7 and 23 top surface concentration value is higher than sub surface concentration value. While for the rest sampling point (11, 13, 15, 21, and 25) sub surface concentration value is higher than top surface concentration value. This both phenomena were told by previous explanation at lead concentration data above. From the data obtained, when we compare the data with EU permissible exposure limit (PEL) value and US EPA standard limit, it did not exceed this both standard limit.

4.5 Discussion

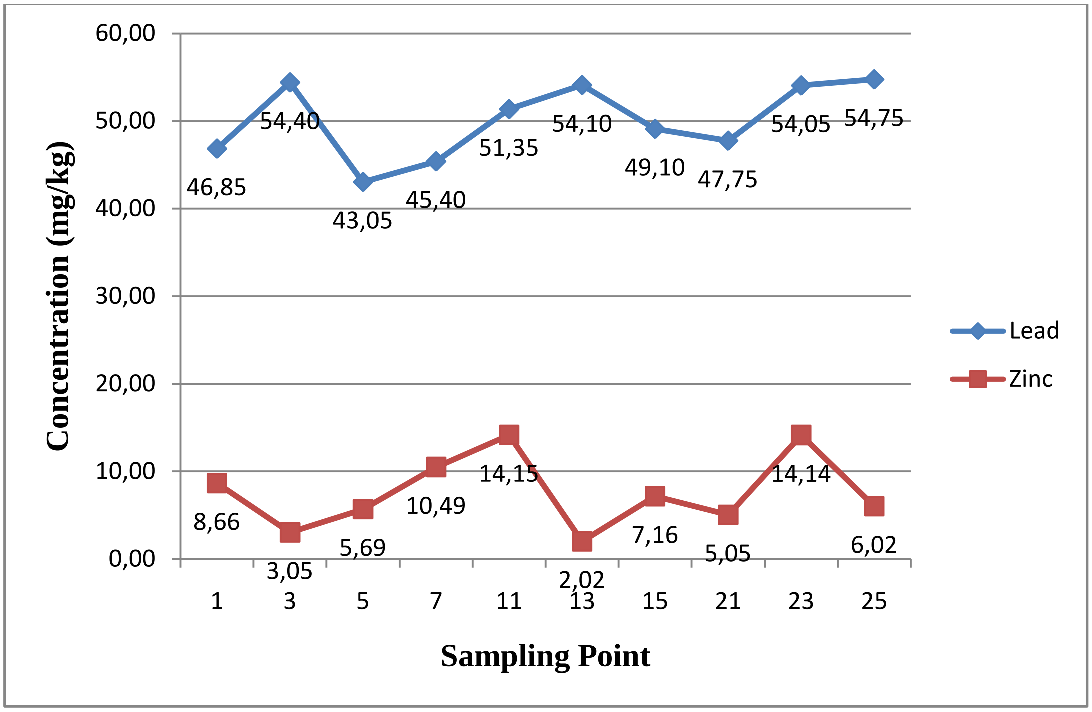

Figure 4.5: Graph of heavy metal concentration vs sampling point in top surface soil

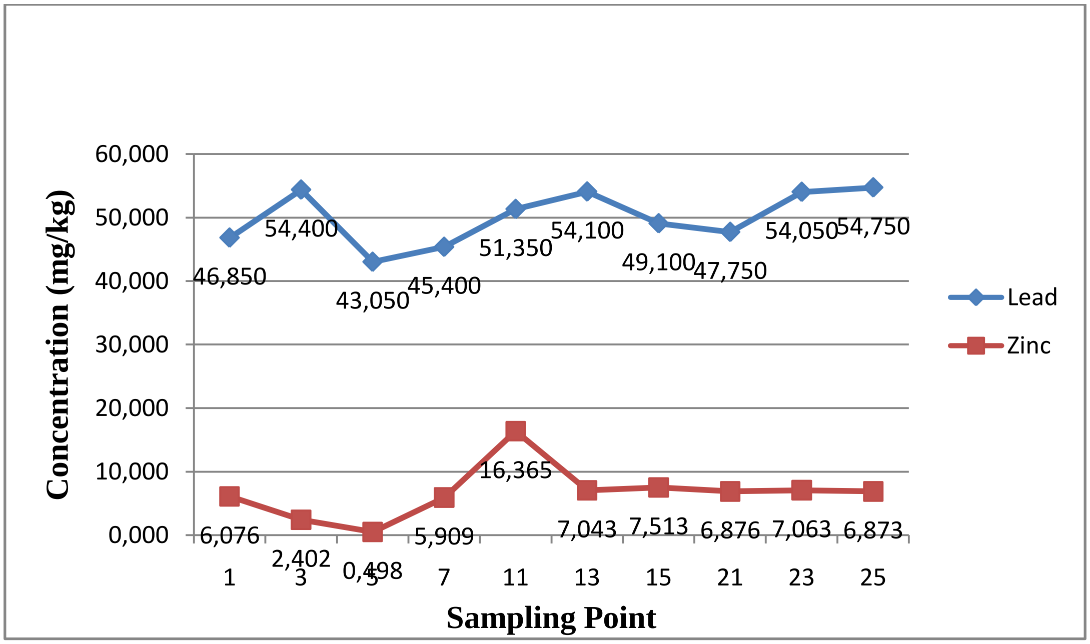

Figure 4.6: Graph of heavy metal concentration vs sampling point in sub surface soil

From the graph above, both shows that concentration of lead is higher compare to zinc concentration in top and sub surface of the soil. This is because Ulu Tualang closed landfill physical properties contain more source of Pb such as plastic color and steel structure painting. This can observe clearly when we are in Ulu Tualang closed landfill and with this condition it makes the soil contain higher concentration of Pb compare than Zn

- source of lead = Paint (plastic color, steel structures paintings), dust painting, cosmetics, children's jewelry and toys, vehicles
- source of Zn = Galvanized wire cages, toys, chains and water or food bowls zinc hardware (washers, nuts, wire)

Figure 4.7: Ulu Tualang Closed Landfill physical properties

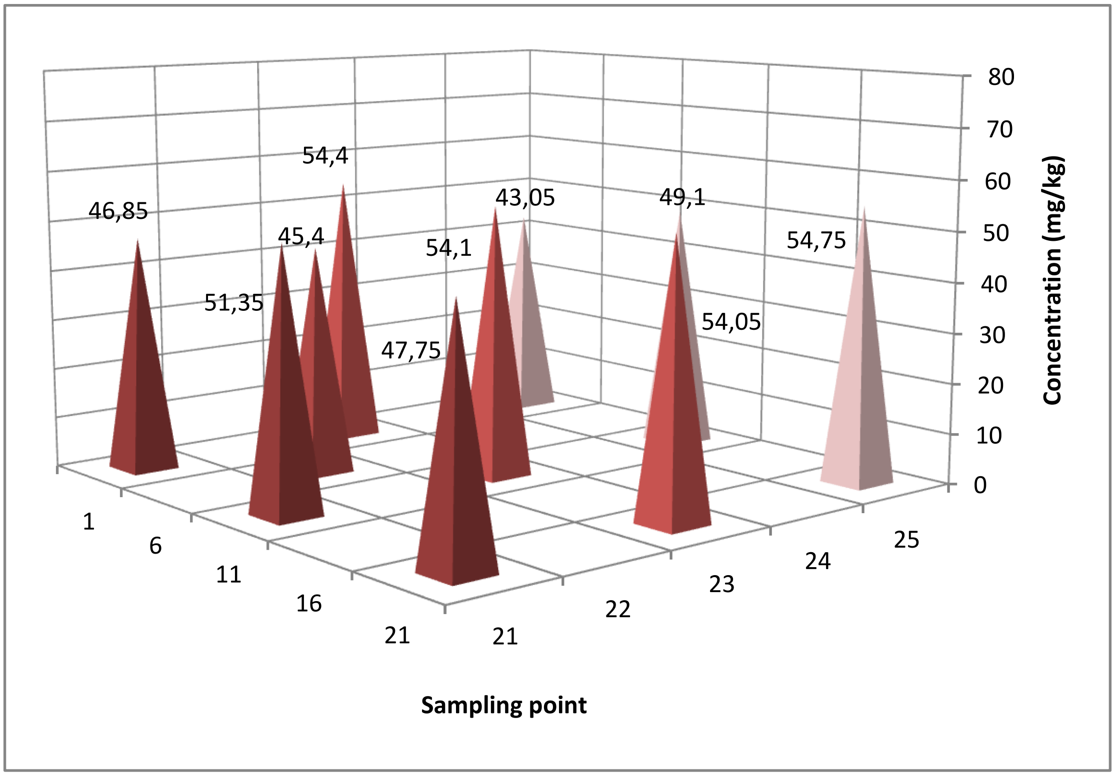

Figure 4.8: 3-D Graph to show distribution of lead in top surface soil

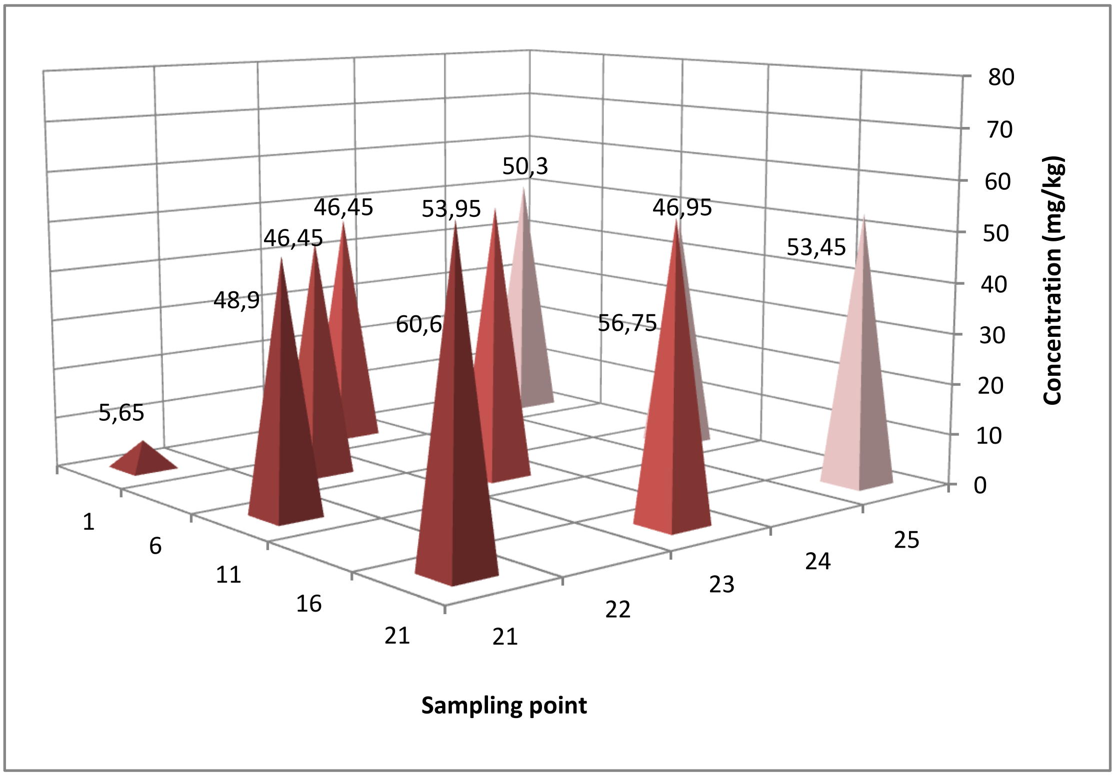

Figure 4.9: 3-D Graph to show distribution of lead in sub surface soil

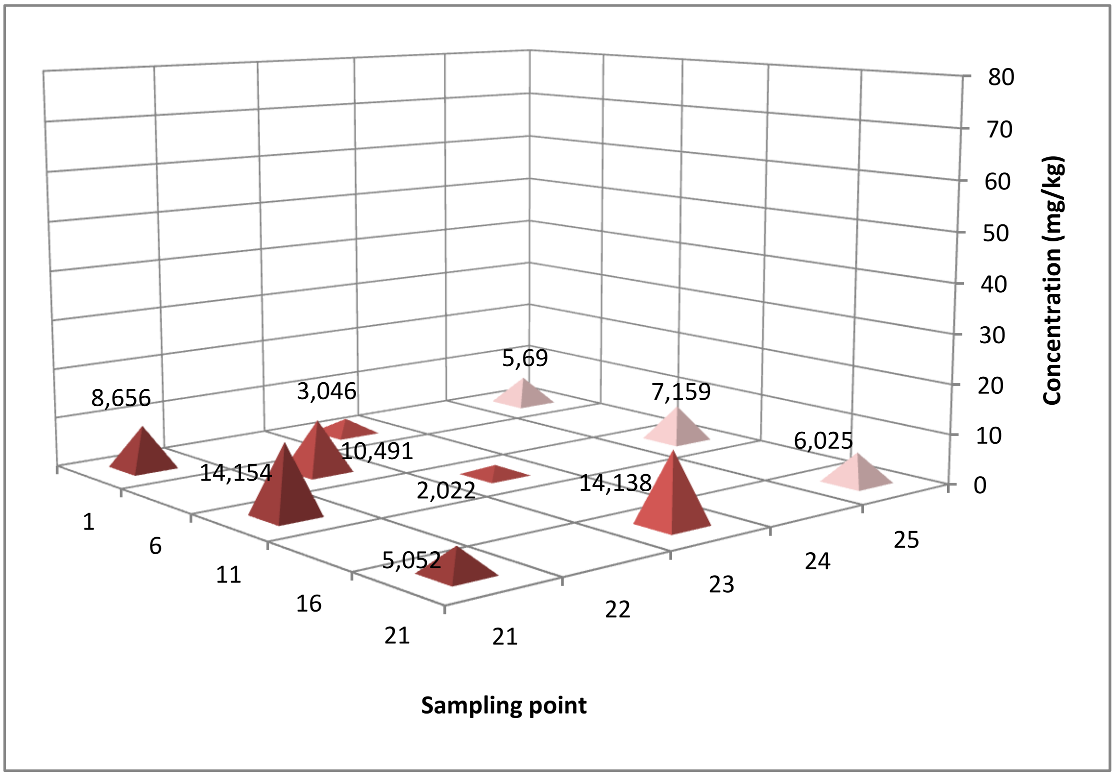

Figure 4.10: 3-D Graph to show distribution of zinc in top surface soil

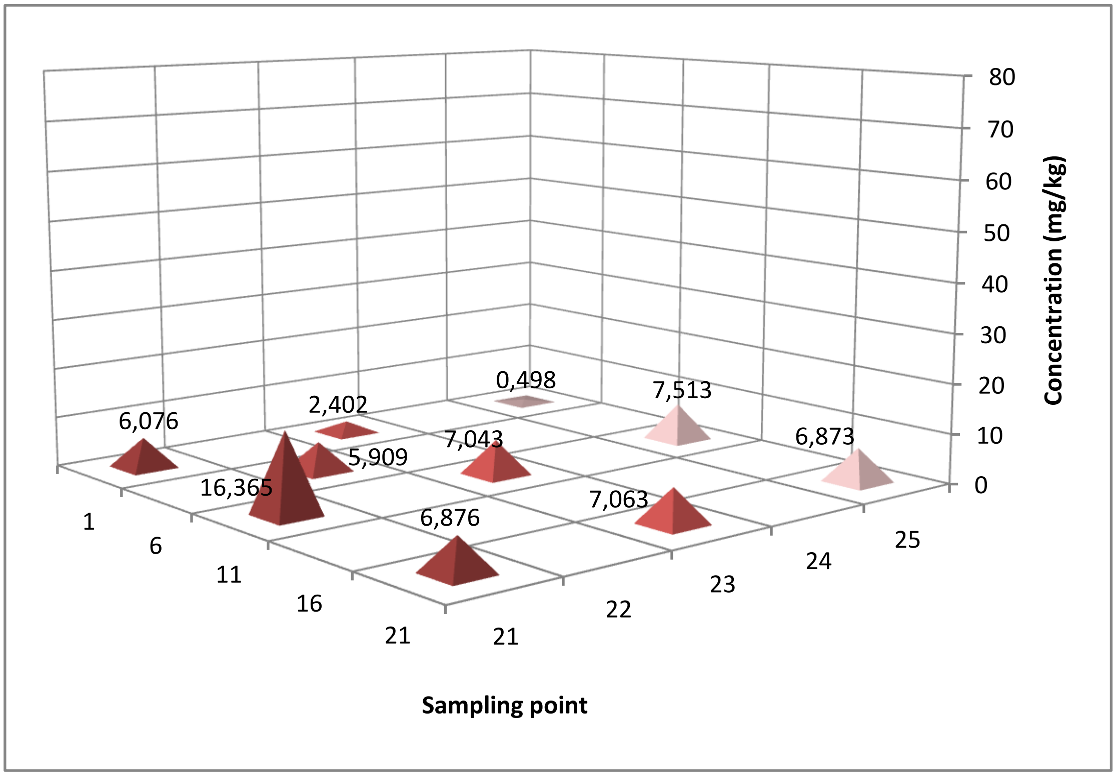

Figure 4.11: 3-D Graph to show distribution of zinc in sub surface soil

Table 4.5: Summary of Lead and Zinc Concentration Data

	Lead		**Zinc**	
	Top Soil	**Sub-surface**	**Top Soil**	**Sub-surface**
Minimum	43.05	5.65	2.022	0.498
Maximum	54.75	60.6	14.154	16.365
Median	52.725	51.425	8.088	11.702
Mean	50.08	46.945	7.6433	6.6618
EU PEL (CCME) Gonzalo M.A. Bermudez et al..2009	**375**		**600**	
US PEL (*Adapted from U.S. EPA, 1993)	**420**		**2800**	

- All values are in mg/kg unit
- CCME (Canadian Council of Ministers of the Environment) (1991)
- Both lead and zinc concentration value did not exceed from EU permissible exposure limit (PEL) and US EPA EPL standard

Table 4.6: pH values for top soil and sub surface soil

Sampling Point	**Top Soil (pH)**	**Sub-surface Soil (pH)**
1	5.11	5.68
3	6.70	5.65
5	6.33	6.33
7	6.29	6.11
11	6.86	6.90
13	6.59	6.89
15	6.55	6.62
21	6.78	6.69
23	7.20	6.72
25	6.60	6.81

Table 4.7: Type of soil

Sampling Point	**Top Soil**	**Sub-surface Soil**
1	Clay, Coarse sand, white-brown	Clay, Coarse sand, white-brown
3	Clay, Coarse sand, white-brown	Clay, Coarse sand, white-brown
5	Clay, white-brown	Clay, white-brown
7	Clay, Coarse sand, white-brown	Clay, Coarse sand, white-brown
11	Peat, dark-brown	Peat, dark-brown
13	Peat, dark-brown	Peat, dark-brown
15	Peat, dark-brown	Peat, dark-brown
21	Laterite, red-brown	Laterite, red-brown
23	Laterite, red-brown	Laterite, red-brown
25	Laterite, red-brown	Laterite, red-brown

From table 5.6 and 5.7, the data also showed that clay type soil have lower pH than other type of soil. For peat soil, the pH value ranged 6.55-6.62 while laterite soil pH value ranged from 6.60-7.20.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

As a conclusion, the determination of heavy metals in sample collected in Ulu Tualang closed landfill was run successfully. Starting with first stage which is sample collecting and pre-treatment procedure, followed by second stage which is digestion procedure and end by third stage which is sample analysis using AAS, it show that soil sample that taken from this landfill contain lead (Pb) and zinc (Zn)

Based on result and discussion, it shows us that the concentration value of Pb and Zn did not exceed permissible exposure limit (PEL) data (Pb = 43.05 - 54.75 mg/kg for top surface and Pb = 5.65 - 60.6 mg/kg for sub surface soil, Zn = 2.022 - 14.154 mg/kg for top surface and Zn = 0.498 - 16.365 mg/kg for sub surface soil). This heavy metal distribution data are very useful for become main references or guidelines for environmental assessment and also for soil treatment in order to avoid environmental pollution from becoming worst.

5.2 Recommendation

The work done in this study shows that all equipment uses, handling procedure and sample collecting procedure can be uses to determine heavy metal concentration in soil sample. However, in order to produce good result, it is suggested that future study will consider to;

Soil Sample

- Increase the gridding area that can scattered the whole landfill area
- Taken randomly sample but it must cover all the landfill area
- Increase the depth for taking sample and taking sample with several layer at one sampling point because concentration of the heavy metal may different at different layer of the soil

Method

- Reduces the uses of steel equipment to minimize the potential of external contamination factor

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APPENDICES

Figure A.1: Ulu Tualang Landfill

Figure A.2: Collected Samples

Figure A.3: Sample (Lump form)

Figure A.4: Sample (Powder form)

Figure A.5: Digestion Stage

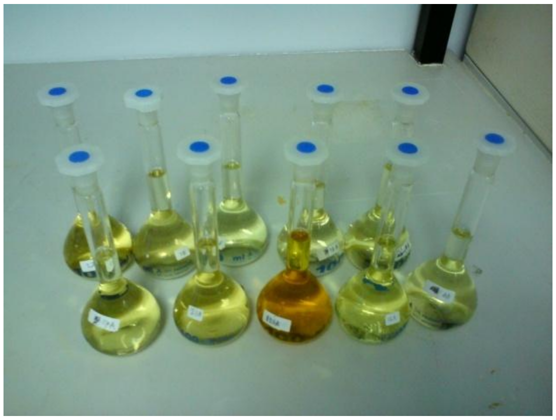

Figure A.6: Sample (Liquid form)

Figure A.6: Standard solution of zinc, Zn and lead, Pb

Figure A.7: Atomic Adsorption Spectrometer (AAS)

Table A.1: Lead (Pb) reading in ppm at first test

Sample	A (top surface)	B (sub surface)
1.	- 5.17	- 4.07
2.	-5.20	- 4.41
3.	- 3.87	- 5.28
4.	- 4.57	- 4.67
5.	- 3.81	- 2.58
6.	- 4.81	- 3.94
7.	- 2.18	- 4.76
8.	- 4.18	- 4.97
9.	- 5.11	- 4.87
10.	-4.72	- 5.15
11.	- 1.92	- 1.70
12.	-4.62	- 1.61
13.	- 5.05	- 2.00
14.	- 4.13	- 0.87
15.	- 5.17	- 4.11
16.	- 4.17	- 4.28
17.	-4.60	- 4.09
18.	- 4.33	- 5.05
19.	- 4.78	- 3.99
20.	- 1.64	- 5.26
21.	- 4.44	- 4.78
22.	- 4.49	- 4.60
23.	- 2.20	- 4.24
24.	- 4.48	- 2.27
25.	- 5.15	- 5.26

Table A.2: Lead (Pb) reading after 5 ppm spike method

Sample	**A (top surface)**	**B (sub surface)**
1.	14.37	6.13
3.	6.13	14.29
5.	13.61	15.06
7.	14.08	14.29
11.	15.27	14.78
13.	15.82	15.79
15.	14.82	14.39
21.	14.55	17.12
23.	15.81	16.35
25.	15.95	15.69

Table A.3: Zinc (Zn) reading in ppm at first test

Sample	**A (top surface)**	**B (sub surface)**
1.	86.56	60.76
2.	30.46	24.02
3.	56.90	4.98
4.	7.61	- 10.64
5.	67.27	66.53
6.	52.24	64.98
7.	104.91	59.09
8.	10.19	- 27.51
9.	26.73	- 29.31
10.	19.64	23.10
11.	141.54	163.65
12.	73.44	45.85
13.	20.22	70.43
14.	79.17	78.99
15.	71.59	75.13
16.	44.16	41.27
17.	29.14	71.58
18.	70.66	16.63
19.	51.76	63.51
20.	92.88	7.52
21.	50.52	68.76
22.	23.22	19.73
23.	141.38	70.63
24.	61.32	92.71
25.	60.25	68.73